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14 November 2011

Mr. Rafael Casanova
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U.S. Environmental Protection Agency (EPA) Region 6
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Subject: **Final Sampling and Analysis Plan, Rev. 01**
State Marine of Port Arthur Superfund Site
First Five-Year Review
EPA Region 6 Remedial Action Contract 2
Contract: EP-W-06-004
Task Order: 0079-FRFE-06BX

Dear Mr. Casanova:

EA Engineering, Science, and Technology, Inc. (EA) is enclosing three hard copies of the Final Sampling and Analysis Plan, Rev. 01, which includes the 8 November 2011 EPA comments, for the above-referenced Task Order. In addition, an electronic copy of this document was placed on the EPA/EA SharePoint website on 14 November 2011.

If you have any questions regarding this submittal, please contact me at (972) 315-3922.

Sincerely,

Stan Wallace
Project Manager

Enclosure

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Sampling and Analysis Plan

First Five-Year Review
State Marine of Port Arthur Superfund Site
Port Arthur, Jefferson County, Texas
EPA ID No. TXD099801102

Remedial Action Contract 2 Full Service
Contract: EP-W-06-004

Prepared for

U.S. Environmental Protection Agency
Region 6
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Prepared by

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Revision: 01
EA Project No. 14342.79

Sampling and Analysis Plan

First Five-Year Review
State Marine of Port Arthur Superfund Site
Port Arthur, Jefferson County, Texas
EPA ID No. TXD099801102

Remedial Action Contract 2 Full Service
Contract: EP-W-06-004

10 October 2011

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EA Program Manager

7 October 2011

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U.S. Environmental Protection Agency Region 6 Task Order Monitor

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6	Standard Operating Procedures
7	Required Volume, Containers, Preservatives, and Holding Times
8	Frequency of Field Quality Control Samples

LIST OF ACRONYMS AND ABBREVIATIONS

α	Alpha
β	Beta
bgs	Below ground surface
°C	Degrees Centigrade
CLP	Contract Laboratory Program
COC	Contaminant of concern
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
DQA	Data quality assessment
DQO	Data quality objective
EA	EA Engineering, Science, and Technology, Inc.
EPA	U.S. Environmental Protection Agency
ft	Feet(foot)
H_o	Null hypothesis
H_a	Alternative hypothesis
in.	Inch(es)
LCS	Laboratory control spike
µg/kg	Microgram(s) per kilogram
mg/kg	Milligram(s) per kilogram
MD	Matrix duplicate
MS	Matrix spike
MSD	Matrix spike duplicate
NOAA	National Oceanic and Atmospheric Administration
PARCC	Precision, accuracy, representativeness, completeness, and comparability
QA	Quality assurance
QC	Quality control
RAC	Remedial Action Contract
RPD	Relative percent difference

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

SAP	Sampling and Analysis Plan
Site	State Marine of Port Arthur Superfund Site
SOP	Standard operating procedure
SOW	Statement of Work
SVOC	Semivolatile organic compound
VOC	Volatile organic compound
VSP	Visual Sample Plan

DISTRIBUTION LIST

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1. PROJECT DESCRIPTION AND MANAGEMENT

EA Engineering, Science, and Technology, Inc. (EA) has been authorized by the U.S. Environmental Protection Agency (EPA), under Remedial Action Contract (RAC) Number EP-W-06-004, Task Order 0079-FRFE-06BX, to conduct a Five-Year Review at the State Marine of Port Arthur Superfund Site (Site). The Site Record of Decision has recommended that a sampling event be performed along with the Five Year Review, and EA has prepared this Sampling and Analysis Plan (SAP) in accordance with: (1) specifications provided in the EPA Statement of Work (SOW), dated 5 August 2011 (EPA 2011a); and (2) the EPA-approved EA Work Plan (Revision 01), dated 1 September 2011 (EA 20011a).

This SAP is a combination Quality Assurance Project Plan and Field Sampling Plan. It details the field sampling schedule, sample collection procedures, and analytical methods required to collect sufficient data to perform a Five-Year Review for the Site. Combining the Quality Assurance Project Plan and Field Sampling Plan into the SAP allows a streamlining of the planning process, while ensuring that data collected are of sufficient quality for their intended use.

This SAP was prepared in conjunction with the independent site-specific Health and Safety Plan (EA 20011b), which specifies employee training, protective equipment, personal air monitoring procedures, medical surveillance requirements, standard operating procedures (SOPs), and contingency planning procedures. Together, the SAP and Health and Safety Plan present the overall approach for implementing the Five-Year Review field program:

This SAP was prepared in accordance with EA's Quality Management Plan (EA 2005) and meets requirements set forth in *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operation (QA/R-5)* (EPA 2001) and *Guidance for Quality Assurance Project Plans (QA/G-5)* (EPA 2002a).

This SAP describes procedures to assure that the project-specific data quality objectives (DQOs) are met, and that the quality of data (represented by precision, accuracy, representativeness, completeness, and comparability [PARCC], and sensitivity) is known and documented. The SAP presents the project description, project organization and responsibilities, and quality assurance (QA) objectives associated with the sampling and analytical services to be provided in support of the Five-Year Review. Table 1 demonstrates how this SAP complies with elements of a Quality Assurance Project Plan currently required by EPA guidance (EPA 2001, 2002a).

TABLE 1 ELEMENTS OF EPA QA/R-5 IN RELATION TO THIS SAMPLING AND ANALYSIS PLAN

EPA QA/R-5 Quality Assurance Project Plan Element	EA SAP (QTRAK NO. 08-056)
A1 Title and Approval Sheet	Title and Approval Sheet
A2 Table of Contents	Table of Contents
A3 Distribution List	Distribution List
A4 Project/Task Organization	1.0 Project Description and Management
A5 Problem Definition/Background	1.1 Problem Definition and Background
A6 Project/Task Description	1.2 Description of Project Objectives and Tasks

EPA QA/R-5 Quality Assurance Project Plan Element		EA SAP (QTRAK NO. 08-056)	
A7	Quality Objectives and Criteria	1.3 1.4	DQOs QA Objectives for Measurement Data
A8	Special Training/Certification	1.5	Special Training and Certification
A9	Documents and Records	1.6	Documents and Records
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B2	Sampling Methods	2.3 2.4 2.7	Sampling Methodology Sample Processing Sample Designation
B3	Sample Handling and Custody	2.8 2.9	Sample Container, Volume, Preservation, and Holding Time Requirements Sample Handling and Custody
B4	Analytical Methods	2.10	Analytical Methods Requirements
B5	Quality Control (QC)	2.11	QC Requirements
B6	Instrument/Equipment Testing, Inspection, and Maintenance	2.11.4	Instrument and Equipment Testing, Inspection, and Maintenance Requirements
B7	Instrument/Equipment Calibration and Frequency	2.12	Instrument Calibration and Frequency
B8	Inspection/Acceptance of Supplies and Consumables	2.13	Requirements for Inspection and Acceptance of Supplies and Consumables
B9	Non-Direct Measurements	2.14	Data Acquisition Requirements (Non-Direct Measurements)
B10	Data Management	2.15	Data Management
C1	Assessment and Response Actions	3.1	Assessment and Response Actions
C2	Reports to Management	3.2	Reports to Management
D1	Data Review, Verification, and Validation	4.1	Data Review and Reduction Requirements
D2	Validation and Verification Methods	4.2	Validation and Verification Methods
D3	Reconciliation with User Requirements	4.3	Reconciliation with DQOs

The overall QA objectives are as follows:

- Attain QC requirements for analyses specified in this SAP
- Obtain data of known quality to support goals set forth for this project
- Document aspects of the quality program, including performance of the work and required changes to work at the Site.

The EPA Region 6 Task Order Monitor, Mr. Rafael A. Casanova, is responsible for the project oversight. The Project Officer for EPA Region 6 is Ms. Rena McClurg. The Contracting Officer for EPA Region 6 is Mr. Michael Pheeny. EA will perform tasks under this Task Order in accordance with this SAP. The EA Project Manager, Mr. Stan Wallace, is responsible for implementing activities required by this Task Order. Figure 1 presents the proposed project organization for this Task Order.

1.1 PROBLEM DEFINITION AND BACKGROUND

This section describes the following:

- Purpose of the investigation and sampling events (Section 1.1.1)
- Site background and description (Section 1.1.2).

1.1.1 Purpose of the Investigation and Sampling Events

The purpose of this Task Order is to perform the Five-Year Review at the site as described in the April 2007 Record of Decision (EPA 2007a). A Five-Year Review generally determines whether the remedy at a site is protective of human health and the environment. The Five-Year Review should confirm that immediate threats have been addressed and that EPA expects the remedy to be protective when the remedial action is completed. The Five-Year Review does not reconsider decisions made during the selection of the remedy, but evaluates the implementation and performance of the selected remedy. The purpose of the sampling event to be performed along with the review is to confirm that contaminant levels in sediment are still protective of human health and ecological receptors.

1.1.2 Site Background and Description

This description of the Site was adopted from the supplemental remedial investigation (CH2M HILL 2006).

1.1.2.1 Site Background

The Site is located on Old Yacht Club Road, Pleasure Islet, within the City of Port Arthur, Jefferson County, Texas. Pleasure Islet is a peninsula located approximately 0.5 mile southwest of the confluence of the Neches River and Sabine Lake. The approximately 17.2-acre Site consists of tracts 3 and 6 on Old Yacht Club Road. The Sabine-Neches Canal forms the eastern border of the Site. The Site is bordered to the north by the Palmer Barge Lines Superfund Site, to the west by Old Yacht Club Road, and to the south by undeveloped land owned by the State Marine property owner. Figure 2 provides an overview of the Site.

The Islet is a man-made landmass consisting of dredge materials generated during the construction and maintenance of the Sabine-Neches Canal. The canal was constructed between 1898 and approximately 1920, near Sabine Lake and the Neches River, between the current Site location and the mainland. Pleasure Islet did not exist at the time as the area encompassing the Site was actually part of the northern tip of Pleasure Island. Between 1955 and 1957, a portion of the canal along the western side of Pleasure Islet was abandoned and a new canal cut along the eastern and southern sides of Pleasure Islet. Pleasure Islet was created when a land bridge was constructed across the abandoned portions of the canal, between the northern tip of Pleasure Island and the mainland.

During a routine aerial surveillance of Jefferson County in July 1980, the Site was identified as a potentially hazardous waste site. The Site was a barge maintenance, cleaning, and salvage facility from 1974 through the early 1980s. Prior to that time, the City of Port Arthur used the Site as a municipal landfill between 1963 and 1974. As a result of subsequent investigations by the Texas Department of Water Resources, predecessor agency to the Texas Water Commission, then the Texas Natural Resources Conservation Commission, which is currently the Texas

Commission on Environmental Quality, the Site became a Listed Hazardous Waste Site. Following Texas Water Commission scoring efforts in 1987 and later in 1992 using the Hazard Ranking Scoring system, the Site was referred to EPA Region 6 because the Site scored a 50.00 under the new Hazard Ranking Scoring, indicating eligibility for listing on the National Priorities List. EPA completed two separate Hazard Ranking Scoring efforts, the last of which occurred in 1997. The Site was officially placed on the National Priorities List on 27 August 1998. In June 1998, EPA requested CH2M HILL to conduct a preliminary remedial investigation/feasibility study and prepare a Technical Memorandum. A remedial investigation was performed at the site in Autumn 2001.

1.1.2.2 Operational History

Prior to 1957, Pleasure Islet was a part of Pleasure Island. Before 1955, few changes were observed in the area of Pleasure Islet. The islet was heavily vegetated and undeveloped. The islet received dredge materials, most likely from the construction and routine maintenance of the initial intercoastal canal. Ownership of Pleasure Islet was transferred from the State of Texas to the City of Port Arthur in 1955. A land bridge was constructed across the abandoned portions of the Sabine-Neches Canal, located on the western side of the islet, and development in the area began in 1957.

The City of Port Arthur began municipal landfill operations on the northern and central portions of the islet in approximately 1963. The landfill operation initially consisted of a burn pit in which wastes were incinerated. Burn operations ceased in 1969, after which time the landfill was used only for disposal of wastes. Trench methods were used for disposal of wastes in the landfill. In general, 50-foot (ft) wide trenches were excavated to depths of approximately 6-9 ft. Approximately 6 inches (in.) of cover were placed over the wastes every 2 days, and a minimum of 2 ft of soil was placed over the area once a trench was filled.

The City of Port Arthur closed the landfill in December 1974 in accordance with Texas Department of Health regulations. This required that the entire landfill be covered with approximately 2 ft of fine-grained fill material. It is believed that the cover material used was dredge materials that originated on the islet.

Operations at the Site began about 1973. The Site was initially operated under the names “State Welding and Marine Works” and “Golden Triangle Shipyard.” The specific operations at the Site during this time are unknown. However, operations likely included marine salvage and repairs, and off-loading and bulk storage of petroleum products.

The construction of wastewater impoundments in the northwestern portion of the Site was also reported. The impoundments were reported to have been approximately 2 acres in size, unlined, and constructed with earthen dikes. The impoundments were used to store oil and wastewater from barge cleaning operations. Inspection reports from the Texas Air Control Board and Texas Department of Water Resources indicate that wastewater from the barge cleaning operations was directed to two aboveground storage tanks and then pumped to the wastewater impoundments. Some oil from the aboveground storage tanks was diverted to an old ship located on land that was used as an oil/water separator.

A Texas Department of Water Resources inspection report from August 1980 indicated that facility modifications were made at the Site to allow process waste oils to be converted into bunker fuels. The facility modifications included a distillation column, three aboveground storage tanks, one 20,000-barrel tank, and two 10,000-barrel tanks. Additional smaller tanks were later constructed at the Site. A Texas Department of Water Resources inspection report associated these storage tanks with the Lauren Refining Company, which was also owned and operated by the owners of the Site. It is not known when the Lauren Refining Company first began operations at the Site, but it is likely to have occurred sometime in 1980.

1.1.2.3 Source Description

The wastes generated and/or disposed at the Site appear to have included both raw product (original barge contents) and effluent generated during the cleaning and maintenance of barges. Waste generated and stored at the Lauren Refining Company included wastes similar to those generated at the Site. Five different waste areas that were identified are listed below:

- Wastewater treatment facility (excluding the impoundments)
- Wastewater impoundments
- Former Lauren Tank Farm
- Sunken dock barges and offshore sunken barges
- Vehicle storage and maintenance and waste storage/handling areas.

The locations and approximate sizes of the waste management areas, shown in Figure 2, were compiled from aerial photographs and Site inspection reports. Additional information regarding these areas is provided below:

- The former wastewater treatment facility includes all structures and work areas between the dock barges and the former impoundments, including an area of blasting sand occurring at the shoreline and dock barges.

- The impoundments include the areas previously identified on historical aerial photographs.

- The former tank farm, which is considered a waste management area, includes the boundary of the former tank farm as well as the boundary of a spill that migrated outside of this area.

- The dock barges (sunken and/or floating) and sunken barges are considered waste management areas and include sediments within and around the barges.

- The vehicle storage, maintenance, and waste storage/handling areas include most of the area between the former tank farm and the former wastewater treatment facility. This area includes wastes associated with the repair or storage of vehicles or salvage materials, i.e., marine engines. Also included are at least three storage tanks from the Former

Lauren Tank Farm, which were moved to the area between the office building and pump house.

1.1.2.4 Site Environment and Features

This section provides an overview of the Site environmental setting. Described in this section are the Site physical characteristics including topography, Site features, surface water, geology and hydrogeology, ground water use, and ecological resources.

Topography

Ground elevations range from sea level along the shoreline to a maximum of 11 ft above mean sea level in the north-central portions. The highest relief on Pleasure Islet is associated with the City of Port Arthur's former municipal landfill, which underlies most of the central and northern portions of the Islet. Onsite, elevations range from approximately 2 to 7 ft above mean sea level. Drainage on the Islet is toward the adjacent waterways with surface drainage on the Site occurring to the east-southeast.

Site Features

The understanding of current conditions and Site features is based on aerial photographs, a Site inspection conducted in August 1998, and the 2003 remedial investigation. Vehicle access to the Site is limited to a single dirt road originating at the western Site border along Old Yacht Club Road. Within the Site, dirt roads and trails connect various areas of current or historical operations. Along the shoreline are two sunken barge docks, orientated parallel to and forming part of the shoreline. These two structures are the primary location where barges were moored during cleaning or maintenance. Two additional sunken barges lie perpendicular to the shoreline, as well as a third barge that is oriented parallel to the shoreline. These features are clearly shown on 1998 aerial photographs, as well as older photographs. Dredging of the canal in this area occurs approximately every 2 years.

Old marine equipment, including cranes and marine salvage debris, are present on the dock barges and inland areas. Several structures still exist, including a maintenance shed, a former office building, former wastewater treatment facility structures (boiler house, compressor building, and a former pump house), and at least one additional structure of unknown use. Most of these structures are partially collapsed and appear abandoned except for the maintenance shed, which may still be in use. Several aboveground storage tanks and 55-gallon drums sit at the Site. The status of the tanks and their contents was not investigated during the preliminary inspection, although several drums appeared to be partially full. Numerous vehicles, including old cranes, pickup trucks, and several tractors and tractor-trailers, are parked at the Site. None of the vehicles looked operational and several appear to be in various states of salvage or repair. The soil around many of the vehicles is oil stained.

The Site is partially vegetated with native shrubbery and grasses. Much of the Site is overgrown and few structures are recognizable relative to the original operations observed on historical aerial photographs. Less than 10 ft of shoreline is exposed at most locations. However, aerial

photographs indicate a broader shoreline between the two dock barges, which were not visited during the preliminary inspection. The area is tidally influenced (1-2 ft) and the amount of shoreline exposed is likely to vary between tides. During the preliminary inspection, passing ships were observed to generate significant wave action at the shoreline. Based on the wave action and proximity of the canal, it is likely that the shoreline is eroding. Several trees exist and wetland vegetation occurs near or along some areas of the shoreline. Sea gulls were observed roosting on the offshore sunken barges.

Surface Water

The surface water bodies near the Site include Sabine Lake, the Sabine and Neches rivers, and the Sabine-Neches Canal. The Site is located along the shore of Sabine Lake. These surface water bodies are used for a variety of purposes.

Sabine Lake is used for both commercial and recreational fishing. The lake also serves as a tourist and recreational water body for activities such as swimming and boating. Sabine Lake is an estuarine environment that supports vegetation, fish, crustacean, amphibian, reptile, and marine mammal species. In addition, the lake is used by migratory and fish eating species of birds. The lake is home to several threatened and endangered species including the green turtle (*Chemlondia mydas*), loggerhead sea turtle (*Caretta caretta*), ridley sea turtle (*Lepidochelys kmpii*), gulf salt marsh snake (*Nerodia clarkia*), and the Texas diamondback terrapin (*Malachlemys terrapin*).

The Sabine-Neches Canal connects the Gulf of Mexico to the cities of Port Arthur, Beaumont, and Orange. The canal has been improved and dredged over the years with the expansion of oil operations and chemical industries in the area. The canal is a busy water body with the shipping of assorted cargo and petroleum or petroleum-related products.

Site Geology and Hydrogeology

The geology and hydrogeology of the Site are unusual because the Site is situated on a man-made landmass within a waterway of considerable flow volume. This landmass consists of dredge materials generated during construction and maintenance of the Sabine-Neches Canal. The City of Port Arthur used the Site as a municipal landfill from approximately 1963 until 1974. Trench and fill methods were employed for disposal of the municipal waste. The entire Site was subsequently capped with what is believed to be dredge materials. Municipal landfill debris is present in the shallow subsurface across the majority of the Site.

During the remedial investigation conducted by Weston Solutions, Inc. in 2001, the presence of landfill debris prohibited recovery of subsurface soil samples in most instances; thus, detailed information on the subsurface soil conditions across the majority of the Site could not be obtained. The landfill material is usually encountered approximately 2 ft below the surface and typically ranges in thickness from 3 to 10 ft. Based on observations during monitor well installation activities, the soils underlying the landfill debris consist of dredge materials underlain by alluvial deposits to the depths investigated (40-55 ft below ground surface [bgs]).

These materials consisted primarily of saturated, very soft, gray silty clay. At one location, MW061, saturated, gray, silty sand was encountered at a depth of approximately 35 ft bgs.

Monitor wells were installed at three locations during the remedial investigation field activities. The well locations were selected to obtain samples downgradient of the most significant source areas. In addition, based on observations during monitor well installation, ground water was typically encountered at depths of approximately 2-4 ft bgs. Below this depth, soils remained saturated throughout the depths investigated (40-55 ft bgs) without the presence of a distinct confining layer. Due to the extensive saturated thickness, two nested wells were installed at each of the three monitor well locations, one to screen the upper 20 ft and one to screen the next 20 ft below. Because no previous ground water sampling had been conducted, the wells were used as a screening assessment of the ground water conditions.

Although an obvious aquitard was not observed during the well installations, the water quality data indicate the presence of two separate zones. Salinity measurements were consistently higher in the deeper wells, which could indicate that a shallow perched zone is present and has a fresh water impact from precipitation. In addition, the piezometric surface of the shallow well measurements appears to follow the land surface topography. This is commonly seen in shallow perched aquifers. Based on the depth-to-water measured in the shallow screened wells, the base of the landfill may serve as the lower aquitard of a perched shallow ground water zone. For both the deep and shallow screened wells, ground water appears to flow to the southeast with a hydraulic gradient of 1.18×10^{-3} ft/ft and 2.13×10^{-2} ft/ft, respectively.

Ground Water Use

The main source of deep ground water in the area of the Site is the Gulf Coast Aquifer. The Gulf Coast Aquifer consists of alternating beds of clay, silt, sand, and gravel that are hydraulically connected and form a large, leaky, artesian aquifer system. Ground water is used for industrial and irrigation purposes approximately 1.5 miles from the Site. There is one domestic well located approximately 1 mile from the site. This well is hydraulically upgradient from the Site. In addition, 33 public, industrial, unknown use, test, and domestic wells were identified within a 4-mile radius of the Site. Based on the ground water flow direction documented during the remedial investigation, these wells appear to be located upgradient or crossgradient from the Site.

Ground water at the Site is not known to be used for drinking purposes. There is no current or anticipated future use of ground water as a source of potable water at the Site. Because of the underlying landfill, which precludes use of shallow ground water and the proximity to brackish surface water, ground water is believed non-potable.

Ecological Resources

Pleasure Islet is situated in the northwest corner of Sabine Lake, a tidally-influenced estuary. Part of the designated use of this lake is high quality aquatic habitat and shellfish waters. The lake, which is an active habitat for a variety of water fowl and aquatic wildlife, contains a number of environmentally sensitive wetlands and bird rookeries, most of which have been

designated as a National Wildlife Refuge. Critical habitats for various threatened or endangered species occur within a 15-mile radius of the Site. The high quality aquatic habitat provided by Sabine Lake supports a variety of wetland plants. Detailed information regarding wetlands, protected species, and critical habitat locations was not available in the reviewed site literature. Both commercial and noncommercial fishing occur in the area. There are three surface water intakes used only for industrial purposes within 15 miles downstream of the site. There are no drinking water intakes downstream of the site.

1.2 DESCRIPTION OF PROJECT OBJECTIVES AND TASKS

This section describes the project objectives and tasks for this SAP.

1.2.1 Project Objectives

The primary objective of the Five-Year Review is to determine whether the remedy at the Site is protective of human health and the environment. Specifically, the Five-Year Review involves a review of site documents and site-specific requirements, a site inspection, interviews with regulatory and site management personnel, and collection and analysis of sediment samples.

1.2.2 Project Tasks

To complete the Five-Year Review site activities, EA will perform the following tasks (with subtasks), which are generally outlined in the Task Order SOW (EPA 2011a). This SAP specifically addresses the sediment sampling and monitoring task, which is detailed in Sections 2, 3, and 4 of this SAP:

- Project planning and support
- Community involvement
- Document review
- Standards (applicable or relevant and appropriate requirement) review
- Site visit/interview
- Site inspection/technology review
- Five-Year Review Report preparation
- Sediment sampling and monitoring.

EA's field activities will be conducted in accordance with this SAP to ensure proper sample management, including accurate chain-of-custody procedures for sample tracking, protective sample packaging techniques, and proper sample preservation techniques, as well as EA's site-specific Health and Safety Plan (EA 2011b). Sample management will be conducted using the EPA-approved Forms II Lite software.

1.3 DATA QUALITY OBJECTIVES

The following sections present the DQOs for this project. Much of the information used to develop DQOs was obtained from the SOW issued by the EPA (2011a) and the EPA-approved Work Plan (EA 2011a). This DQO assessment follows EPA's 7-step DQO process (Table 2),

which is outlined in *Guidance on Systematic Planning Using the Data Quality Objectives Process (QA/G-4)* (EPA 2006a) and *Systematic Planning: A Case Study for Hazardous Waste Site Investigations (QA/CS-1)* (EPA 2006b). Additional information is referenced, as appropriate, in the following sections:

- Section 1.3.1 Purpose and Goal
- Section 1.3.2 Step 1 – State the Problem
- Section 1.3.3 Step 2 – Identify the Goal of the Study
- Section 1.3.4 Step 3 – Identify Information Inputs
- Section 1.3.5 Step 4 – Define the Boundaries of the Study
- Section 1.3.6 Step 5 – Develop the Analytical Approach
- Section 1.3.7 Step 6 – Specify the Performance or Acceptance Criteria
- Section 1.3.8 Step 7 – Develop the Plan for Obtaining Data.

1.3.1 Purpose and Goal

The purpose of defining the DQOs for the Site is to support decision making by applying a systematic planning and statistical hypothesis testing methodology to decide between alternatives. The goal was to develop an analytic approach and data collection strategy that was effective and efficient.

1.3.2 Step 1 – State the Problem

The first step in systematic planning process, and therefore the DQO process, is to define the problem that has initiated the study. As environmental problems are often complex combinations of technical, economic, social, and political issues, it is critical to the success of the process to separate each problem, define it completely, and express it in an uncomplicated format.

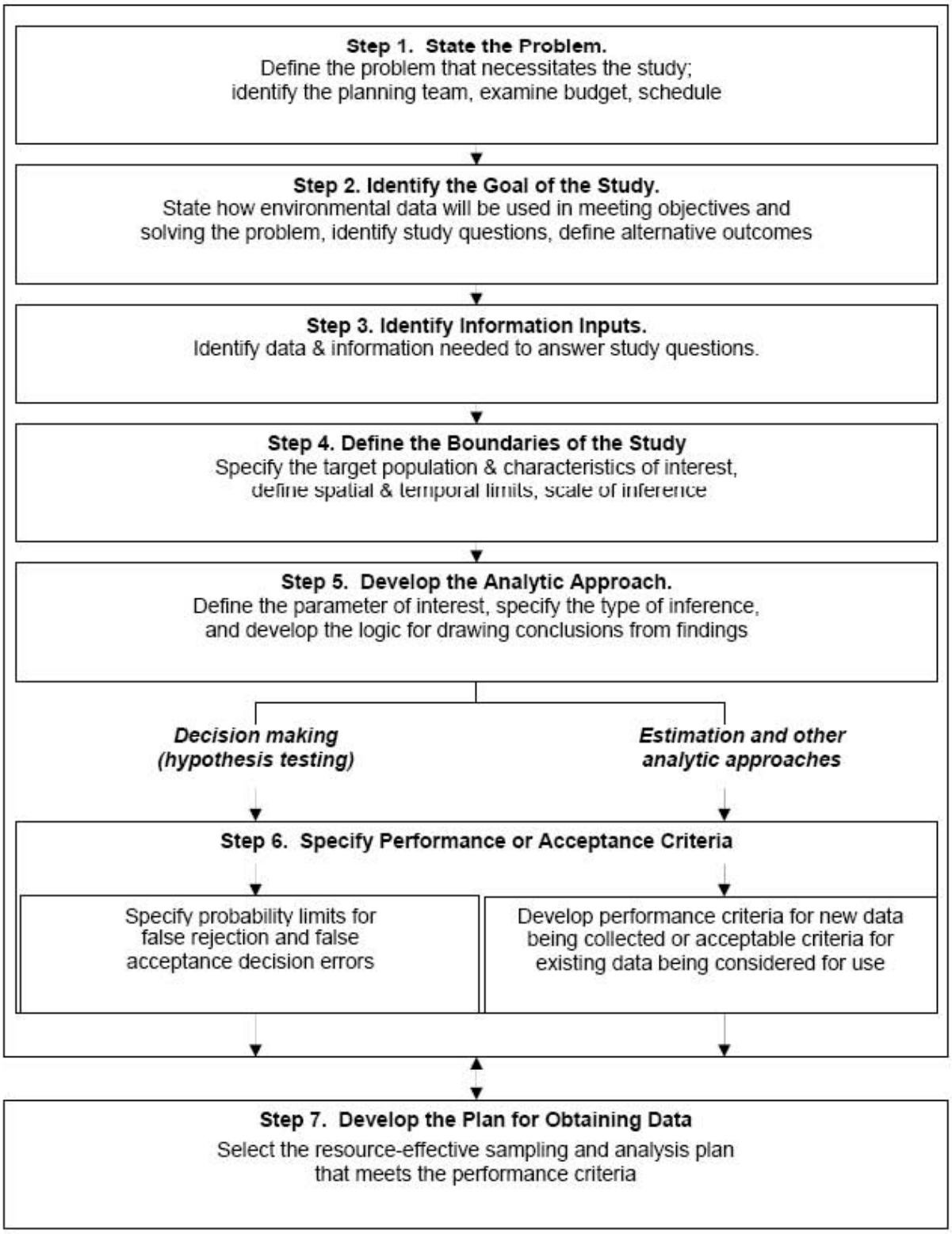
The most important activities in DQO Step 1 are as follows:

- Give a concise description of the problem
- Identify leader and members of the planning team
- Develop a conceptual site model of the environmental hazard to be investigated
- Determine resources (i.e., budget, personnel, and schedule).

1.3.2.1 Problem Description

As part of the Five-Year Review for the State Marine of Port Arthur, it must be confirmed that the selected remedy regarding potential exposure to sediment is protective of human health and ecological receptors.

TABLE 2 DATA QUALITY OBJECTIVE PROCESS



1.3.2.2 Planning Team Members and Stakeholders

A proven effective approach to formulating a problem and establishing a plan for obtaining information that is necessary to resolve the problem is to involve a team of experts and stakeholders that represent a diverse, multidisciplinary background. Such a team provides the ability to develop a concise description of complex problems, and multifaceted experience and awareness of potential data uses. Planning team members (including the leader) and stakeholders are presented below.

Planning Team Members

- Rafael A. Casanova, EPA Task Order Monitor
- Jeff Patterson, Texas Commission on Environmental Quality Project Manager.

Stakeholders

- EPA Region 6 Superfund Division Management
- EPA Headquarters
- Texas Game and Fish Department
- Jessica White, National Oceanic and Atmospheric Administration
- Tommy Mobley, Texas General Land Office
- Clare Lee, U.S. Fish and Wildlife Service
- Don Pitts, Texas Parks and Wildlife Department
- Andy Tirpak, Texas Parks and Wildlife Department
- Richard Seiler, Texas Commission on Environmental Quality
- Town of Port Arthur and citizenry
- Other interested parties.

If additional planning team members and/or stakeholders are identified as the Five-Year Review progresses, they will be incorporated into the decision-making process as appropriate.

1.3.2.3 Conceptual Site Model

It is critical to carefully develop an accurate conceptual site model of the environmental problem, as this model will serve as the basis for subsequent inputs and decisions.

The conceptual site model for the Site has been developed and is presented in the original remedial investigation (Weston Solutions, Inc. 2003) and the supplemental remedial investigation (CH2M HILL 2006). This information will not be represented for brevity. A summary of Site conditions is provided in Section 1.1.

1.3.2.4 Determine Resources

Resources should be identified by the planning team so that constraints (e.g., budget, time, etc.) associated with collecting/evaluating data can be anticipated during the project life cycle. To assist in this evaluation, the DQO process (e.g., developing performance or acceptance criteria)

and the SAP (i.e., for collecting and analyzing samples, and interpreting and assessing the collected data) should be completed.

EPA has tasked EA to perform the investigation and prepare the deliverables required for the Site Five-Year Review. EA will utilize the services of EPA's Region 6 Laboratory or EPA Contract Laboratory Program (CLP), depending on the needs of the Five-Year Review and the availability of the laboratory's services.

The EPA will perform a review of each required deliverable and provide comments as necessary. EPA will also solicit comments from other planning team members or stakeholders as appropriate. EPA anticipates that a Five-Year Review Report draft will be submitted to the EPA prior to 18 February 2012. Additional details pertaining to the schedule of events and deliverables necessary to meet this milestone are provided in the Five-Year Review Work Plan (EA 20011a).

1.3.3 Step 2 – Identify the Goal of the Study

Step 2 of the DQO process involves identifying the key questions that the study attempts to address, along with alternative actions or outcomes that may result based on the answers to these key questions. These two items are combined to develop a decision statement, which is critical for defining decision performance criteria later in Step 6 of the DQO process.

The most important activities in DQO Step 2 are as follows:

- Identify principal study question(s)
- Consider alternative actions that can occur upon answering the question(s)
- Develop decision statement(s) and organize multiple decisions.

1.3.3.1 Principal Study Question

The principal study question defines the question to be answered by the Five-Year Review. The principal study question is as follows:

> ***Do sediment concentrations present a potential unacceptable risk to human health and ecological receptors?***

1.3.3.2 Alternative Actions

The alternative actions provide principal study question alternatives in the Five-Year Review, as follows. Potential alternative actions include, but are not limited to:

- Conducting additional site characterization
- Updating the human health or ecological risk assessment utilizing the new sediment data
- Removing or remediating impacted sediment.

1.3.3.3 Decision Statement

For decision-making problems, the principal study questions and alternative actions are combined to develop decision statements, which are critical for defining decision performance criteria later in DQO Step 6.

The decision statement is as follows:

> ***Determine whether sediment concentrations present a potential unacceptable risk to human health and ecological receptors.***

1.3.4 Step 3 – Identify Information Inputs

Step 3 of the DQO process determines the types and sources of information needed to resolve: (1) the decision statement or produce the desired estimates, (2) whether new data collection is necessary, (3) the information basis the planning team will need for establishing appropriate analysis approaches and performance or acceptance criteria, and (4) whether appropriate sampling and analysis methodology exists to properly measure environmental characteristics for addressing the problem.

The most important activities in DQO Step 3 are as follows:

- Identify types and sources of information needed to resolve decisions or produce estimates
- Identify the basis of information that will guide or support choices to be made in later steps of the DQO process
- Select appropriate sampling and analysis methods for generating the information.

The EPA Five-Year Review SOW (EPA 2011a) and EPA approved Work Plan (EA 2011a) sets forth the framework and requirements for this effort.

1.3.4.1 Necessary Information and Sources

A variety of sources and types of information form the basis for resolving the decision statements. The following information and sources are necessary to resolve this step of the DQO process. The decision statement is supported by the following:

> ***Determine whether sediment concentrations present a potential unacceptable risk to human health and ecological receptors.***

- Sediment data will be collected and analyzed to evaluate the current concentrations of contaminants of potential concern.

- Risk-based screening levels developed for the remedial investigation and supplemental remedial investigation will be compared to current toxicity data and updated if necessary.

1.3.4.2 Basis of Information

The basis of information will guide or support choices to be made in later steps of the DQO process. The basis of information is supported by the following:

Determine whether sediment concentrations present a potential unacceptable risk to human health and ecological receptors.

- For the Site, Visual Sample Plan Version 6.1b (Pacific Northwest National Laboratory 2010) was used to establish a virtual random-start systematic grid for selecting systematic random samples (Appendix A). Biased or judgmental sample locations will be based on historical data, photographs, or visual cues. Details pertaining to the sediment sampling effort are contained in Section 2 and will not be repeated here for brevity.

- An evaluation of previous investigation data will utilize EPA guidance documents including, but not limited to: Guidance for Data Usability in Risk Assessment (EPA 1992), Data Quality Assessment – Statistical Methods for Practitioners (EPA 2006c), Guidance for Data Quality Assessment (EPA 2000), and Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA 2006a).

1.3.4.3 Sampling and Analysis Methods

A field investigation has been proposed to collect sediment data. Details pertaining to this effort are contained in Section 2 and will not be repeated here for brevity.

1.3.5 Step 4 – Define the Boundaries of the Study

In Step 4 of the DQO process, the target population of interest and spatial/temporal features pertinent for decision making should be identified. The most important activities in DQO Step 4 are as follows:

- Define the target population of interest

- Specify temporal or spatial boundaries and other practical constraints associated with sample/data collection.

1.3.5.1 Target Population

The target population consists of sediment in the vicinity of the Site. Sediment is considered mobile and can migrate significantly throughout the year. Migration is influenced by surface water flow in the waterway, tidal influences, and the physical characteristics of the sediment material. Fine grain materials are routinely subject to migration and may be transported some distance from sources. Therefore, a temporal aspect to the concentration of sediments in a

particular location may occur over time. Typically sediments have higher deposition rates near bends or where surface water flow is reduced.

1.3.5.2 Temporal or Spatial Boundaries

The Site horizontal boundaries are shown in Figure 3. Sampling will take place in the three offsite areas shown in the figure: intertidal, nearshore, and offshore. The vertical boundary extends to water depths of approximately 15 ft and sediment from 0 to 6 in.

1.3.6 Step 5 – Develop the Analytical Approach

Step 5 of the DQO process involves developing an analytic approach that will guide how to analyze the study results and draw conclusions from the data. It is the intention of this step to integrate the outputs from the previous four steps with the parameters developed in this step. The most important activities in DQO Step 5 are as follows:

- Specify the appropriate population parameters for making decisions
- Choose a workable action level and generate an "If … then … else" decision rule which involves it.

1.3.6.1 Population Parameters

The population parameter is defined as the value used in the decision statement to evaluate a decision point. The population parameter will be the maximum detected sediment concentration, which will be compared to updated screening criteria utilized in previous risk assessments: Screening Human Health Risk Assessment (Weston Solutions, Inc. 2003), Human Health Risk Assessment (CH2M HILL 2006), Environmental Screening Risk Assessment (Weston Solutions. Inc. 2003), and Baseline Environmental Risk Assessment (CH2M HILL 2006).

A population parameter will be determined for each chemical (e.g., selenium), in each exposure area (e.g., sediment near the Site), for each sample group (e.g., the upper 6 in. of sediment). In this example, the population parameter is selenium in sediment near the Site in the upper 6 in. of sediment. The population parameter for site comparisons will be the maximum detected concentration.

1.3.6.2 Action Level Decision Rule

The action levels for the Site are risk-based screening criteria developed during the Site risk assessments (Weston Solutions, Inc. 2003; CH2M HILL 2006). The action levels for each Five-Year Review contaminant of concern (COC) are provided in Table 3.

TABLE 3 ACTION LEVELS AND QUANTITATION LIMITS

Five-Year Review COC	Action Level	CRQL[a]
Ecological COCs[b]		
Selenium	1 mg/kg	2.5 mg/kg
Barium	48 mg/kg	5 mg/kg
Antimony	9.3 mg/kg	1 mg/kg
Arsenic	8.2 mg/kg	0.5 mg/kg
Beryllium	No screening level available	0.5 mg/kg
Cadmium	1.2 mg/kg	0.5 mg/kg
Lead	46.7 mg/kg	0.5 mg/kg
Mercury	0.15 mg/kg	0.1 mg/kg
Pentachlorophenol	17 µg/kg[c]	6.7 µg/kg
3,3-dichlorobenzidine	No screening level available	170 µg/kg
Human Health COCs[d]		
Copper	91 mg/kg[e]	1 mg/kg
Zinc	734 mg/kg	1 mg/kg
Aroclor-1242	0.15 µg/kg	33 µg/kg

a. http://www.epa.gov/superfund/programs/clp/target.htm. The lowest reporting limits available were used for metals and pesticides. In addition, the SIM method was selected for SVOCs in order to obtain reporting limits below the action level for pentachlorophenol.
b. National Oceanic and Atmospheric Administration (NOAA) 1999.
c. The screening level for pentachlorophenol has decreased from 360 to 17 µg/kg according to the most recent NOAA reference tables (2008).
d. Texas Commission on Environmental Quality, 2002.
e. The RfDo for copper, which was used to calculate the action level, has increased from 0.0371 mg/kg-day to 0.040 mg/kg-day, according to the most recent Texas Commission on Environmental Quality tables (2011); however, the original screening level is still protective.

NOTE: CRQL = Contract required quantitation limit.
mg/kg = Milligram per kilogram.
µg/kg = Micrograms per kilogram.

As observed by the comparisons in Table 3, two of the analyte sample quantitation limits, selenium and Aroclor 1242, are greater than their respective risk-based screening values. If these analytes are not detected in an exposure area and sample quantitation limits are greater than risk-based values, then they may be a source of potential risk underestimation.

The decision rule for the Site is as follows:

- If Site sediment concentrations are less than risk based criteria, then the additional site investigation activities or risk evaluations are generally not recommended

- Else, if Site sediment concentrations are greater than risk based criteria, then additional site investigation activities or risk evaluations may be recommended.

1.3.7 Step 6 – Specify the Performance or Acceptance Criteria

Step 6 of the DQO process specifies the tolerable limits on decision errors. Data are subject to various types of errors due (e.g., how samples were collected, how measurements were made, etc.). As a result, estimates or conclusions that are made from the collected data may deviate from what is actually true within the population. Therefore, there is a chance that an erroneous conclusion could be made or that the uncertainty in the estimates will exceed what is acceptable. The performance or acceptance criteria for collected data will be derived to minimize the possibility of either making erroneous conclusions or failing to keep uncertainty in estimates to within acceptable levels. Performance criteria and QA practices will guide the design of new data collection efforts. Acceptance criteria will guide the design of procedures to acquire and evaluate existing data.

The most important activities in DQO Step 6 are as follows:

- Recognizing the total study error and devising mitigation techniques to limit error
- Specify the decision rule as a statistical hypothesis test, examine consequences of making incorrect decisions from the test, and place acceptable limits on the likelihood of making decision errors.

1.3.7.1 Total Study Error

Even though unbiased data collection methods may be used, the resulting data will still be subject to random and systematic errors at different stages of the collection process (e.g., from field sample collection to sample analysis). The combination of these errors is called the "total study error" (or "total variability") associated with the collected data. There can be many contributors to total study error, but there are typically two main components, sampling error and measurement error.

Sampling Error

Sampling error, sometimes called statistical sampling error, is influenced by the inherent variability of the population over space and time, sample collection design, and the number of samples collected. It is usually impractical to measure the entire population space, and limited sampling may miss some features of the natural variation of the measurement of interest. Sampling design error occurs when the data collection design does not capture the complete variability within the population space, to the extent appropriate for making conclusions. Sampling error can lead to random error (i.e., random variability or imprecision) and systematic error (bias) in estimates of population parameters. In general, sampling error is much larger than measurement error and consequently needs a larger proportion of resources to control.

Measurement Error

Sometimes called physical sampling error, measurement error is influenced by imperfections in the measurement and analysis protocols. Random and systematic measurement errors are

introduced in the measurement process during physical sample collection, sample handling, sample preparation, sample analysis, data reduction, transmission, and storage.
The potential for measurement error will be mitigated by using accurate measurement techniques. As described in Sections 2.3 and 2.4, sampling techniques were selected to limit the measurement error, including the following:

- Sample collection procedures, sample processing, and field sample analysis protocols are fairly standard. This will ensure that the methodology remains consistent and limits the potential for measurement error.

- Field teams will be trained and will perform specific tasks (e.g., sample collection or processing) throughout the field sampling effort to limit the potential for measurement error.

- Potential for measurement error in the sample analysis will be limited by the analysis of QC samples (e.g., duplicates), as described in Section 2.11.

1.3.7.2 Statistical Hypothesis Testing and Decision Errors

Decision-making problems are often transformed into one or more statistical hypothesis tests that are applied to the collected data. Data analysts make assumptions on the underlying distribution of the parameters addressed by these hypothesis tests, in order to identify appropriate statistical procedures for performing the chosen statistical tests.

Due to the inherent uncertainty associated with the collected data, the results of statistical hypothesis tests cannot establish with certainty whether a given situation is true. There will be some likelihood that the outcome of the test will lead to an erroneous conclusion (i.e., a decision error).

When a decision needs to be made, there are typically two possible outcomes: either a given situation is true, or it is not. Although it is impossible to know whether an outcome is really true, data are collected and statistical hypothesis testing is performed to make an informed decision. In formulating the statistical hypothesis test, one of the two outcomes is labeled the "baseline condition" and is assumed to represent the *de facto*, true condition going into the test, and the other situation is labeled the "alternative condition." The baseline condition is retained until the information (data) from the sample indicates that it is highly unlikely to be true.

The statistical theory behind hypothesis testing allows for defining the probability of making decision errors. However, by specifying the hypothesis testing procedures during the design phase of the project, the performance or acceptance criteria can be specified.

There are four possible outcomes of a statistical hypothesis test. Two of the four outcomes may lead to no decision error; there is no decision error when the results of the test lead to correctly adopting the true condition, whether it is the baseline or the alternative condition. The remaining two outcomes represent the two possible decision errors. The first is a false rejection decision error, which occurs when the data lead to a decision that the baseline condition is false when, in

reality, it is true. The second is a false acceptance decision error, which occurs when the data are insufficient to change the belief that the baseline condition is true when, in reality, it is false. In the statistical language of hypothesis testing, the baseline condition is called the "null hypothesis" (H_o) and the alternative condition is called the "alternative hypothesis" (H_a). A false rejection decision error, or a Type I error, occurs when you reject the null hypothesis when it is actually true. The probability of this error occurring is called alpha (α) and is called the hypothesis test's level of significance. A false acceptance decision error, or a Type II error, occurs when you fail to reject the null hypothesis when it is actually false. The probability that this error will occur is called beta (β). Frequently, a false rejection decision error is the more severe decision error and, therefore, criteria placed on an acceptable value of alpha (α) are typically more stringent than for beta (β). Statisticians call the probability of rejecting the null hypothesis when it is actually false the statistical power of the hypothesis test. Statistical power is a measure of how likely the collected data will allow you to make the correct conclusion that the alternative condition is true rather than the default baseline condition and is a key concept in determining DQOs for decision-making problems. Note that statistical power represents the probability of "true rejection" (i.e., the opposite of false acceptance) and, therefore, is equal to 1-β.

Decision errors can never be totally eliminated when performing a statistical hypothesis test. However, the primary aim of this step is to arrive at the upper limits on the probabilities of each of these two types of decision errors that the planning team finds acceptable. Analytes that are detected in sediment will be subject to a screening level evaluation to determine whether Site concentrations exceed screening levels. To perform this evaluation the arithmetic mean and maximum concentrations will be compared against screening levels. The null hypothesis is that the arithmetic mean concentration does not exceed the screening level and the alternative hypothesis is that the mean concentration of a contaminant does exceed the screening level as follows:

H_o = Mean Media Analyte Concentration <= Screening Level
H_a = Mean Media Analyte Concentration > Screening Level

For the statistical evaluations conducted for the Site, the probability of a Type I error occurring was established at 5 percent and a Type II error was established at 20 percent.

1.3.8 Step 7 – Develop the Plan for Obtaining Data

In the Steps 1 through 6 of the DQO process, performance or acceptance criteria were developed. The goal of Step 7 is to develop a resource-effective sampling design for collecting and measuring environmental samples, or for generating other types of information needed to address the principal study question. In addition, this sampling design will lead to data that will achieve the performance and acceptance criteria.

The most important activity in DQO Step 7 is as follows:

- Use the information from Steps 1 through 6 of the DQO process to identify a sampling and analysis design that will answer the principal study question and achieve the performance or acceptance criteria.

1.3.8.1 Visual Sample Plan

A simple random sampling approach using Visual Sample Plan (VSP) Version 6.1b was taken to select sample locations. Because selenium was a COC for ecological receptors, this analyte was used in VSP. The data for selenium from composite sediment samples are presented in Table 4. During the evaluation, VSP indicated that sample I-SD was a potential outlier; therefore, this sample was removed from consideration. Once the outlier was removed, VSP indicated that there were no additional outliers. The average selenium concentration was 3.38 mg/kg and standard deviation was 0.77 mg/kg. These statistics were used to determine the appropriate number of random sample locations using VSP. The selenium dataset (minus the outlier) was determined to have a normal distribution by VSP (Appendix A).

TABLE 4 HISTORICAL SAMPLE DATA

Station ID	Selenium Concentration (mg/kg)	Northing	Easting	Water Depth (ft)	Analyses
A-SD	4.66	13933044.74	3596047.16	8-9	VOCs, SVOCs, Metals, Pesticides/PCBs
B-SD	3.15	13932763.24	3596008.76	8-9	VOCs, SVOCs, Metals, Pesticides/PCBs
C-SD	3.69	13932209.76	3595899.878	15	VOCs, SVOCs, Metals, Pesticides/PCBs
D-SD	2.22	13932411.64	3596033.712	10	VOCs, SVOCs, Metals, Pesticides/PCBs
E-SD	3.85	13932368.54	3595772.85	3-4	VOCs, SVOCs, Metals, Pesticides/PCBs
F-SD	2.41	13932613.53	3596128.984	8	VOCs, SVOCs, Metals, Pesticides/PCBs
G-SD	2.96	13932014.68	3595629.942	8-10	VOCs, SVOCs, Metals, Pesticides/PCBs
H-SD	3.87	13932127.59	3595471.329	4	VOCs, SVOCs, Metals, Pesticides/PCBs
H-SD	3.63	13932127.59	3595471.329	4	VOCs, SVOCs, Metals, Pesticides/PCBs
I-SD	0.549			2	VOCs, SVOCs, Metals, Pesticides/PCBs

VSP provides a statistical output based on the comparison of two populations (e.g., Site versus background) or based on the comparison of a population against a fixed value (e.g., screening level or threshold value). Because the Site is potentially impacted, it was determined that comparing the Site population against a fixed value (i.e., Ecological Screening Level of 1 mg/kg) was appropriate. This is the same approach that was used at two EPA Region 6 Superfund Sites (i.e., Petro-Chemical Systems, Inc. Superfund Site and Many Diversified Interests Superfund Site).

VSP used a simple random sampling approach to discern a difference of 1 standard deviation between a mean selenium concentration and its screening level value; this “gray region” is the concentration range in which site decisions cannot be made at the specified Type I or Type II error rates. The smaller the tolerable gray region, the greater the numbers of samples that are required. A gray region of less than one standard deviation is more difficult to resolve unless a larger number of measurements are available and relative differences of more than three standard deviations are easier to resolve, but may lack statistical robustness (EPA 2002b, Pacific Northwest National Laboratory 2010). Therefore, the use of 1 standard deviation for the gray region was considered appropriate for the Site.

A simple random sampling approach using VSP (Pacific Northwest National Laboratory 2010) was used to establish the randomized sample locations. VSP determined that eight random samples were necessary to test the null hypothesis. The random samples are indicated by circles in the VSP output (Appendix A).

1.3.8.2 Biased or Judgmental Samples

The sediment sampling approach also contains biased or judgmental samples. Sediment samples will be biased toward impacted areas using historical site evidence (e.g., photographic documentation, previous sampling results, etc.). This does introduce bias and potential sampling error; however, the bias is likely to increase the sample concentration, which will likely result in a more protective evaluation. Sampling error that is considered more protective of human health or the environment is acceptable for this evaluation.

Eight composite samples were collected during the supplemental remedial investigation (CH2M HILL, 2006). Generally, the center of the composite samples was selected as the location for the judgmental sampling locations (i.e., indicated by a cross in Appendix A). These sample locations will be resampled to evaluate temporal differences. In addition, two judgmental samples will be collected to bolster the dataset. Sample locations may be adjusted based on field conditions at the time of sampling (e.g., barge locations); the relocation of sample locations will be conducted at the direction of EPA. Additional details pertaining to the sampling and analysis design are detailed throughout this document and will not be repeated here for brevity.

1.3.8.3 Sampling Summary

The eight random sample locations, eight judgmental sampling locations to evaluate previous sampling locations, and two judgmental sample locations will comprise the dataset. Two field duplicate samples will also be collected for QA/QC.

1.4 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

A well-defined QA/QC process is integral to the generation of analytical data of known and documented quality. The QC process includes those activities required during data collection to produce data of sufficient quality to support the decisions that will be made based on the data (e.g., decisions to be made prior to, during, and after site remedial actions) (EPA 2006a). After environmental data are collected, QA activities focus on evaluating the quality of the data in order to determine the data usability with respect to support for remedial or enforcement decisions. Table 5 presents the acceptance criteria for definitive onsite and offsite laboratory data for chemical analyses of investigation samples only.

1.4.1 Data Categories

In order to produce data suitable for decision-making, an appropriate analytical technique must be selected. The EPA Superfund program has developed two descriptive categories of analytical techniques: (1) field-based techniques, and (2) fixed-laboratory techniques. The type of data generated depends on the qualitative and quantitative DQOs developed for a project. Regardless

of whether the data were analyzed utilizing field or laboratory techniques, they must be of adequate quality for the decision-making process for which they were collected. For this project, data from both types of techniques will be collected. Section 2 discusses the methods that will be used to analyze the samples. Both field-based and definitive analytical data will be used to support decisions made for this project.

Rigorous analytical methods (e.g., EPA CLP methods) are used to generate analyte-specific, definitive data. The definitive quality of the data is assured by: (1) using SOPs and QC processes during data collection; (2) documented control and traceability of reference standards, calibrations, and instrument performance; and (3) acceptable performance of field and laboratory QC procedures within the defined limits established for these procedures.

TABLE 5 QUALITY ASSURANCE INDICATOR CRITERIA

Indicator Parameter	Analytical Parameter	QC Sample	Acceptance Criteria for Laboratory Analysis
Accuracy (percent recovery)	VOCs, SVOCs, pesticides, polychlorinated biphenyls	MS, MSD Blanks	50 to 150 percent recovery Less than CRQL
	Target Analyte List Metals and cyanide	MS LCS Blanks[(a)]	75 to 125 percent recovery 80 to 120 percent recovery Less than CRQL
Precision	VOCs, SVOCs, pesticides, polychlorinated biphenyls	MS, MSD Field duplicates	30 percent RPD 50 percent RPD
	Metals	MS, MD Field duplicates	20 percent RPD (aqueous) 35 percent RPD (solid) 50 percent RPD
Sensitivity (quantitation limits)	Analytical tests	MS, MD, MSD Field duplicates	Not applicable
Completeness	The objective for data completeness is 90 percent.		
Representativeness	The sampling network analytical methods for this site are designed to provide data that are representative of site conditions.		
Comparability	The use of standard published sampling and analytical methods, and the use of QC samples, will ensure data of known quality. These data can be compared to other data of known quality.		

a. May include method blanks, reagent blanks, instrument blanks, calibration blanks, and other blanks collected in the field (i.e., field blanks).

NOTE: CRQL = Contract-required quantitation limit.
LCS = Laboratory control spike.
MD = Matrix duplicate.
MS = Matrix spike.
MSD = Matrix spike duplicate.
RPD = Relative percent difference.
SVOC = Semivolatile organic compound.
VOC = Volatile organic compound.

The majority of the fixed-laboratory analysis for samples collected during the Five-Year Review sampling event will be conducted by the EPA Region 6 Laboratory and/or an EPA-designated CLP laboratory. It is not anticipated that a subcontracted, non-CLP laboratory will be used for the Five-Year Review field effort.

1.4.2 Measurement Quality Objectives

Soil analytical results will be evaluated in accordance with PARCC parameters to document the quality of the data and to ensure that the data are of sufficient quality to meet the project objectives. Of these PARCC parameters, precision and accuracy will be evaluated quantitatively by collecting the QC check samples listed in Table 5.

The sections below describe each of the PARCC parameters and how they will be assessed within this project.

1.4.2.1 Precision

Precision is the degree of mutual agreement between individual measurements of the same property under similar conditions. Usually, combined field and laboratory precision is evaluated by collecting and analyzing field duplicates and then calculating the variance between the samples, typically as a RPD.

RPD is calculated as follows:

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

where

A = First duplicate concentration.
B = Second duplicate concentration.

Field sampling precision is evaluated by analyzing field duplicate samples. For every 10 samples collected, one soil blind duplicate sample will be collected.

Laboratory analytical precision is evaluated by analyzing laboratory duplicates (also called MDs) or MS and MSDs. For this project, MS/MSD and MS/MD samples will be generated for analytes. The results of the analysis of each MS/MSD or MS/MD pair will be used to calculate the RPD as a measure of laboratory precision. The RPD acceptance criteria are listed in Table 5.

1.4.2.2 Accuracy

A program of sample spiking will be conducted to evaluate laboratory accuracy. This program includes analysis of the MS and MSD samples, LCSs or blank spikes, surrogate standards, and method blanks. MS and MSD samples will be prepared and analyzed at a frequency of 5 percent for sediment samples. LCSs or blank spikes are also analyzed at a frequency of 5 percent. Surrogate standards, where available, are added to every sample analyzed for inorganic constituents. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy.

$$\text{Percent Recovery} = \frac{S - C}{T} \times 100\%$$

where

S = Measured spike sample concentration
C = Sample concentration
T = True or actual concentration of the spike.

The objective for accuracy of field measurements is to achieve and maintain factory specifications for the field equipment.

1.4.2.3 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that they are intended to represent. For this project, representative data will be obtained through careful selection of sampling locations and analytical parameters. Representative data will also be obtained through proper collection and handling of samples to avoid interference and minimize contamination.

Representativeness of data will also be ensured through the consistent application of established field and laboratory procedures. Field blanks (if appropriate) and laboratory blank samples will be evaluated for the presence of contaminants to aid in evaluating the representativeness of sample results. Data determined to be non-representative, by comparison with existing data, will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

1.4.2.4 Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this SAP, and when none of the QC criteria that affect data usability are exceeded. When data validation is completed, the percent completeness value will be calculated by dividing the number of useable sample results by the total number of sample results planned for this investigation.

As discussed further in Section 4.2, completeness will also be evaluated as part of the data quality assessment process (EPA 2006c, 2006d). This evaluation will help determine whether limitations are associated with the decisions to be made based on the data collected.

1.4.2.5 Comparability

Comparability expresses the confidence with which one data set can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data. Standard EPA analytical methods and QC will be used to support the comparability of analytical results with those obtained in other testing. Calibrations will be performed in accordance with EPA or manufacturer's specifications and will be checked with the frequency specified in the EPA CLP SOW(s).

1.4.3 Detection and Quantitation Limits

The analytical parameters and their quantitation limits for use on this project are determined under the EPA CLP SOW(s). The CRDL is the minimum concentration of an analyte that can be reliably distinguished from background noise for a specific analytical method. The quantitation limit represents the lowest concentration of an analyte that can be accurately and reproducibly quantified in a sample matrix. CRQLs are contractually specified maximum quantitation limits for specific analytical methods and sample matrices, i.e., soil or water, and are typically several times the method detection limit to allow for matrix effects.

For this project, analytical methods have been selected so that the CRQL for each target analyte is below the applicable regulatory screening criteria, wherever practical. For this project, samples results will be reported as estimated values if concentrations are less than CRQLs but greater than CRDLs. The CRDL for each analyte will be listed as the detection limit in the laboratory's electronic data deliverable.

1.5 SPECIAL TRAINING AND CERTIFICATION

This section outlines the training and certification required to complete the activities described in this SAP. The following sections describe the requirements for the EA team and subcontractor personnel working onsite.

1.5.1 Health and Safety Training

EA field team personnel who work at hazardous waste project sites are required to meet the Occupational Safety and Health Administration training requirements defined in 29 Code of Federal Regulations 1910.120(e). These requirements include: (1) 40 hours of formal off-site instruction, (2) a minimum of 3 days of actual onsite field experience under the supervision of a trained and experienced field supervisor, and (3) 8 hours of annual refresher training. Field personnel who directly supervise employees engaged in hazardous waste operations also receive at least 8 additional hours of specialized supervisor training. Copies of the field team's health and safety training records, including course completion certifications for the initial and refresher health and safety training, and specialized supervisor training are maintained in project files.

For more health and safety details, see EA's site-specific Health and Safety Plan (EA 2011b).

1.5.2 Subcontractor Training

Employees of associate and professional services firms and technical services subcontractors will attend a safety briefing and complete the Safety Meeting Sign-Off Sheet before they conduct onsite work (EA 2011b). This briefing is conducted by the EA Site Health and Safety Officer or other qualified person.

Subcontractors are responsible for conducting their own safety briefings; EA personnel may audit these briefings. Alternatively, the subcontractors may elect to attend the EA safety briefings.

1.6 DOCUMENTS AND RECORDS

The following sections discuss the requirements for documenting field activities and for preparing laboratory data packages. This section also describes reports that will be generated as a result of this project.

1.6.1 Field Documentation

Field personnel will use permanently bound field logbooks with sequentially numbered pages to record and document field activities and will follow SOP No. 016 for completing field logbooks (Appendix B). The logbook will list the contract name and number; site name; and names of subcontractors, service client, and Project Manager. At a minimum, the following information will be recorded in the field logbook:

- Name and affiliation of onsite personnel or visitors
- Weather conditions during the field activity
- Summary of daily activities and significant events
- Notes of conversations with coordinating officials
- References to other field logbooks or forms that contain specific information (Appendix C)
- Discussions of problems encountered and their resolution
- Discussions of deviations from the SAP or other governing documents
- Description of photographs taken.

1.6.2 Laboratory Documentation

This section describes the data reporting requirements for EA field personnel and laboratories (e.g., EPA CLP laboratories, EPA Region 6 laboratory, or subcontracted non-CLP laboratories) that submit field and laboratory measurement data under the EPA Region 6 RAC II program. EA will require fixed offsite non-CLP laboratories to prepare and submit data packages in accordance with the EPA CLP protocols (2007b; 2008; 2010a,b,c; 2011b) for hardcopy and electronic data deliverable format of lead data. Data packages will include applicable documentation for independent validation of data and verification of the DQOs. The following documentation will be required for full data validation, if applicable:

- Case narratives, which will describe QC non-conformances that are encountered during the analysis of samples in addition to corrective actions that are taken:

- — Statement of samples received
- — Description of deviations from the specified analytical method
- — Explanations of data qualifiers that are applied to the data
- — Other significant problems that were encountered during analysis.

- Tables that cross-reference field and laboratory sample numbers

- Chain-of-custody forms, which pertain to each sample delivery group or sample batch that is analyzed

- Laboratory reports, which must show traceability to the sample analyzed and must contain specified information:

  - — Project identification

  - — Field sample number

  - — Laboratory sample number

  - — Sample matrix description

  - — Dates and times of sample collection, receipt at the laboratory, preparation, and analysis

  - — Description of analytical method and reference citation

  - — Results of individual parameters, with concentration units, including second column results, second detector results, and other confirmatory results, where appropriate

  - — Quantitation limits achieved

  - — Dilution or concentration factors

- Data summary forms and QC summary forms showing analytical results, if applicable:

  - — Samples
  - — Surrogates
  - — Blanks
  - — Field QC samples
  - — LCS
  - — Initial and continuing calibrations
  - — Other QC samples

- Laboratory control charts

  - Raw data
  - Instrument printouts
  - Laboratory bench sheets for preparation of samples

- Method detection limit study results.

EA's Project Manager, in cooperation with the QA Officer, will define site-specific requirements for data reporting. Requests for analytical services define these requirements, the turnaround time for receipt of the data deliverables specified, and requirements for retaining samples and laboratory records. Laboratory QA managers are responsible for ensuring that laboratory data reporting requirements in this SAP are met.

1.6.3 Full Data Package

When a full data package is required, the laboratory will prepare data packages in accordance with the instructions provided in the EPA CLP SOWs (2007b; 2008; 2010a,b,c; 2011b). Full data packages will contain the information from the summary data package and associated raw data. Full data packages are due to EA within 35 days after the last sample in the sample delivery group is received. Unless otherwise requested, the subcontractor will deliver one copy of the full data package.

1.6.4 Reports Generated

Following the completion of the Five-Year Review field program and receipt of validated data, EA will prepare a Five-Year Review Report.

The specific requirements and elements of this report are discussed in detail in the EPA-approved Five-Year Review Work Plan (EA 2011a).

2. DATA GENERATION AND ACQUISITION

This section describes the requirements for the following:

- Sampling process design (Section 2.1)
- Consent for property access (Section 2.2)
- Sampling methodology (Section 2.3)
- Sample processing (Section 2.4)
- Decontamination (Section 2.5)
- Management of investigation-derived waste (Section 2.6)
- Sample designation (Section 2.7)
- Sample container, volume, preservation, and holding time requirements (Section 2.8)
- Sample handling and custody (Section 2.9)
- Analytical methods requirements (Section 2.10)
- QC requirements (Section 2.11)
- Instrument calibration and frequency (Section 2.12)
- Requirements for inspection and acceptance of supplies and consumables (Section 2.13)
- Data acquisition requirements (Section 2.14
- Data management (Section 2.15).

2.1 SAMPLING PROCESS DESIGN

For the activities associated with this Task Order and SAP, main elements of the sampling design include the numbers and types of samples to be collected, sampling locations, sampling frequencies, and sample matrices.

As stated in the DQOs (Section 1.3), the following principle study question was formulated for the Five-Year Review:

Do sediment concentrations present a potential unacceptable risk to human health and ecological receptors?

The primary objective of the sampling design is to collect data of sufficient quantity and quality to resolve the study question and support remedy evaluation. The purpose of the Five-Year Review is to determine whether the remedy at a site is protective of human health and the environment, as follows:

Determine whether sediment concentrations present a potential unacceptable risk to human health and ecological receptors.

The goal is to develop the minimum amount of data necessary to ensure that the site remedy described in the Record of Decision (EPA 2007a) is still protective of human health and ecological receptors. To achieve this goal, sediment will be sampled during the Five-Year Review at the Site. Table 4 summarizes the historical sediment data that were used during the

VSP process to determine the number and location of sediment samples for the Five-Year Review field program.

2.2 CONSENT FOR PROPERTY ACCESS

It is not anticipated that access to private property will be required for the Five-Year Review field effort. However, if access to private property becomes necessary, EA will attempt to obtain Consent for Property Access Agreements from the private property owners that have been identified for investigation under the Five-Year Review. If EA is unsuccessful in our attempts to obtain Consent for Property Access Agreements, EPA will request access from private property owners. For properties where the property owner cannot be identified and/or the property owner is not responsive, other properties may be identified for characterization as appropriate.

2.3 SAMPLING METHODOLOGY

This section describes the procedures for sample collection. Table 6 lists the SOPs that will be implemented during this field program. The SOPs are provided in Appendix B.

Sample collection and handling procedures for samples that will be analyzed using the CLP will follow CLP protocols in accordance with EPA's *Contract Laboratory Program Guidance for Field Samplers* (EPA 2011b).

During sample collection, preparation, and field analysis, chain-of-custody will be maintained and documented as required in Section 2.9. Sample locations will be documented photographically and sketched in the field logbook; an accompanying photograph log will be completed in the field logbook (Section 1.6.1).

TABLE 6 STANDARD OPERATING PROCEDURES

SOP Number	SOP Title
001	Labels
002	Chain-of-Custody Form
003	Subsurface/Utility Clearance
004	Sample Packing and Shipping
005	Field Decontamination
016	Surface Water, Groundwater, and Soil/Sediment Logbooks
021	Sediment Sampling
022	Sediment and Benthic Sampling
039	Sample Preservation and Container Requirements
042	Disposal of Investigation-Derived Material

2.3.1 Sediment Sampling

During the Five-Year Review field program, EA will collect surface sediment samples in accordance with SOP No. 021 (Appendix B) as projected in Table 4. A virtual random-start systematic sampling approach using VSP (Pacific Northwest National Laboratory 2010) was used to establish the random sample locations. Figure 3 illustrates the proposed sediment sampling locations associated with the intertidal, nearshore, and offshore areas of the Site. Biased or judgmental sample are also depicted on the figure, they will be taken from approximately the same locations as the samples for the 2006 Supplemental Remedial Investigation Report (CH2M HILL 2006).

Sediment samples will be collected from a subcontracted boat using a ponar dredge sampler, at water depths up to 15 ft. If there is insufficient sediment material from the initial grab, multiple grab samples may be collected from that location to obtain sufficient volume. However, in order to minimize the loss of VOCs, the VOC sample will be collected from the initial grab, prior to the collection of additional grab samples. In addition, separate grab samples will not be homogenized to prevent loss of organic compounds. Each sediment sampling location will be surveyed using a hand-held global positioning system device. Samples will be analyzed for VOCs, SVOCs, metals, and polychlorinated biphenyls.

During the sediment sampling, descriptive information from each location, including depth of surface water, sediment color, general sediment moisture content, sediment grain size, sediment recovery, number of sample locations, number of grab samples collected for each composite.

2.4 SAMPLE PROCESSING

Samples for fixed laboratory analysis will be processed and packaged in accordance with the *Contract Laboratory Program Guidance for Field Samplers* (EPA 2011b).

2.5 DECONTAMINATION

Re-usable field equipment utilized during the Five-Year Review will be decontaminated prior to and after use in accordance with SOP No. 005 (Appendix B).

2.6 MANAGEMENT OF INVESTIGATION-DERIVED WASTE

EA will incorporate best management practices of green remediation as it relates to the management of investigative-derived. Investigative-derived waste will be addressed at the direction of EPA. Personal protective equipment and any disposable sampling equipment will be collected and disposed of in accordance with local, federal, and state regulations.

2.7 SAMPLE DESIGNATION

Each sampling location will be designated with a unique alphanumeric designation according to the following sample classifications:

- ***Sediment Sample Designation*—**Sediment sample designation will include three fields that are separated by dashes; e.g., D-SD5-12.

  — The first field, "D," identifies the sample station.

  — The second field, "SD5," identifies the sample matrix as sediment and the sampling event as the Five-Year Review.

  — The third field, "12," represents the sample identifier.

- ***Field Duplicate Sample Designation*—**Field duplicate samples will be identified by adding a "D" to the end of the sample designations described above; e.g., B-SD5-12D and F-SD5-04D.

- ***MS/MSD Sample Designation (for organic analysis)***—MS and MSD samples will be identified by adding an "MSD" to the end of the sample designations described above; e.g., B-SD5-12MSD.

- ***MS/MD Sample Designation (for inorganic analysis)***—MS and MD samples will be identified by adding an "MD" to the end of the sample designations described above; e.g., B-SD5-12MD.

- ***Field, Trip, and Equipment Rinsate Blank Sample Designation***—Trip and equipment blank samples will be identified sequentially beginning with TB-1, FB-1, and ER-1, respectively.

2.8 SAMPLE CONTAINER, VOLUME, PRESERVATION, AND HOLDING TIME REQUIREMENTS

Table 7 specifies the required sample volume, container type, preservation technique, and holding time for each analysis that is to be conducted during each phase of sampling. Required containers, preservation techniques, and holding times for field QC samples, i.e., field duplicates, will be the same as for investigative samples, but may require additional volumes as described in Section 2.11.

2.9 SAMPLE HANDLING AND CUSTODY

Each sample collected by the EA field team will be traceable from the point of collection through analysis and final disposition to ensure sample integrity. Sample integrity helps to ensure the legal defensibility of the analytical data and subsequent conclusions. Sample handling will

follow CLP protocols as required in EPA's *Contract Laboratory Program Guidance for Field Samplers* (EPA 2011b).

The EA field team will use EPA's data management system known as "Forms II Lite" to generate chain-of-custody records in the field. Applicable copies of generated Forms II Lite files will be delivered to EPA data management personnel as required by CLP protocols.

2.10 ANALYTICAL METHODS REQUIREMENTS

The source of analytical services to be provided will be determined in part by DQOs and the intended use of the resulting data. EA will use EPA-approved methods for laboratory analyses of the samples.

EA will follow the analytical services request procedures that are outlined EA's Analytical Services Delivery Plan (EA 2005b). If an analytical system fails, the QA officer will be notified, and corrective action will be taken. In general, corrective actions will include stopping the analysis, examining instrument performance and sample preparation information, and determining the need to re-prepare and reanalyze the samples.

Laboratories that are subcontracted by EA or EPA will conduct definitive laboratory analysis of samples. Table 7 lists the laboratory analytical methods for this project. In cases, appropriate methods of sample preparation, cleanup, and analyses are based on specific analytical parameters of interest, sample matrices, and required quantitation limits. The following sections briefly discuss each analytical method and required modifications for definitive investigative analyses.

TABLE 7 REQUIRED VOLUME, CONTAINERS, PRESERVATIVES, AND HOLDING TIMES

Parameter	Method	Volume and Container	Preservatives	Holding Time [a]
Investigative Soil or Sediment Samples				
Target Analyte List metals (including mercury and cyanide)	CLP ISM01.3[b]	One 8-ounce glass jar with Teflon™-lined cap	Store at 4±2°C	180 days for analysis (28 days Hg)
VOCs	CLP SOM01.2[c]	One 4-ounce glass jar with Teflon™-lined cap (moisture content)	Methanol; Store at 4±2°C	48 hours
SVOCs	CLP SOM01.2[c]	One 8-ounce glass jar with Teflon™-lined cap	Store at 4±2°C	7 days
Pesticides/ polychlorinated biphenyls	CLP SOM01.2[c]	One 8-ounce glass jar with Teflon™-lined cap	Store at 4±2°C	14 days

a. Holding time is measured from the time of sample collection to the time of sample extraction and analysis.
b. EPA 2010a,c; Appendix D
c. EPA 2007b; Appendix D.

NOTE: °C = Degrees Centigrade.

The source of analytical services to be provided will be determined in part by DQOs and the intended use of the resulting data. EA will use EPA-approved methods for laboratory analyses of the samples.

2.10.1 Field Analytical Methods

The use of field analytical methods is not anticipated during this sampling event.

2.10.2 Fixed Laboratory Analytical Methods

Fixed-laboratory analyses will be conducted by EPA Region 6, a designated CLP laboratory, or a subcontracted non-CLP laboratory. Samples submitted to the analytical laboratory will be analyzed in accordance with CLP SOWs SOM01.2 (EPA 2007b) and ISM01.3 (EPA 2010a,c). Modifications to analytical methods that may be required to manage atypical matrices or to achieve low quantitation limits are not anticipated. Decisions regarding the use and type of method modifications will be made during the procurement of laboratories, as different laboratories have equipment and SOPs that generate varying quantitation limits.

The EPA quick reference fact sheets that list the CRDLs for CLP SOWs SOM01.2 and ISM01.3 are provided in Appendix D.

2.11 QUALITY CONTROL REQUIREMENTS

Various field and laboratory QC samples and measurements will be used to verify that analytical data meet the QA objectives. Field QC samples and measurements will be collected to assess the influence of sampling activities and measurements on data quality. Similarly, laboratory QC samples will be used to assess how the laboratory's analytical program influences data quality. This section describes the QC samples that are to be analyzed during the site sampling activities for: (1) each field and laboratory environmental measurement method, and (2) each sample matrix type. Table 5 shows the acceptance criteria for each type of QC sample, and Table 8 presents the frequency of QC samples to be collected at the site.

2.11.1 Field Quality Control Requirements

Field QC samples will be collected and analyzed to assess the quality of data that are generated by sampling activities. These samples will include laboratory QC samples collected in the field, field duplicates, equipment rinsates, MS/MDs, and temperature blanks. QC samples collected in the field for fixed-laboratory analysis are presented in Table 8.

Field duplicates are independent samples that are collected as close as possible, in space and time, to the original investigative sample. Field duplicates can measure the influence of sampling and field procedures on the precision of an environmental measurement. They can also provide information on the heterogeneity of a sampling location. Field duplicates will be collected at a frequency of 1 for every 10 investigative samples, as listed in Table 8. Immediately following collection of the original sample, the field duplicates are collected using the same collection method.

Field blanks are collected to assess: (1) impact from ambient air conditions during sample collection; (2) cross-contamination during sample collection, preservation, and shipment, as well as in the laboratory; and (3) cleanliness of the sample containers and preservatives. Field blank samples consist of sample containers filled with analytically-certified, organic-free water. Field blank samples are typically collected for VOC analysis at a frequency of one field blank for each day of sampling activities (specifically for VOC analysis). If contaminant is present in the blank samples above the method detection limit, the result for associated field samples that contain the same contaminant will be qualified as potentially not detected if the concentration of the field sample is less than five times the concentration found in the blank.

Equipment rinsate blanks are collected when nondedicated or nondisposable sampling equipment is used to collect samples and put the samples into containers. These blanks assess the cleanliness of the sampling equipment and the effectiveness of equipment decontamination. Equipment rinsate blanks are collected by pouring analyte-free water over the decontaminated surfaces of sampling equipment that contacts sampling media. Equipment rinsate blanks are collected after sampling equipment has been decontaminated, but before the equipment is reused for sampling. If nondedicated or nondisposable equipment is used, equipment rinsate blanks will be collected in accordance with the frequency listed in Table 8.

TABLE 8 FREQUENCY OF FIELD QUALITY CONTROL SAMPLES

Field QC Sample	Frequency[a]
Trip blank	1 per cooler containing aqueous samples for VOC analysis
Field blank	1 per day, if site conditions render this sample necessary
Field duplicate	1 per 10 samples
MS/MD[b] (inorganics)	1 per 20 samples (or per Region 6 EPA Laboratory requirements)
MS/MSD[b] (organics)	1 per 20 samples (or per Region 6 EPA Laboratory requirements)
Temperature blank	1 per cooler

a. The QC sample collection frequency applies to samples collected for fixed-laboratory analysis (EPA 2011b).
b. MS, MSD, and MD analyses are technically not field QC samples; however, they generally require that the field personnel collect additional volumes of samples and are, therefore, included on this table for easy reference.

MS/MSD samples are laboratory QC samples that are collected for organic methods; MS/MD samples are collected for inorganic methods. MS/MSDs require double or triple the normal sample volume, depending on analytical laboratory specifications, and MS/MDs require double the normal sample volume. In the laboratory, MS/MSD and MS/MD samples are split and spiked with known amounts of analytes. Analytical results for MS/MSD and MS/MD samples are used to measure the precision and accuracy of the laboratory's organic and inorganic analytical programs, respectively. Each of these QC samples will be collected and analyzed at a frequency of 1 for every 20 investigative samples for CLP laboratories or subcontract non-CLP laboratories, or in accordance with the requirements of the EPA Region 6 Laboratory.
Trip blanks are analyzed for VOCs only. VOC samples are susceptible to contamination by diffusion of organic contaminants through the Teflon-lined septum of the sample vial; therefore, a VOC trip blank will be analyzed to monitor for possible sample contamination. Also, the trip blank will screen for possible contamination of VOC samples during handling and shipment

from the field to the laboratory. The trip blanks will be provided by the laboratory and placed in each cooler that contains VOC samples.

Temperature blanks are containers of deionized or distilled water that are placed in each cooler shipped to the laboratory. Their purpose is to provide a container to test the temperature of the samples in the respective cooler.

2.11.2 Laboratory Quality Control Requirements

Laboratories that perform analytical work under this project must adhere to a QA program that is used to monitor and control laboratory QC activities. Each laboratory must have a written QA manual that describes the QA program in detail. The laboratory QA manager is responsible for ensuring that laboratory internal QC checks are conducted in accordance with EPA methods and protocols, the laboratory's QA manual, and the requirements of this SAP.

Many of the laboratory QC procedures and requirements are described in EPA-approved analytical methods, laboratory method SOPs, and method guidance documents. The EPA methods specify the preparation and analysis of QC samples, and may include, but are not limited to, the following types: (1) LCSs; (2) method blanks; (3) MS, MSD, and MD samples; (4) surrogate spikes; and (5) standard reference materials or independent check standards. The following subsections discuss the QC checks that will be required for this project. The EPA multimedia, multi-concentration analytical service fact sheets for CLP SOWs SOM01.2 and ISM01.3 are provided in Appendix D.

2.11.2.1 Laboratory Control Sample

LCSs are thoroughly characterized, laboratory-generated samples that are used to monitor the laboratory's day-to-day performance of analytical methods. The results of LCS analyses are compared to well-defined laboratory control limits to determine whether the laboratory system is in control for the particular method. If the system is not in control, corrective action will be implemented. Appropriate corrective actions will include: (1) stopping the analysis, (2) examining instrument performance or sample preparation and analysis information, and (3) determining whether samples should be re-prepared or reanalyzed.

2.11.2.2 Method Blanks

Method blanks, which are also known as preparation blanks, are analyzed to assess the level of background interference or contamination in the analytical system and the level that may lead to elevated concentration levels or false-positive data. Method blanks will be required for laboratory analyses and will be prepared and analyzed at a frequency of one method blank per every 20 samples or one method blank per batch, if the batches consist of fewer than 20 samples. A method blank consists of reagents that are specific to the analytical method and are carried through every aspect of the analytical procedure, including sample preparation, cleanup, and analysis. The results of the method blank analysis will be evaluated in conjunction with other QC information to determine the acceptability of the data generated for that batch of samples. Ideally, the concentration of a target analyte in the method blank will be below the reporting

limit for that analyte. For some common laboratory contaminants, a higher concentration may be allowed.
If the method blank for analysis is beyond control limits, the source of contamination must be investigated, and appropriate corrective action must be taken and documented. This investigation includes an evaluation of the data to determine the extent of the contamination and its effect on sampling results. If a method blank is within control limits but analysis indicates a concentration of analytes that is above the reporting limit, an investigation should be conducted to determine whether corrective action could eliminate an ongoing source of target analytes.
For organic and inorganic analyses, the concentration of target analytes in the method blank must be below the reporting limit for that analyte for the blank to be considered acceptable. An exception may be made for common laboratory contaminants (i.e., methylene chloride, acetone, 2-butanone, and phthalate esters) that may be present in the blank at up to five times the reporting limit. These compounds are frequently detected at low levels in method blanks from materials that are used to collect, prepare, and analyze samples for organic parameters.

2.11.2.3 Matrix Spikes

MSs and MSDs are aliquots of an environmental sample for organic analysis to which known concentrations of target analytes and compounds have been added. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis. If there are many target analytes, they will be divided into two to three spike standard solutions. Each spike standard solution will be used alternately. The MS, in addition to an unspiked aliquot, will be taken through the entire analytical procedure, and the recovery of the analytes will be calculated. Results will be expressed in terms of percent recoveries and RPD. The percent recoveries of the target analytes and compounds are calculated and used to determine the effects of the matrix on the precision and accuracy of the method. The RPD between the MS and MSD results is used to evaluate method precision.

The MS/MSD is divided into three separate aliquots, two of which are spiked with known concentrations of target analytes. The two spiked aliquots, in addition to an unspiked sample aliquot, are analyzed separately, and the results are compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results will be expressed as RPD and percent recovery and compared to control limits that have been established for each analyte.
If results fall outside control limits, corrective action will be performed.

2.11.2.4 Laboratory (Matrix) Duplicates

MDs, which are also called laboratory duplicates, are prepared and analyzed for inorganic analyses to assess method precision. Two aliquots of sample material are taken from the sample and processed simultaneously without adding spiking compounds. The MD and the original sample aliquot are taken through the entire analytical procedure, and the RPD of the duplicate result is calculated. Results are expressed as RPD and are compared to control limits that have been established for each analyte.

2.11.2.5 Surrogate Spikes

Surrogates are organic compounds that are similar to the analytes of interest in chemical properties but are not normally found in environmental samples. Surrogates are added to field and QC samples, before the samples are extracted, to assess the efficacy of the extraction procedure and to assess the bias that is introduced by the sample matrix. Results are reported in terms of percent recovery. Individual analytical methods may require sample reanalysis based on surrogate criteria.

The laboratory will use surrogate recoveries mainly to assess matrix effects on sample analysis. Obvious problems with sample preparation and analysis (i.e., evaporation to dryness or a leaking septum) that can lead to poor surrogate spike recoveries must be eliminated before low surrogate recoveries can be attributed to matrix effects.

2.11.3 Common Data Quality Indicators

This section describes how QA objectives for precision, accuracy, completeness, and sensitivity are measured, calculated, and reported.

2.11.3.1 Precision

Precision of many analyses is assessed by comparing analytical results of MS and MSD sample pairs for organic analyses, field duplicate samples, laboratory duplicate samples (MDs), and field replicate measurements. If precision is calculated from two measurements, it is normally measured as RPD. If precision is calculated from three or more replicates, relative standard deviation is calculated.

2.11.3.2 Accuracy

The accuracy of many analytical methods is assessed by using the results of MS and MSD samples for organic analyses, MS samples for inorganic analyses, surrogate spike samples, LCSs, standard reference materials, independent check standards, and measurements of instrument responses against zero and span gases.

For measurements in which spikes are used, percent recovery will be calculated.

2.11.3.3 Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this SAP, and when none of the QC criteria that affect data usability are exceeded. When data validation is completed, the percent completeness value will be calculated by dividing the number of useable results by the total number of sample results planned for this investigation. Completeness will also be evaluated as part of the data quality assessment (DQA) process (EPA 2006c, 2006d). This evaluation will help determine whether limitations are associated with the decisions to be made based on the data collected.

2.11.3.4 Sensitivity

The achievement of method detection limits depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure data quality and to ensure that analyses meet the QA objectives that have been established for sensitivity.

2.11.4 Instrument and Equipment Testing, Inspection, and Maintenance Requirements

This section outlines testing, inspection, and maintenance procedures for field equipment and instruments and for laboratory instruments.

2.11.4.1 General Requirements

Testing, inspection, and maintenance methods and frequency will be based on the: (1) type of instrument; (2) instrument's stability characteristics; (3) required accuracy, sensitivity, and precision of the instrument; (4) instrument's intended use, considering project-specific DQOs; (5) manufacturer's recommendations; and (6) other conditions that affect measurement or operational control. For most instruments, preventive maintenance is performed in accordance with procedures and schedules recommended in (1) the instrument manufacturer's literature or operating manual, or (2) SOPs associated with particular applications of the instrument. In some cases, testing, inspection, and maintenance procedures and schedules will differ from the manufacturer's specifications or SOPs. This can occur when a field instrument is used to make critical measurements or when the analytical methods that are associated with a laboratory instrument require more frequent testing, inspection, and maintenance.

2.11.4.2 Field Equipment and Instruments

Leased field equipment will be used to conduct sediment sampling. The vendor will be responsible for thoroughly checking field equipment and instruments before they are shipped or transported to the field. Copies of testing, inspection, and maintenance procedures will be shipped to the field with the equipment and instruments.
After the field equipment arrives in the field, it will be inspected for damage. Damaged equipment will be replaced or repaired immediately. Battery-operated equipment will be checked to ensure full operating capacity; if needed, batteries will be recharged or replaced.

Following use, field equipment will be decontaminated properly before being returned to the source. When the equipment is returned, copies of field notes regarding equipment problems will be included so that problems are not overlooked and necessary equipment repairs are performed.

2.11.4.3 Laboratory Instruments

Laboratories that analyze samples collected under the EPA Region 6 RAC II program must have a preventive maintenance program that addresses: (1) testing, inspection, and maintenance procedures; and (2) the maintenance schedule for each measurement system and required support activity. This program is usually documented by a SOP for each analytical instrument that is to

be used. Typically, the program will be laboratory-specific; however, it should follow requirements outlined in EPA-approved guidelines. Some of the basic requirements and components of such a program are as follows:

- As a part of its QA/QC program, each laboratory will conduct a routine preventive maintenance program to minimize instrument failure and other system malfunction.

- An internal group of qualified personnel will maintain and repair instruments, equipment, tools, and gauges. Alternatively, manufacturers' representatives may provide scheduled instrument maintenance and emergency repair under a repair and maintenance contract.

- The laboratory will perform instrument maintenance on a regularly scheduled basis. The scheduled service of critical items should minimize the downtime of the measurement system. The laboratory will prepare a list of critical spare parts for each instrument. The laboratory will request the spare parts from the manufacturer and will store the parts.

- Testing, inspection, and maintenance procedures described in laboratory SOPs will be performed in accordance with manufacturer's specifications and the requirements of the specific analytical methods that are used.

- Maintenance and service must be documented in service logbooks (or the site-specific logbook) to provide a history of maintenance records. A separate service logbook should be kept for each instrument; however, due to the limited scope of this project, the service records will be maintained in the site-specific field logbook. Maintenance records will be traceable to the specific instrument, equipment, tool, or gauge.

- The laboratory will maintain and file records that are produced as a result of tests, inspections, or maintenance of laboratory instruments. These records will be available for review by internal and external laboratory system audits that are conducted under the EPA Region 6 RAC II program.

2.12 INSTRUMENT CALIBRATION AND FREQUENCY

This section describes the procedures for maintaining the accuracy of field equipment and laboratory instruments that are used for field tests and laboratory analyses. The equipment and instruments should be calibrated before each use or, when not in use, on a scheduled periodic basis.

2.12.1 Field Equipment

The use of field equipment for testing and analysis is not anticipated during this field event.

2.12.2 Laboratory Instruments

Laboratory equipment that is used to analyze samples collected under the EPA Region 6 RAC II program will be calibrated on the basis of written SOPs that are maintained by the laboratory. Calibration records (including the dates and times of calibration and the names of the personnel performing the calibration) will be filed at the location at which the analytical work was performed and maintained by the laboratory personnel who performed QC activities. Subcontractor laboratories may conduct laboratory work under the EPA Region 6 RAC II program. The laboratory QA manager is responsible for ensuring that laboratory instruments are calibrated in accordance with the requirements of this SAP.

The laboratories will follow the method-specific calibration procedures and requirements for laboratory measurements. Calibration procedures and requirements will also be provided, as appropriate, for laboratory support equipment, i.e., balances, mercury thermometers, pH meters, and other equipment that is used to take chemical and physical measurements.

2.13 REQUIREMENTS FOR INSPECTION AND ACCEPTANCE OF SUPPLIES AND CONSUMABLES

The EA Project Manager is responsible for identifying the types and quantities of supplies and consumables that are needed for collecting the samples for this Task Order. The Project Manager is also responsible for determining acceptance criteria for these items. When supplies are received, the EA field personnel will check packing slips against purchase orders and inspect the condition of supplies before the supplies are accepted for use on a project. If the supplies do not meet the acceptance criteria, deficiencies will be noted on the packing slip and purchase order. Afterward, the item will be returned to the vendor for replacement or repair.

2.14 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

For this project, EA anticipates acquiring data from non-direct measurements, i.e., databases, spreadsheets, and literature files.

2.15 DATA MANAGEMENT

Data for this project will be obtained from a combination of sources, including EPA Region 6 Laboratory and CLP laboratories. The data-gathering process requires a coordinated effort and will be conducted by project staff members in conjunction with potential data producers. The data will be obtained from the analytical service provider, when appropriate, in the form of an electronic data deliverable, in addition to the required hard copy analytical data package. Formal verification (or validation) of data will be conducted before associated results are presented or are used in subsequent activities.

Data tracking is essential to ensure timely, cost effective, and high quality results. Data tracking begins with sample chain-of-custody. When the analytical service provider receives custody of the samples, the provider will send a sample acknowledgment to EA. The sample acknowledgment will confirm sample receipt, condition, and required analyses. The EPA tracking software (Forms II Lite) will contain pertinent information about each sample and can track the data at each phase of the process. The tracking software carries the data through completion of the data validation.

EA will validate 10 percent of the investigative analytical data received from subcontract laboratories (other than the EPA Region 6 Laboratory or CLP laboratories) to ensure that the confirmatory data are accurate and defensible. A partial review will be conducted on the remaining 90 percent of the data received from subcontract laboratories. Data will be evaluated for usability by EA in accordance with EPA CLP guidelines for data review (EPA 2002a, 2008, 2010b).

As a part of the data validation process, electronic data deliverables will be reviewed against hard copy deliverables to ensure accurate transfer of data. In addition, the hard copy will be evaluated for errors in the calculation of results. After the data validation, qualifiers can be placed on the data to indicate the usability of the data. These qualifiers will be placed into an electronic data file. Upon approval of the data set with the appropriate data qualifiers, the electronic data will be released to the Project Manager for reporting.

3. ASSESSMENT AND OVERSIGHT

This section describes the field and laboratory assessments that may be conducted during this project, the individuals responsible for conducting assessments, corrective actions that may be implemented in response to assessment results, and how quality-related issues will be reported to EA and EPA.

3.1 ASSESSMENT AND RESPONSE ACTIONS

Under the EPA Region 6 RAC II program, performance and system audits of field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with the following:

- Performance and system audits
  - Audit personnel
  - Audit scope of work
  - Audit frequencies
  - Audit reports
- Corrective action
  - Sample collection
  - Laboratory analyses.

Nonconforming items and activities are those that do not meet the project requirements, procurement document criteria, and approved work procedures. Nonconformance may be detected and identified by the following personnel:

- ***Project Personnel***—During field operations, supervision of subcontractors, and field inspections
- ***Testing Personnel***—During preparation for and performance of tests, equipment calibration, and QC activities
- ***QA Personnel***—During the performance of audits, surveillance, and other QA activities.

Each nonconformance that affects quality will be documented by the person who identifies or originates the nonconformance. Documentation of nonconformance will include the following components:

- Description of nonconformance
- Identification of personnel who are responsible for correcting the nonconformance and, if verification is required, for verifying satisfactory resolution

- Method(s) for correcting the nonconformance (corrective action) or description of the variance granted

- Proposed schedule for completing corrective action and the corrective action taken.

Nonconformance documentation will be made available to the Project Manager, QA Manager, and subcontractor (e.g., non-CLP subcontract laboratories) management personnel, as appropriate.

The field personnel and QA personnel, as appropriate, are responsible for notifying the Project Manager and the QA manager of the nonconformance. In addition, the Project Manager and the project staff, as appropriate, will be notified of significant nonconformances that could affect the results of the work. The Project Manager is responsible for determining whether notification of EPA is required.

The completion of corrective actions for significant nonconformances will be documented by QA personnel during future auditing activities. Significant recurring nonconformance will be evaluated by project and QA personnel, as appropriate, to determine its cause. Appropriate changes will be instituted, under corporate or project procedures, to prevent recurrence. When such an evaluation is performed, the results will be documented.

3.2 REPORTS TO MANAGEMENT

Effective management of environmental data collection operations requires timely assessment and review of measurement activities. It is essential that open communication, interaction, and feedback be maintained among project participants, including the: (1) EA QA Manager, Program Manager, Project Manager, technical staff, and laboratory subcontractors; and (2) EPA Region 6 Task Order Monitor and QA Officer.

During the remedial investigation field program, EA will prepare weekly reports that summarize the following elements:

- Work progress since the last weekly report

- Site observations, problems, and decisions

- Problems that may impede planned progress

- Safety-related observations, incidents, or potential safety problems and the corrective action(s) taken to mitigate the problem(s)

- Corrective measures and procedures to regain the planned schedule, if required

- Work scheduled for the next work period.

EA prepares monthly progress reports for each Task Order that is conducted under the EPA Region 6 RAC II program. These reports address QA issues that are specific to the Task Order and facilitate timely communication of such issues.

At the program level, the QA Manager prepares quarterly status reports of QA issues that are related to EA's work on the EPA Region 6 RAC II program. These reports are distributed to EA's President, Corporate QA Manager, RAC II Program Manager, and, upon request, the EPA Region 6 Project Officer. QA status reports address the following areas:

- Results of QA audits and other inspections, including quality improvement; opportunities that have been identified for further action
- Instrument, equipment, or procedural problems that affect QA
- Subcontractor performance issues
- Corrective actions
- Status of previously reported activities and quality improvement initiatives
- Work planned for the next reporting period.

4. DATA VALIDATION AND USABILITY

This section describes the procedures that are planned to review, verify, and validate field and laboratory data. Procedures for verifying that the data are sufficient to meet DQOs and measurement quality objectives for the project are also discussed. Section 4.1 focuses on data review and reduction requirements for work conducted under the EPA Region 6 RAC II program. Section 4.2 addresses data validation and verification requirements. Section 4.3 addresses reconciliation with DQOs.

4.1 DATA REVIEW AND REDUCTION REQUIREMENTS

Data reduction and review are essential functions for preparing data that can be used effectively to support project decisions and DQOs. These functions must be performed accurately and in accordance with EPA-approved procedures and techniques. Data reduction includes computations and data manipulations that produce the final results that are used during the investigation. Data review includes procedures that field or laboratory personnel conduct to ensure that measurement results are correct and acceptable in accordance with the QA objectives that are stated in this SAP. Field and laboratory measurement data reduction and review procedures and requirements are specified in previously discussed field and laboratory methods, SOPs, and guidance documents.

The EPA Region 6 Laboratory, CLP laboratory, and/or subcontracted non-CLP laboratory will complete data reduction for chemical and physical laboratory measurements and will complete an in-house review of laboratory analytical results. The laboratory QA manager will be responsible for ensuring that laboratory data reduction and review procedures follow the requirements that are stated in this SAP. The laboratory QA manager will also be responsible for assessing data quality and for advising the EA QA Manager of possible QA/QC problems with laboratory data.

4.2 VALIDATION AND VERIFICATION METHODS

Data that are used to support activities under the EPA Region 6 RAC II program must be valid for their intended purposes. This section outlines the basic data validation procedures that will be followed for field and laboratory measurements. The following sections identify personnel who are responsible for data validation and the general data validation process and EPA data validation guidance that will be followed.

4.2.1 Data Validation Responsibilities

When analytical services are provided by laboratories subcontracted by EA, EA is responsible for data validation. The QA Manager has primary responsibility for coordinating EA's data validation activities. EA will conduct full validation on 10 percent of subcontracted laboratory data for investigation samples. Partial validation will be conducted on the remaining 90 percent of subcontracted laboratory data. Data validation and review will be completed by one or more experienced data reviewers. When data are generated by the EPA Region 6 Laboratory, it will

be used as received from the laboratory, with no further validation. Data from CLP laboratories are validated by EPA's Environmental Services Assistance Team.

4.2.2 Data Validation Procedures

The validity of a data set is determined by comparing the data with a predetermined set of QC limits. EA data reviewers will conduct a systematic review of the data for compliance with established QC limits (i.e., sensitivity, precision, and accuracy), on the basis of spike, duplicate, and blank sampling results that are provided by the laboratory. The data review will identify out-of-control data points or omissions. EA data reviewers will evaluate laboratory data for compliance with the following information:

- Method and project-specific analytical service requests
- Holding times
- Initial and continuing calibration acceptance criteria
- Field, trip, and method blank acceptance criteria
- Surrogate recovery
- Field duplicates and MS and MSD acceptance criteria
- MD precision
- LCS accuracy
- Other laboratory QC criteria specified by the method or on the project-specific analytical service request form
- Compound identification and quantitation
- Overall assessment of data, in accordance with project-specific objectives.

EA will follow the most current EPA CLP guidelines (EPA 2002a; 2008; 2010b) for completing data validation for applicable test methods. General procedures in the CLP guidelines will be modified, as necessary, to fit the specific analytical method that is used to produce the data. In cases, data validation requirements will depend on: (1) DQO levels that are defined in Section 1.3; (2) reporting requirements that are defined in Section 1.4; and (3) data deliverables that are requested from the laboratory, as discussed in Section 1.6.

4.3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

The main purpose of a QA system is to define a process for collecting data that are of known quality, are scientifically valid, are legally defensible, and fully support decisions that will be

based on the data. To achieve this purpose, the SAP requires that DQOs be fully defined (Section 1.3). Other parts of the QA system must then be planned and implemented in a manner that is consistent with the DQOs. QA system components that follow directly from the DQOs include: (1) documentation and reporting requirements (Section 1.6), (2) sample process design and sampling methods requirements (Sections 2.1 through 2.9), (3) analytical methods and analytical service requests (Section 2.10), (4) QC requirements (Section 2.11), and (5) data reduction and validation and reporting methods (Sections 4.1 and 4.2).

After environmental data have been collected, reviewed, and validated, the data will undergo a final evaluation to determine whether the DQOs specified in this SAP have been met. EA will follow EPA's DQA process to verify that the type, quality, and quantity of data that are collected are appropriate for their intended use (EPA 2006c, 2006d).

The DQA process involves: (1) verifying that the data have met the assumptions under which the data collection design and DQOs were developed, (2) taking appropriate corrective action if the assumptions have not been met, and (3) evaluating the extent to which the data support the decision that must be made so that scientifically valid and meaningful conclusions can be drawn from the data. To the extent possible, EA will follow DQA methods and procedures that have been outlined by EPA (EPA 2006c, 2006d).

Following the conclusion of the Five-Year Review field program and receipt of fixed-laboratory data, the data evaluation will include:

- ***Data Usability Evaluation and Field QA/QC***—The usability of the laboratory analytical data in terms of the CLP data validation summaries and field QA/QC will be evaluated.

- ***Data Reduction and Tabulation***—Field sampling data and analytical results will be reduced and tabulated.

- ***Data Evaluation Summary Report***—A Data Evaluation Summary Report will be submitted along with the Five-Year Review report that documents and summarizes the analytical data collected during this Five-Year Review, including the data quality and usability as related to the site-specific DQOs. The Data Evaluation Summary Report shall also include previous data collected during previous site investigations (if made available) for statistical comparisons to the data collected during the Five-Year Review. Field QA/QC results will be summarized in context with fixed-laboratory sample results.

EA will compile analytical and field data into a format that is compatible with EPA Region 6 or National Electronic Data Management Network. EA will use the data to prepare the Five-Year Review report. The specific requirements and elements of this report are discussed in detail in the EPA-approved Five-Year Review Work Plan (EA 2011a).

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FIGURES

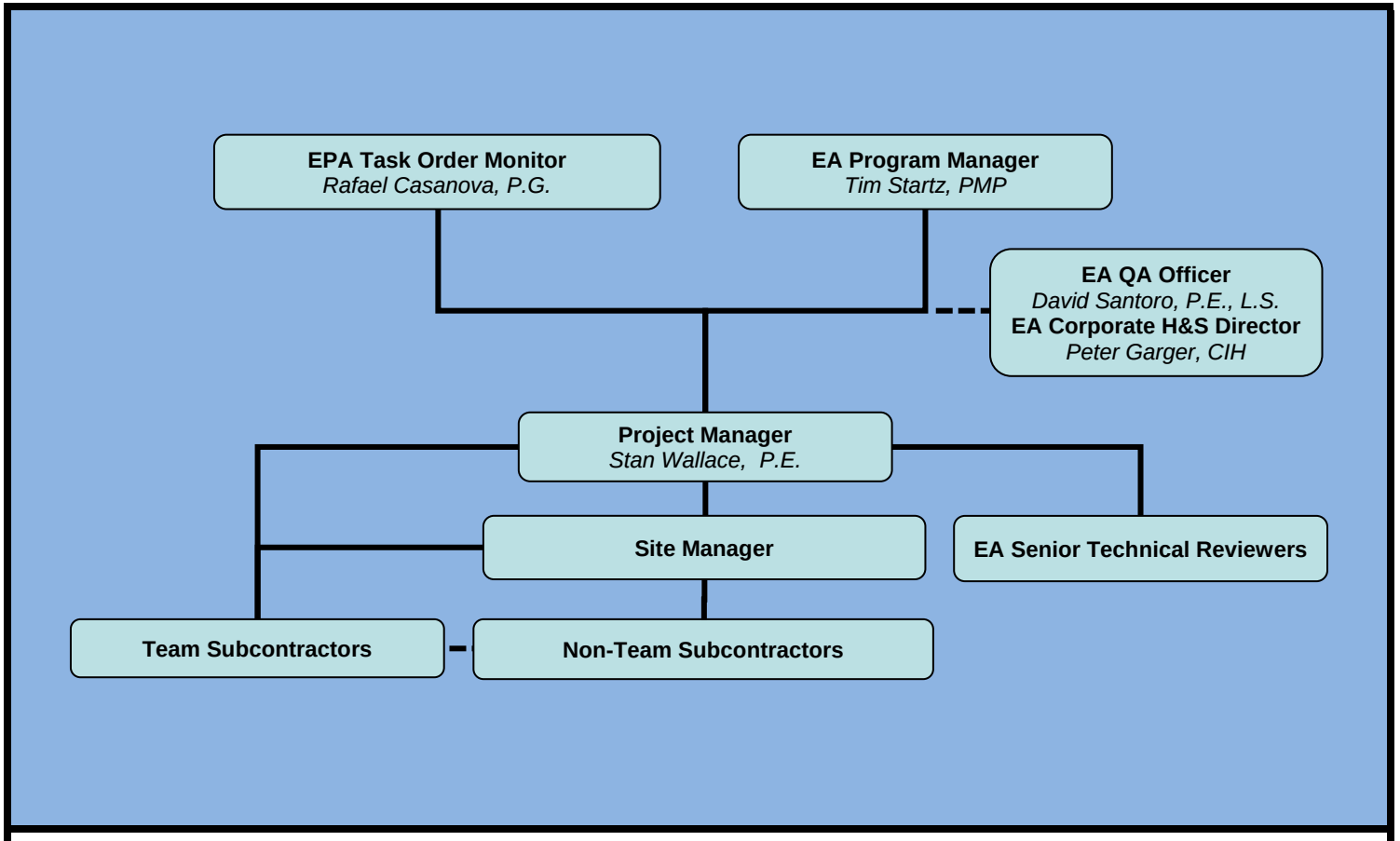
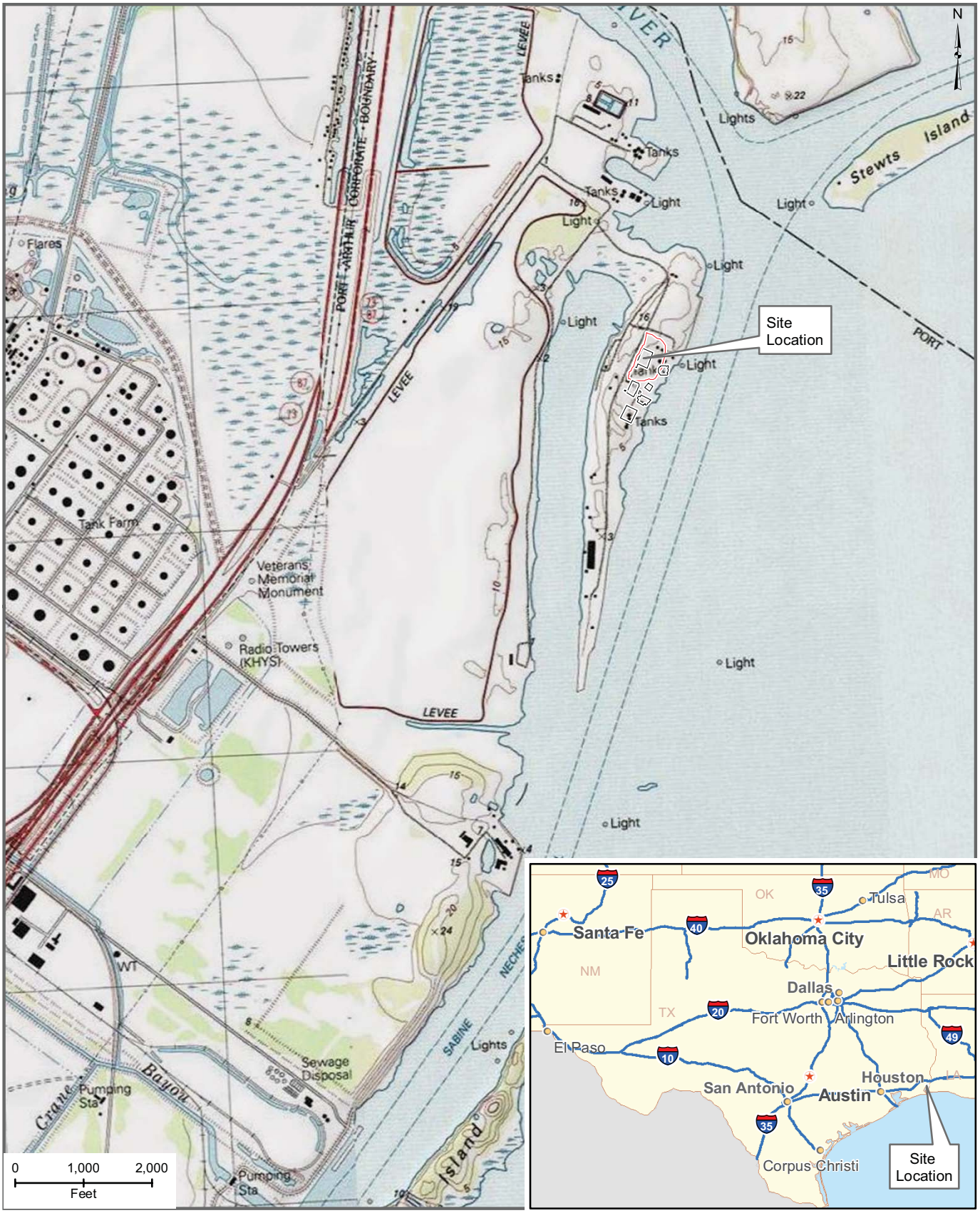


Figure 1 - Project Organization

Five Year Review
State Marine of Port Arthur Superfund Site
Port Arthur, Jefferson County, Texas



Source: ESRI ArcGIS Online and data partners including USGS and © 2007 National Geographic Society, 2009 Redlands, CA: Environmental Systems Research Institute

First Five-Year Review
State Marine of Port Arthur Superfund Site
Port Arthur, Jefferson County, Texas

Figure 2
Site Overview and Location Map

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Sample Name	Zone	X-Coordinate	Y-Coordinate
J-SD5-1	Intertidal	3595800.1259	13932498.4722
H-SD5-1	Intertidal	3596047.1601	13933044.7378
A-SD5-1	Intertidal	3595471.3289	13932127.5854
K-SD5-1	Nearshore	3596063.0657	13932882.2644
L-SD5-1	Nearshore	3595665.2655	13931968.5040
M-SD5-1	Nearshore	3596277.2658	13933286.8301
N-SD5-1	Nearshore	3595481.6654	13931842.9491
O-SD5-1	Nearshore	3595527.5654	13932094.0588
B-SD5-1	Nearshore	3596008.7603	13932763.2380
E-SD5-1	Nearshore	3595772.8496	13932368.5416
G-SD5-1	Nearshore	3595629.9421	13932014.6758
P-SD5-1	Offshore	3596081.7332	13932317.7429
Q-SD5-1	Offshore	3596301.1005	13933009.6570
F-SD5-1	Offshore	3595899.8783	13932209.7555
D-SD5-1	Offshore	3596033.7123	13932411.6406
C-SD5-1	Offshore	3596128.9838	13932613.5254
R-SD5-1	Judgemental		
S-SD5-1	Judgemental		

Image Source: 2008-2009 Texas Orthoimagery Program provided by Texas Strategic Mapping Program (StratMap), TNRIS

Note:
VSP - Visual Sampling Plan

First Five-Year Review
State Marine of Port Arthur Superfund Site
Port Arthur, Jefferson County, Texas

Figure 3
Proposed Sampling Location

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Appendix A

Visual Sample Plan
Results

(Included on Electronic Copy Only)

Random sampling locations for comparing a mean with a fixed threshold (parametric)

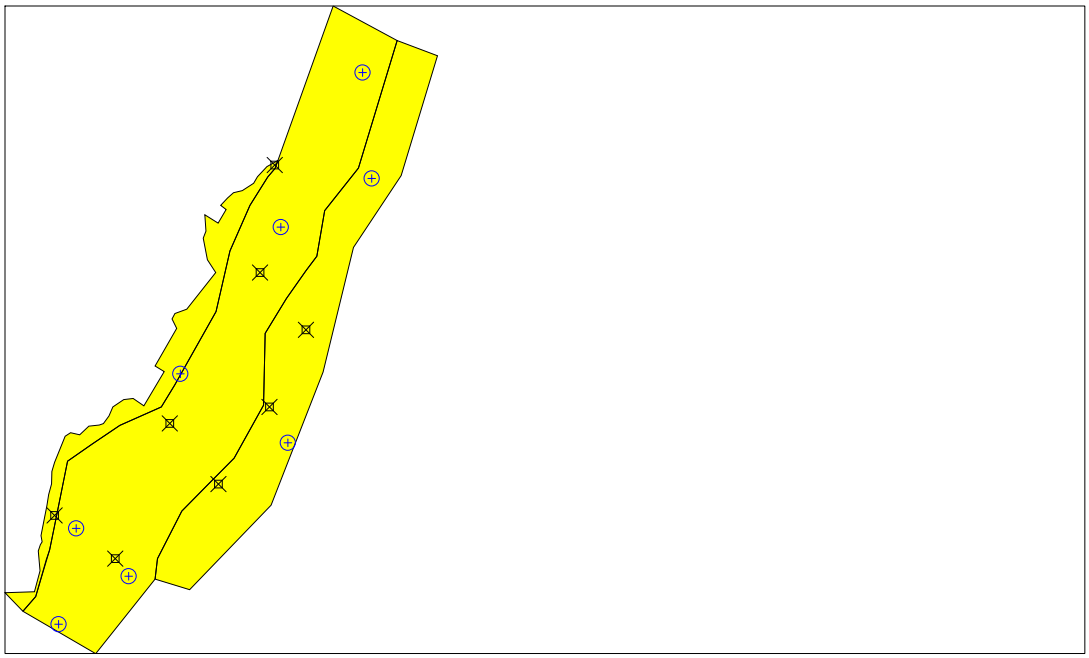
Summary

This report summarizes the sampling design, associated statistical assumptions, as well as general guidelines for conducting post-sampling data analysis. Sampling plan components presented here include how many sampling locations to choose and where within the sampling area to collect those samples. The type of medium to sample (i.e., soil, groundwater, etc.) and how to analyze the samples (in-situ, fixed laboratory, etc.) are addressed in other sections of the sampling plan.

The following table summarizes the sampling design. A figure that shows sampling locations in the field and a table that lists sampling location coordinates are also provided below.

SUMMARY OF SAMPLING DESIGN	
Primary Objective of Design	Compare a site mean to a fixed threshold
Type of Sampling Design	Parametric
Sample Placement (Location) in the Field	Simple random sampling
Working (Null) Hypothesis	The mean value at the site is less than the threshold
Formula for calculating number of sampling locations	Student's t-test
Calculated total number of samples	8
Number of samples on map [a]	16
Number of selected sample areas [b]	3
Specified sampling area [c]	660403.57 ft^2
Total cost of sampling [d]	$4,100.00

[a] This number may differ from the calculated number because of 1) grid edge effects, 2) adding judgment samples, or 3) selecting or unselecting sample areas.
[b] The number of selected sample areas is the number of colored areas on the map of the site. These sample areas contain the locations where samples are collected.
[c] The sampling area is the total surface area of the selected colored sample areas on the map of the site.
[d] Including measurement analyses and fixed overhead costs. See the Cost of Sampling section for an explanation of the costs presented here.



Area: Area 1					
X Coord	**Y Coord**	**Label**	**Value**	**Type**	**Historical**
3595800.1259	13932498.4722			Random	
3596047.1601	13933044.7378			Manual	
3595471.3289	13932127.5854			Manual	

Area: Area 2					
X Coord	**Y Coord**	**Label**	**Value**	**Type**	**Historical**
3596063.0657	13932882.2644			Random	
3595665.2655	13931968.5040			Random	
3596277.2658	13933286.8301			Random	
3595481.6654	13931842.9491			Random	
3595527.5654	13932094.0588			Random	
3596008.7603	13932763.2380			Manual	
3595772.8496	13932368.5416			Manual	
3595629.9421	13932014.6758			Manual	

Area: Area 3					
X Coord	**Y Coord**	**Label**	**Value**	**Type**	**Historical**
3596081.7332	13932317.7429			Random	
3596301.1005	13933009.6570			Random	
3595899.8783	13932209.7555			Manual	
3596033.7123	13932411.6406			Manual	
3596128.9838	13932613.5254			Manual	

Primary Sampling Objective

The primary purpose of sampling at this site is to compare a mean value of a site with a fixed threshold. The working

hypothesis (or 'null' hypothesis) is that the mean value at the site is less than the threshold. The alternative hypothesis is that the mean value is equal to or exceeds the threshold. VSP calculates the number of samples required to reject the null hypothesis in favor of the alternative hypothesis, given a selected sampling approach and inputs to the associated equation.

Selected Sampling Approach
A parametric random sampling approach was used to determine the number of samples and to specify sampling locations. A parametric formula was chosen because the conceptual model and historical information (e.g., historical data from this site or a very similar site) indicate that parametric assumptions are reasonable. These assumptions will be examined in post-sampling data analysis.

Both parametric and non-parametric approaches rely on assumptions about the population. However, non-parametric approaches typically require fewer assumptions and allow for more uncertainty about the statistical distribution of values at the site. The trade-off is that if the parametric assumptions are valid, the required number of samples is usually less than the number of samples required by non-parametric approaches.

Locating the sample points randomly provides data that are separated by many distances, whereas systematic samples are all equidistant apart. Therefore, random sampling provides more information about the spatial structure of the potential contamination than systematic sampling does. As with systematic sampling, random sampling also provides information regarding the mean value, but there is the possibility that areas of the site will not be represented with the same frequency as if uniform grid sampling were performed.

Number of Total Samples: Calculation Equation and Inputs
The equation used to calculate the number of samples is based on a Student's t-test. For this site, the null hypothesis is rejected in favor of the alternative hypothesis if the sample mean is sufficiently larger than the threshold. The number of samples to collect is calculated so that 1) there will be a high probability (1-β) of rejecting the null hypothesis if the alternative hypothesis is true and 2) a low probability (α) of rejecting the null hypothesis if the null hypothesis is true.

The formula used to calculate the number of samples is:

$$n = \frac{S^2}{\Delta^2}\left(Z_{1-\alpha} + Z_{1-\beta}\right)^2 + 0.5Z_{1-\alpha}^2$$

where
n is the number of samples,
S is the estimated standard deviation of the measured values including analytical error,
Δ is the width of the gray region,
α is the acceptable probability of incorrectly concluding the site mean exceeds the threshold,
β is the acceptable probability of incorrectly concluding the site mean is less than the threshold,
$Z_{1-\alpha}$ is the value of the standard normal distribution such that the proportion of the distribution less than $Z_{1-\alpha}$ is 1-α,
$Z_{1-\beta}$ is the value of the standard normal distribution such that the proportion of the distribution less than $Z_{1-\beta}$ is 1-β.

The values of these inputs that result in the calculated number of sampling locations are:

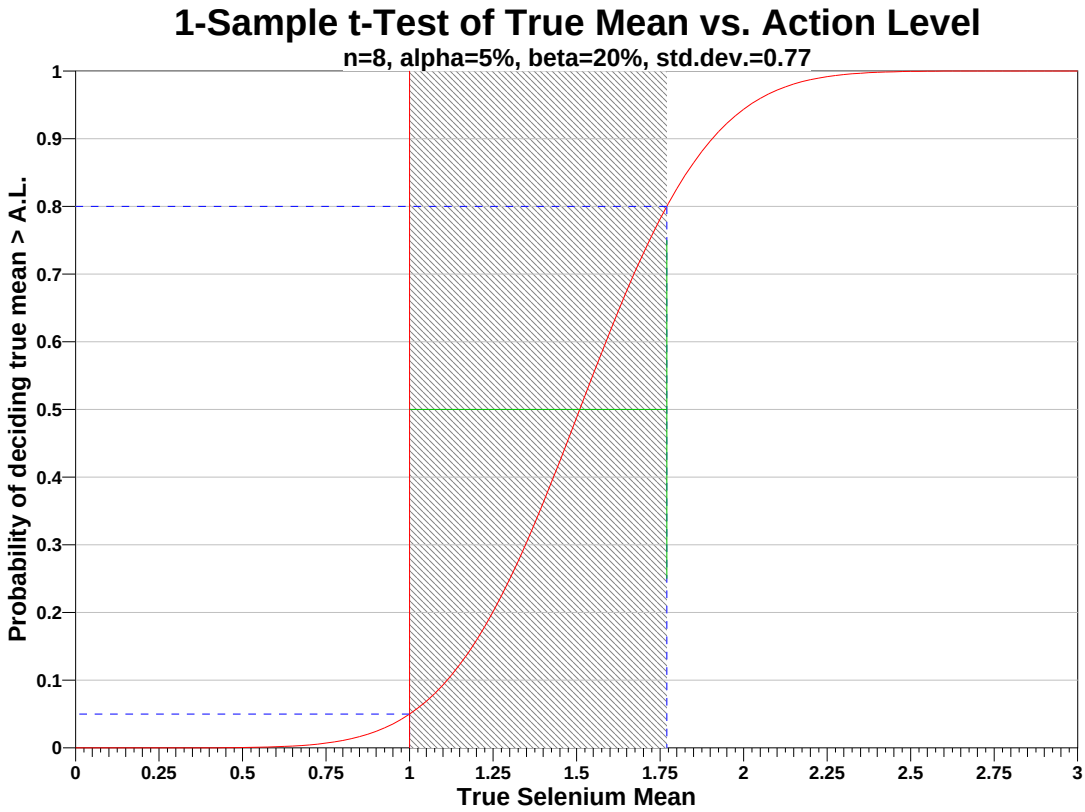
Analyte	n	Parameter					
		S	Δ	α	β	$Z_{1-\alpha}$ [a]	$Z_{1-\beta}$ [b]
Selenium	8	0.77	0.77	0.05	0.2	1.64485	0.841621

[a] This value is automatically calculated by VSP based upon the user defined value of α.
[b] This value is automatically calculated by VSP based upon the user defined value of β.

The following figure is a performance goal diagram, described in EPA's QA/G-4 guidance (EPA, 2000). It shows the probability of concluding the sample area is dirty on the vertical axis versus a range of possible true mean values for the site on the horizontal axis. This graph contains all of the inputs to the number of samples equation and pictorially represents the calculation.

The red vertical line is shown at the threshold (action limit) on the horizontal axis. The width of the gray shaded area is equal to Δ; the lower horizontal dashed blue line is positioned at α on the vertical axis; the upper horizontal dashed blue

line is positioned at 1-β on the vertical axis. The vertical green line is positioned at one standard deviation above the threshold. The shape of the red curve corresponds to the estimates of variability. The calculated number of samples results in the curve that passes through the lower bound of Δ at α and the upper bound of Δ at 1-β. If any of the inputs change, the number of samples that result in the correct curve changes.



Statistical Assumptions

The assumptions associated with the formulas for computing the number of samples are:

1. the sample mean is normally distributed (this happens if the data are roughly symmetric or the sample size is more than 30; for extremely skewed data sets, additional samples may be required for the sample mean to be normally distributed),
2. the variance estimate, S^2, is reasonable and representative of the population being sampled,
3. the population values are not spatially or temporally correlated, and
4. the sampling locations will be selected randomly.

The first three assumptions will be assessed in a post data collection analysis. The last assumption is valid because the sample locations were selected using a random process.

Sensitivity Analysis

The sensitivity of the calculation of number of samples was explored by varying the standard deviation, upper bound of gray region (% of action level), beta (%), probability of mistakenly concluding that μ < action level and alpha (%), probability of mistakenly concluding that μ > action level. The following table shows the results of this analysis.

Number of Samples							
AL=1		**α=5**		**α=10**		**α=15**	
		s=1.54	**s=0.77**	**s=1.54**	**s=0.77**	**s=1.54**	**s=0.77**
UBGR=110	**β=15**	1707	428	1276	320	1020	256
	β=20	1468	368	1070	269	838	210
	β=25	1278	321	909	228	695	175
UBGR=120	**β=15**	428	108	320	81	256	65

	β=20	368	93	269	68	210	53
	β=25	321	82	228	58	175	44
UBGR=130	**β=15**	191	49	143	37	114	29
	β=20	165	43	120	31	94	24
	β=25	144	37	102	27	78	20

s = Standard Deviation
UBGR = Upper Bound of Gray Region (% of Action Level)
β = Beta (%), Probability of mistakenly concluding that μ < action level
α = Alpha (%), Probability of mistakenly concluding that μ > action level
AL = Action Level (Threshold)

Cost of Sampling
The total cost of the completed sampling program depends on several cost inputs, some of which are fixed, and others that are based on the number of samples collected and measured. Based on the numbers of samples determined above, the estimated total cost of sampling and analysis at this site is $4,100.00, which averages out to a per sample cost of $512.50. The following table summarizes the inputs and resulting cost estimates.

COST INFORMATION			
Cost Details	**Per Analysis**	**Per Sample**	**8 Samples**
Field collection costs		$100.00	$800.00
Analytical costs	$400.00	$400.00	$3,200.00
Sum of Field & Analytical costs		**$500.00**	**$4,000.00**
Fixed planning and validation costs			$100.00
Total cost			**$4,100.00**

Data Analysis for Selenium
The following data points were entered by the user for analysis.

Selenium										
Rank	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
0	2.22	2.41	2.96	3.15	3.63	3.69	3.85	3.87	4.66	

SUMMARY STATISTICS for Selenium	
n	9
Min	2.22
Max	4.66
Range	2.44
Mean	3.3822
Median	3.63
Variance	0.59672
StdDev	0.77248
Std Error	0.25749
Skewness	-0.079358
Interquartile Range	1.175

Percentiles								
1%	**5%**	**10%**	**25%**	**50%**	**75%**	**90%**	**95%**	**99%**
2.22	2.22	2.22	2.685	3.63	3.86	4.66	4.66	4.66

Outlier Test
Dixon's extreme value test was performed to test whether the lowest value is a statistical outlier. The test was conducted at the 5% significance level.

Data should not be excluded from analysis solely on the basis of the results of this or any other statistical test. If any values are flagged as possible outliers, further investigation is recommended to determine whether there is a plausible explanation that justifies removing or replacing them.

DIXON'S OUTLIER TEST for Selenium	
Dixon Test Statistic	0.11515
Dixon 5% Critical Value	0.512

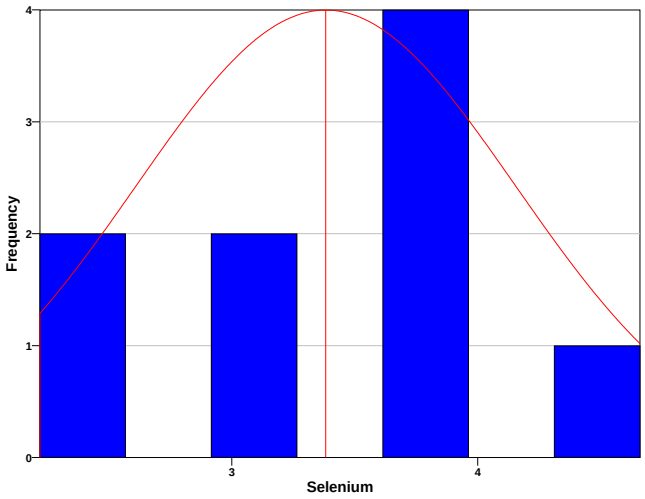
The calculated test statistic does not exceed the critical value, so the test cannot reject the null hypothesis that there are no outliers in the data, and concludes that the minimum value 2.22 is not an outlier at the 5% significance level.

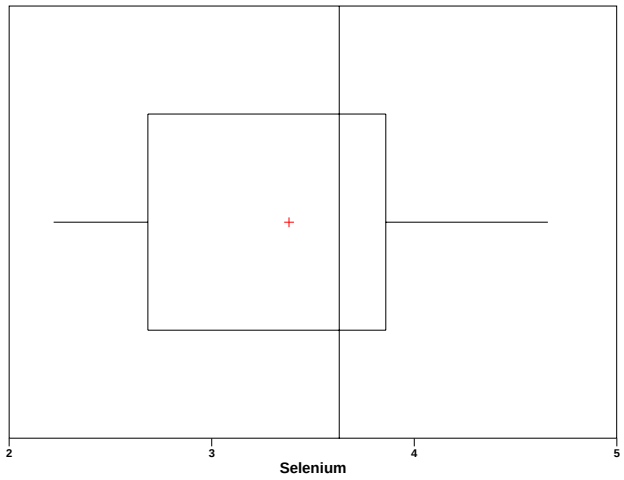
Data Plots for Selenium
Graphical displays of the data are shown below.

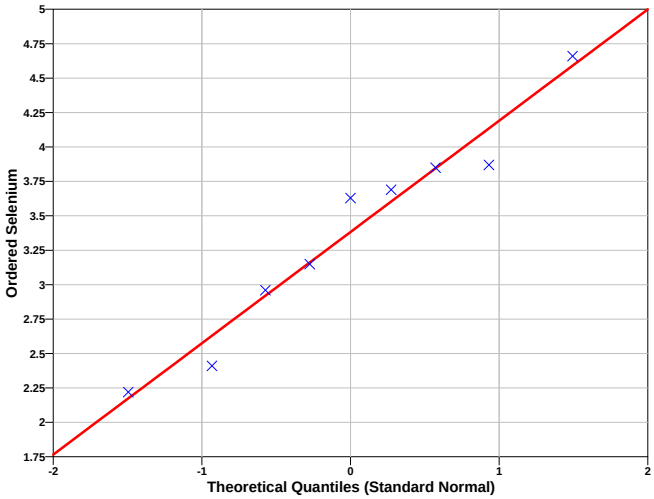
The Histogram is a plot of the fraction of the n observed data that fall within specified data “bins.” A histogram is generated by dividing the x axis (range of the observed data values) into "bins" and displaying the number of data in each bin as the height of a bar for the bin. The area of the bar is the fraction of the n data values that lie within the bin. The sum of the fractions for all bins equals one. A histogram is used to assess how the n data are distributed (spread) over their range of values. If the histogram is more or less symmetric and bell shaped, then the data may be normally distributed.

The Box and Whiskers plot is composed of a central box divided by a line, and with two lines extending out from the box, called the "whiskers". The line through the box is drawn at the median of the n data observed. The two ends of the box represent the 25th and 75th percentiles of the n data values, which are also called the lower and upper quartiles, respectively, of the data set. The sample mean (mean of the n data) is shown as a "+" sign. The upper whisker extends to the largest data value that is less than the upper quartile plus 1.5 times the interquartile range (upper quartile minus the lower quartile). The lower whisker extends to the smallest data value that is greater than the lower quartile minus 1.5 times the interquartile range. Extreme data values (greater or smaller than the ends of the whiskers) are plotted individually as blue Xs. A Box and Whiskers plot is used to assess the symmetry of the distribution of the data set. If the distribution is symmetrical, the box is divided into two equal halves by the median, the whiskers will be the same length, and the number of extreme data points will be distributed equally on either end of the plot.

The Q-Q plot graphs the quantiles of a set of n data against the quantiles of a specific distribution. We show here only the Q-Q plot for an assumed normal distribution. The pth quantile of a distribution of data is the data value, x_n, for which a fraction p of the distribution is less than x_n. If the data plotted on the normal distribution Q-Q plot closely follow a straight line, even at the ends of the line, then the data may be assumed to be normally distributed. If the data points deviate substantially from a linear line, then the data are not normally distributed.







For more information on these plots consult Guidance for Data Quality Assessment, EPA QA/G-9, pgs 2.3-1 through 2.3-12. (http://www.epa.gov/quality/qa-docs.html).

Tests for Selenium

A goodness-of-fit test was performed to test whether the data set had been drawn from an underlying normal distribution. The Shapiro-Wilk (SW) test was used to test the null hypothesis that the data are normally distributed. The test was conducted at the 5% significance level, i.e., the probability the test incorrectly rejects the null hypothesis was set at 0.05.

NORMAL DISTRIBUTION TEST	
Shapiro-Wilk Test Statistic	0.9569
Shapiro-Wilk 5% Critical Value	0.829

The calculated SW test statistic exceeds the 5% Shapiro-Wilk critical value, so we cannot reject the hypothesis that the data are normal, or in other words the data appear to follow a normal distribution at the 5% level of significance. The Q-Q plot displayed above should be used to further assess the normality of the data.

Lower Confidence Limit on the True Mean

Two methods were used to compute the lower confidence limit (LCL) on the mean. The first is a parametric method that assumes a normal distribution. The second is the Chebyshev method, which requires no distributional assumption.

LCLs ON THE MEAN	
95% Parametric LCL	2.9034

95% Non-Parametric (Chebyshev) LCL	2.2598

Because the data appear to be normally distributed according to the goodness-of-fit test performed above, the parametric LCL (2.903) may be a more accurate lower confidence limit on the true mean.

One-Sample t-Test

A one-sample t-test was performed to compare the sample mean to the action level. The null hypothesis used is that the true mean is less than the action level (AL). The t-test was conducted at the 5% significance level. The sample value *t* was computed using the following equation:

$$t = \frac{\bar{x} - AL}{SE}$$

where

x is the sample mean of the n=9 data,

AL is the action level or threshold (1),

SE is the standard error = (standard deviation) / (square root of n).

This *t* was then compared with the critical value $t_{0.95}$, where $t_{0.95}$ is the value of the t distribution with n-1=8 degrees of freedom for which the proportion of the distribution to the left of $t_{0.95}$ is 0.95. The null hypothesis will be rejected if $t >= t_{0.95}$.

ONE-SAMPLE t-TEST		
t-statistic	**Critical Value $t_{0.95}$**	**Null Hypothesis**
9.2516	1.8595	Reject

The test rejected the null hypothesis that the mean value at the site is less than the threshold, therefore conclude the true mean exceeds the threshold.

This report was automatically produced* by Visual Sample Plan (VSP) software version 6.1b.

Software and documentation available at http://vsp.pnl.gov

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* - The report contents may have been modified or reformatted by end-user of software.

Appendix B

Standard Operating Procedures

(Included on Electronic Copy Only)

Standard Operating Procedure No. 001
for
Sample Labels

Prepared by

EA Engineering, Science, and Technology, Inc.
11019 McCormick Road
Hunt Valley, Maryland 21031

Revision 0
August 2007

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for the use of sample labels. Every sample will have a sample label uniquely identifying the sampling point and analysis parameters. An example label is provided below. Other formats with similar levels of detail are acceptable.

PROJECT NAME _____ PROJECT NUM. _____
SAMPLE LOCATION/SITE ID _____
DATE: ____/____/____ TIME: _____:_____
ANALYTES: METALS VOC EXPLOSIVES ORGANICS OTHER
FILTERED: [NO] [YES]
PRESERVATIVE: [NONE] [HNO_3] [OTHER _____]
SAMPLER: _____

2. MATERIALS

The following materials may be required: sample label and indelible laboratory marker.

3. PROCEDURE

The following sections describe how to use the sample labeling system.

3.1 LABEL INFORMATION

As each sample is collected/selected, fill out a sample label. Enter the following information on each label:

- Project name
- Project number
- Location/site identification—Enter the media type (i.e., well number, surface water, soil, etc.) sampling number, and other pertinent information concerning where the sample was taken
- Date of sample collection

- Time of sample collection
- Analyses to be performed (NOTE: Due to number of analytes, details of analysis should be arranged with laboratory *prior to start of work)*
- Whether filtered or unfiltered (water samples only)
- Preservatives (water samples only)
- Number of containers for the sample (e.g., 1 of 2, 2 of 2).

3.2 ROUTINE CHECK

Double-check the label information to make sure it is correct. Detach the label, remove the backing, and apply the label to the sample container. Cover the label with clear tape, ensuring that the tape completely encircles the container.

3.3 RECORD INFORMATION

Record the sample number and designated sampling point in the field logbook, along with the following sample information:

- Time of sample collection (each logbook page should be dated)
- Location of the sample
- Organic vapor meter or photoionization meter readings for the sample (when appropriate)
- Any unusual or pertinent observations (oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, etc.)
- Number of containers required for each sample
- Whether the sample is a quality assurance sample (split, duplicate, or blank).

3.3.1 Logbook Entry

A typical logbook entry might look like this:

- 7:35 a.m. Sample No. MW-3. PID = 35 ppm
- Petroleum odor present. Sample designated MW-3-001.

NOTE: Duplicate samples will be given a unique sample designation rather than the actual sample number with an added prefix or suffix. This will prevent any indication to the laboratory that this is a duplicate sample. This fictitious sample number will be listed in the logbook along with the actual location of the sample.

3.4 SHIPMENT

Place the sample upright in the designated sample cooler. Make sure there is plenty of ice in the cooler at all times.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

5.1 INCIDENTAL ODORS

Note that although incidental odors should be noted in the logbook, it is unwise from a safety and health standpoint to routinely "sniff test" samples for contaminants.

5.2 DUPLICATE SAMPLE

No indication of which samples are duplicates is to be provided to the laboratory.

6. REFERENCES

U.S. Environmental Protection Agency. 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans. QAMS-005/80.

Standard Operating Procedure No. 002
for
Chain-of-Custody Form

Prepared by

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Revision 0
August 2007

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2. MATERIALS 1

3. PROCEDURE 1

4. MAINTENANCE 1

5. PRECAUTIONS 2

6. REFERENCES 2

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for use of the chain-of-custody form. An example is provided as Figure SOP002-1. Other formats with similar levels of detail are acceptable.

2. MATERIALS

The following materials may be required: chain-of-custody form and indelible ink pen.

3. PROCEDURE

- Give the site name and project name/number.
- Enter the sample identification code.
- Indicate the sampling dates for all samples.
- List the sampling times (military format) for all samples.
- Indicate “grab” or “composite” sample with an “X.”
- Specify the sample location.
- Enter the total number of containers per cooler.
- List the analyses/container volume.
- Obtain the signature of sample team leader.
- State the carrier service and airbill number, analytical laboratory, and custody seal numbers.
- Sign, date, and time the “relinquished by” section.
- Upon completion of the form, retain the shipper copy, and affix the other copies to the inside of the sample cooler, in a zip-seal bag to protect from moisture, to be sent to the designated laboratory.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

U.S. Environmental Protection Agency (U.S. EPA). 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.

U.S. EPA. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C. December.

U.S. EPA. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response. January.

Company Name:				Project Manager or Contact: Phone:	Parameters/Method Numbers for Analysis														Chain of Custody Record	
Project No. Dept.: Task:				Project Name:															EA Laboratories 19 Loveton Circle Sparks, MD 21152 Telephone: (410) 771-4950 Fax: (410) 771-4077	
Sample Storage Location:				ATO Number:															**Report Deliverables:** **1 2 3 4 D E** **EDD: Yes/No** **DUE TO CLIENT: _____**	
Page of				**Report #:**																
Date	Time	Water	Soil	Sample Identification 19 Characters	**No. of Containers**														**EA Labs Accession Number**	Remarks
																				LPM:

Samples by: (Signature)	Date/Time	Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time
Relinquished by: (Signature)	Date/Time	Received by Laboratory: (Signature)	Date/Time	Airbill Number:	Sample Shipped by: (Circle) Fed Ex. Puro. UPS Hand Carried Other:

Cooler Temp. _____C pH: ___ Yes ___ No Comments: Custody Seals Intact ___ Yes ___ No

NOTE: Please indicate method number for analyses requested. This will help clarify any questions with laboratory techniques.

Standard Operating Procedure No. 003
for
Subsurface/Utility Clearance

Prepared by

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August 2007

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APPENDIX A: SUBSURFACE CLEARANCE PROCEDURE CHECKLIST
APPENDIX B: UNIFORM COLOR CODES FOR MARKING OF UNDERGROUND FACILITIES

1. SCOPE AND APPLICATION

1.1 PURPOSE

The purpose of this Standard Operating Procedure is to prevent injury to workers and damage to subsurface structures (including tanks, pipe lines, water lines, gas lines, electrical service, etc.) during ground disturbance activities (including drilling, augering, sampling, use of direct-push technologies, excavation, trenching, concrete coring or removal, fence post installation, grading, or other similar operations).

1.2 LIMITATIONS

The procedures set forth in this document are the suggested procedures but may not be applicable to particular sites based on the site-specific considerations. The Project Manager is responsible for making a site-specific evaluation of each site to determine whether the Subsurface/Clearance Procedures should be utilized or require modification. If safety or other site-specific considerations require a modified or different procedure, the Project Manager should review the modified procedure with the Business Unit Director, Profit Center Manager, or Senior Technical Reviewer.

1.3 SCOPE

This procedure provides minimum guidance for subsurface clearance activities, which must be followed prior to and during ground disturbance activities at EA project sites. Even after completing the subsurface clearance activities required in this procedure, all ground disturbance activities should proceed with due caution.

Deviations from this procedure may be provided on an exception basis for specific situations, such as underground storage tank systems removals, verified aboveground/overhead services/lines, undeveloped land/idle facilities, shallow groundwater conditions, soil stability, or well construction quality assurance/quality control concerns, etc.

EA or its subcontractors are responsible for, and shall ensure that, all ground disturbance activities are completed safely, without incident, and in accordance with applicable federal, state, and local regulations.

This procedure shall not override any site-specific or consultant/contractor procedures that are more stringent or provide a greater degree of safety or protection of health or the environment.

2. PROCEDURES

The EA Project Manager or his designee must complete the Subsurface Clearance Procedure Checklist (Appendix A) in conjunction with the following procedures. The checklist must be completed before initiating any ground disturbance activities. The completed checklist must be submitted to the appropriate team individuals, subcontractors, and/or the client and included in the project files.

2.1 SAFETY

A Health and Safety Plan must be available onsite and followed by all contractors and subcontractors.

All work areas shall be defined and secured with safety cones, safety tape, construction fence, other barriers, or signs as appropriate.

Site work permits must be obtained as required by site procedures. Based on site conditions or classification, the use of intrinsically-safe equipment may be required.

To ensure the safety of all onsite personnel and subsurface structure integrity, consideration should be given to de-energizing and locking out selected site utilities or temporarily shutting down a portion of or the entire facility.

2.2 PREPARATION TASKS

Objective—To gather all relevant information about potential subsurface structures prior to the actual site visit.

2.2.1 Obtain Permits and Site Access

The consultant/contractor is responsible for following all applicable laws, guidance, and approved codes of practice; obtaining all necessary permits and utility clearances; and securing site access permission.

2.2.2 Historic Site Information

Obtain most recent as-built drawings and/or site plans (including underground storage tank, product, and vent lines) as available.

NOTE: As-built drawings may not accurately depict the locations and depths of improvements and subsurface structures and should, therefore, not be **solely** relied upon.

EA should obtain any other site information such as easements, right-of-ways, historical plot plans, fire insurance plans, tank (dip) charts, previous site investigations, soil surveys, boring logs, and aerial photographs, etc. as relevant to the planned ground disturbance activities.

Where applicable, EA should also contact contract personnel who may have historic site knowledge.

2.2.3 Mark-Outs

Objective—To identify location of subsurface structures on surface.

EA must ensure that a thorough mark-out at the site is completed to locate electrical, gas, telephone, water, sewer, low voltage electric lines, product delivery pipelines, fiber optic, and all other subsurface utilities/services.

- Where available, public utility companies must be contacted to identify underground utilities. (This can be accomplished through the One-Call system in most instances.)

- In addition, where available and warranted by site conditions, a private utility/pipeline mark-out company should be contracted to perform an electronic subsurface survey to identify the presence of suspected hazardous or critical underground utilities and subsurface structures. In some cases, this is necessary to confirm public utility mark-outs in the vicinity of planned ground disturbance activities.

EA will review all available site plan subsurface information with the private mark-out company to assist in locating utilities and other subsurface structures.

NOTE: Mark-outs may not accurately depict the exact locations of improvements and subsurface structures and should, therefore, not be **solely** relied upon.

Where possible, EA personnel are encouraged to be onsite at the time of subsurface mark-outs. This is to ensure accuracy and understanding of subsurface structures identified and provides an opportunity to exchange information with mark-out company personnel regarding planned work activities.

Subsurface structures should be marked throughout the entire work area(s) with adequate materials (e.g., site conditions may require paint and tape/flags). Ground disturbance activities must be started within 30 days of mark-out, unless local ordinances specify a shorter time period. If activities are not started within required time period or markings have faded, mark-outs must be redone.

EA personnel will record time and date of mark-out request and list all companies contacted by the service and confirmation number. This should be available for review onsite and checked off after visual confirmation of markings.

2.2.4 Initial Site Visit

Objective—To compare the site plan to actual conditions based on information gathered in Procedures 2 and 3 above, obtain additional site information needed, and prepare a vicinity map.

EA will document all findings and update the site plan with this information. On third party sites, close coordination with the site owner's representatives for mark-outs, review of as-builts, and other information reviews should be conducted prior to work. Project Managers are encouraged to provide updated as-built information to the client.

In some regions, it may be more effective and efficient to conduct the site visit at the same time the contractor and drill rig are mobilized to the site. The inspection should include the following activities and may include others as determined by the consultant/contractor and the Project Manager.

2.2.5 Utilities

EA shall perform a detailed site walk-through for the purpose of identifying all aboveground indicators of subsurface utilities/services that may be leading to or from buildings within the planned work area. The inspection shall include, but not be limited to, the following:

- Utility mark-outs
- Aboveground utilities
- Area lights/signs
- Phones
- Drains
- Junction boxes
- Natural gas meters or connections
- Other utilities including: fire hydrants, on/below grade electrical transformers, splice cages, sewer lines, pipeline markers, cable markers, valve box covers, clean-outs/traps, sprinkler systems, steam lines (including insulated tanks that may indicate steam lines), and cathodic protection on lines/tanks
- Observe paving scars (i.e., fresh asphalt/concrete patches, scored asphalt/concrete).

NOTE: In many cases, the onsite location of low-voltage electrical lines and individual property water and sewer line branches may be approximated by using the following technique:

- Locate the entry/connection location at the facility building
- Attempt to identify utility connections for the mains (water sewer, etc.) by locating clean-outs, valve manways, etc. The location path of the utility is likely with the area between the main connection and facility building connection. Subsurface electrical line locations from the facility building to signs, lamps, etc. can be estimated with the same process.

2.2.6 Other Subsurface Systems

Some other subsurface systems to be cognizant of during subsurface activities include product delivery systems (i.e., at gas stations) and existing remediation systems.

2.2.7 Selection of Ground Disturbance Locations

EA will utilize the information collected to this point in combination with regulatory requirements and project objectives to select ground disturbance locations. Ground disturbance locations should also consider the location of overhead obstructions (e.g., power lines). Work at active gasoline retail locations must consider several special considerations that should be outlined in the site-specific safety and health plan.

2.2.8 Review of Selected Locations with the Client

EA will review the selected ground disturbance locations with the client. EA will not proceed with the subsurface activities until the plan has been discussed with the client. During execution of the project, subsurface activities are required outside of the area previously approved by the client. EA will submit these changes to the client for approval prior to execution.

2.2.9 Ground Disturbance Activity Sequence

EA will plan ground disturbance activities starting at the point farthest from the location of suspected underground improvements. This is done to determine the natural subsurface conditions and to allow EA site personnel to recognize fill conditions.

Experience has shown that the following warning signs may indicate the presence of a subsurface structure:

- Warning tape (typically indicative of underground services).
- Pea gravel/sand/non-indigenous material (typically indicative of tanks or lines).
- Red concrete (typically indicative of electrical duct banks).

- The abrupt absence of soil recovery in a hand auger. This could indicate pea gravel or sand that has spilled out of the auger. This may not be indicative in areas where native soil conditions typically result in poor hand auger recoveries.

- Any unexpected departure from the native soil or backfill conditions as established by prior onsite digging.

If any of these conditions is encountered by EA site personnel, digging should stop and the client should be contacted.

3. SUBSURFACE CLEARANCE METHODS

The method used to delineate the subsurface should be compatible with the inherent associated risk given the type of facility/property, soil stratigraphy, and the location of the ground disturbance activity, such that required delineation is obtained. It should be noted that in areas where there is paving, sufficient paving should be removed to allow clear visibility of the subsurface conditions during clearance activities. The following is a list of potential clearance methods that may be used on a job site:

- Vacuum digging
- Probing
- Hand digging
- Hand augering
- Post-hole digging.

EA personnel will evaluate the potential for electrical shock or fire/explosion for each subsurface disturbance project and will evaluate as necessary the use of non-conductive or non-sparking tools (i.e., fiberglass hand shovels, and thick electrically insulating rubber grips on hand augers or probes). The potential need for the use of non-conductive materials, electrical safety insulated gloves, and footwear will also be evaluated on a case-by-case basis.

3.1 SUBSURFACE CLEARANCE PROCEDURES FOR DRILLING, DIRECT-PUSH TECHNOLGY, AUGERING, FENCE POST INSTALLATION, OR OTHER BOREHOLE INSTALLATION ACTIVITIES

The area to be delineated will exceed the diameter of the largest tool to be advanced and sufficiently allow for visual inspection of any obstructions encountered.

3.2 SUBSURFACE CLEARANCE PROCEDURES FOR TRENCHING/ EXCAVATION ACTIVITIES

Appropriate subsurface clearance methods should be conducted along the length and width of the excavation at a frequency sufficient to ensure adequate precautions have been applied to the entire work area. The frequency and density of investigations will be based on site knowledge, potential hazards, and risks of the work area to surrounding locations (e.g., proximity to a residential area or school).

Whenever subsurface structures are exposed, EA will cease work and mark the area (e.g., flags, stakes, cross bracing) to ensure the integrity of these exposed structures is maintained during subsequent trenching/excavation/backfilling.

Uniform color codes for marking of underground facilities are provided in Appendix B.

Appendix A

Subsurface Clearance Procedure Checklist

Subsurface Clearance Procedure Checklist

Site Identification: _____

Project Consultant/Contractor: _____

Section 1: Safety, Preparation Tasks, and Mark-Outs

Activity	Yes	No	N/A	Comments including Justification if Response Is No or Not Applicable
Health and Safety Plan is available and all contractors and subcontractors are familiar with it.				
All applicable local, state, and federal permits have been obtained.				
Site access/permission has been secured.				
Most recent as-built drawings and/or site plans (including underground storage tank, product, and vent lines) obtained.				
Reviewed site information to identify subsurface structures relevant to planned site activities (easements, rights-of-way, historical plot plans, fire insurance plans, tank dip charts, previous site investigations, soil surveys, boring logs, aerial photographs, etc.).				
Utility mark-outs have been performed by public utility company(s). Mark-outs clear/visible.				
Subsurface structure mark-outs performed by private mark-out company. Mark-outs clear/visible.				
Additional Activities: Were dig locations reviewed with site representative?				

Section 2: Initial Site Visit and Selecting Ground Disturbance Locations

Activity	Yes	No	N/A	Comments including Justification if Response Is No or Not Applicable
Location of all aboveground indicators of subsurface utilities/services that may be leading to or from buildings within the planned work area has been identified.				
Location of utility mark-outs by all utility companies previously contacted has been identified within required time period.				
Location of all subsurface structure mark-outs by private mark-out company has been identified within required time period.				
Location of area lights/signs and associated subsurface lines identified.				
Location of all phones and associated subsurface lines identified.				
Location of all drains and associated interconnecting lines identified.				
Location of all electrical junction boxes and associated interconnecting lines identified				
Location of all natural gas meters or connections and all interconnecting lines identified				

Completed by: _____
Name

Signature: _____ _____
Company Date

Appendix B

Uniform Color Codes for Excavation

APWA UNIFORM COLOR CODE

WHITE - Proposed Excavation

PINK - Temporary Survey Markings

RED - Electric Power Lines, Cables, Conduit and Lighting Cables

YELLOW - Gas, Oil, Steam, Petroleum or Gaseous Materials

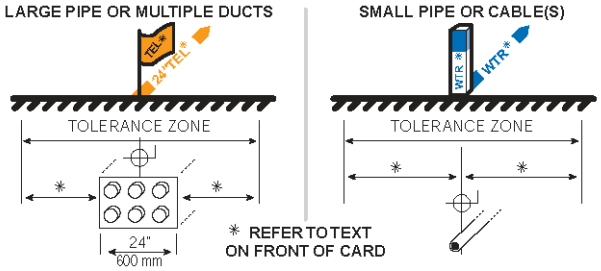
ORANGE - Communication, Alarm or Signal Lines, Cables or Conduit

BLUE - Potable Water

PURPLE - Reclaimed Water, Irrigation and Slurry Lines

GREEN - Sewers and Drain Lines

TYPICAL MARKING



Customize with your center's phone and address information

GUIDELINES FOR UNIFORM TEMPORARY MARKING OF UNDERGROUND FACILITIES

This marking guide provides for universal use and understanding of the temporary marking of subsurface facilities to prevent accidents and damage or service interruption by contractors, excavators, utility companies, municipalities or any others working on or near underground facilities.

ONE-CALL SYSTEMS
The One-Call damage prevention system shall be contacted prior to excavation.

PROPOSED EXCAVATION
Use white marks to show the location, route or boundary of proposed excavation. Surface marks on roadways do not exceed 1.5" by 18" (40 mm by 450 mm). The facility color and facility owner identity may be added to white flags or stakes.

USE OF TEMPORARY MARKING
Use color-coded surface marks (i.e., paint or chalk) to indicate the location or route of active and out-of-service buried lines. To increase visibility, color coded vertical markers (i.e., stakes or flags) should supplement surface marks. Marks and markers indicate the name, initials or logo of the company that owns or operates the line, and width of the facility if it is greater than 2" (50 mm). Marks placed by other than line owner/operator or its agent indicate the identity of the designating firm. Multiple lines in joint trench are marked in tandem. If the surface over the buried line is to be removed, supplementary offset markings are used. Offset markings are on a uniform alignment and clearly indicate the actual facility is a specific distance away.

TOLERANCE ZONE
Any excavation within the tolerance zone is performed with non-powered hand tools or non-invasive method until the marked facility is exposed. The width of the tolerance zone may be specified in law or code. If not, a tolerance zone including the width of the facility plus 18" (450 mm) measured horizontally from each side of the facility is recommended.

ADOPT UNIFORM COLOR CODE
The American Public Works Association encourages public agencies, utilities, contractors, other associations, manufacturers and all others involved in excavation to adopt the APWA Uniform Color Code, using ANSI standard Z535.1 Safety Colors for temporary marking and facility identification.

Rev. 4/99

Standard Operating Procedure No. 004
for
Sample Packing and Shipping

Prepared by

EA Engineering, Science, and Technology, Inc.
11019 McCormick Road
Hunt Valley, Maryland 21031

Revision 0
August 2007

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5. PRECAUTIONS 2

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for the packing and shipping of samples to the laboratory for analysis.

2. MATERIALS

The following materials may be required:

Clear tape	Plastic garbage bags
Custody seals	Sample documentation
Ice	Waterproof coolers (hard plastic or metal)
Metal cans with friction-seal lids (e.g., paint cans)	Zip-seal plastic bags
Packing material[1]	

3. PROCEDURE

Check cap tightness and verify that clear tape covers label and encircles container. Wrap sample container in bubble wrap or closed cell foam sheets. Enclose each sample in a clear zip-seal plastic bag.

Place several layers of bubble wrap, or at least 1 in. of vermiculite on the bottom of the cooler. Line cooler with open garbage bag, place all the samples upright inside a garbage bag, and tie the bag.

Double bag and seal loose ice to prevent melting ice from soaking the packing material. Place the ice outside the garbage bags containing the samples.

Pack shipping containers with packing material (closed-cell foam, vermiculite, or bubble wrap). Place this packing material around the sample bottles or metal cans to avoid breakage during shipment.

Enclose all sample documentation (i.e., Field Parameter Forms, chain-of-custodies) in a waterproof plastic bag and tape the bag to the underside of the cooler lid. If more than one cooler is being used, each cooler will have its own documentation.

Seal the coolers with signed and dated custody seals so that if the cooler were opened, the custody seal would be broken. Place clear tape over the custody seal to prevent damage to the seal.

1. Permissible packing materials are: (a) (non-absorbent) bubble wrap or closed cell foam packing sheets, or (b) (absorbent) vermiculite. Organic materials such as paper, wood shavings (excelsior), and cornstarch packing “peanuts” will not be used.

Refer to SOP Nos. 001, 002, 016, and 039.

Tape the cooler shut with packing tape over the hinges and place tape over the cooler drain. Ship all samples via overnight delivery on the same day they are collected if possible.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Any samples suspected to be of medium/high contaminant concentration or containing dioxin must be enclosed in a metal can with a clipped or sealable lid (e.g., similar to a paint can). Label the outer metal container with the sample number of the sample inside.

6. REFERENCES

U.S. Environmental Protection Agency (U.S. EPA). 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80.

U.S. EPA. 1990. Sampler's Guide to the Contract Laboratory Program. EPA/540/P-90/006, Directive 9240.0-06, Office of Emergency and Remedial Response, Washington, D.C. December.

U.S. EPA. 1991. User's Guide to the Contract Laboratory Program. EPA/540/O-91/002, Directive 9240.0-01D, Office of Emergency and Remedial Response. January.

Standard Operating Procedure No. 005
for
Field Decontamination

Prepared by

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1. SCOPE AND APPLICATION

All personnel or equipment involved in intrusive sampling, or which enter a hazardous waste site during intrusive sampling, must be thoroughly decontaminated prior to leaving the site to minimize the spread of contamination and prevent adverse health effects. This Standard Operating Procedure describes the normal decontamination of sampling equipment and site personnel.

2. MATERIALS

The following materials may be required:

0.01N HCl	Non-phosphate laboratory detergent (liquinox)
0.10N nitric acid	Plastic garbage bags
Aluminum foil or clean plastic sheeting	Plastic sheeting, buckets, etc. to collect wash water and rinsates
Approved water	Pressure sprayer, rinse bottles, brushes
High performance liquid chromatography (HPLC)-grade water[1]	Reagent grade alcohol[2]

3. PROCEDURE

3.1 SAMPLE BOTTLES

At the completion of each sampling activity, the exterior surfaces of the sample bottles must be decontaminated as follows:

- Ensure the bottle lids are on tight.
- Wipe the outside of the bottle with a paper towel to remove gross contamination.

3.2 PERSONNEL DECONTAMINATION

Review the project Health and Safety Plan for the appropriate decontamination procedures.

1. For the purposes of this Standard Operating Procedure, HPLC-grade water is considered equivalent to "deionized ultra filtered water," "reagent-grade distilled water," and "deionized organic-free water." The end product being water which is pure with no spurious ions or organics to contaminate the sample. The method of generation is left to the individual contractor.
2. For the purposes of this Standard Operating Procedure, the term "reagent grade alcohol" refers to either pesticide grade isopropanol or reagent grade methanol.

3.3 EQUIPMENT DECONTAMINATION

3.3.1 Water Samplers

3.3.1.1 Bailers

After each use, polytetrafluoroethelyne (PTFE) double check valve bailers used for groundwater sampling will be decontaminated as follows:

- Discard all ropes used in sampling in properly marked sealable container, or as directed by the Health and Safety Plan. NOTE: No tubing is to be used in conjunction with a bailer in collecting samples.
- Scrub the bailer to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate detergent.
- Rinse off detergent three times with approved water.
- Rinse bailer with reagent grade alcohol.
- Rinse bailer three times with HPLC-grade water.
- Rinse bailer with 0.10N nitric acid solution.
- Rinse bailer three times with HPLC-grade water.
- Allow bailer to air dry.[3]
- Wrap bailer in aluminum foil or clean plastic sheeting, or store in a clean, dedicated polyvinyl chloride or PTFE storage container.
- Dispose of used decontamination solutions with drummed purge water.
- Rinse bailer with HPLC-grade water immediately prior to re-use.

3.3.1.2 Pumps

Submersible pumps will be decontaminated as follows:

3. If the bailer has just been used for purging and is being decontaminated prior to sampling, do not air dry. Double rinse with HPLC-grade water and proceed to collect samples.

- Scrub the exterior of the pump to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate detergent. (Steam cleaning may be substituted for detergent scrub.)

- Calculate the volume of pump plus any tubing which is not disposable and not dedicated to a single well. Pump three volumes of non-phosphate laboratory detergent solution to purge and clean the interior of the pump.

- Rinse by pumping no less than nine volumes of approved water to rinse.

- Rinse pump exterior with reagent grade alcohol.

- Rinse pump exterior with HPLC-grade water.

- Allow pump to air dry.

- Wrap pump in aluminum foil or clean plastic sheeting, or store in a clean, dedicated polyvinyl chloride or PTFE storage container.

- Prior to reusing pump rinse exterior again with HPLC-grade water. (Double rinse in Bullet 5 above may be substituted for this step).

3.3.1.3 Dip Samplers

All dip samplers, whether bucket, long-handled, or short-handled, will be decontaminated in the same manner as provided in Section 3.3.1.1.

3.3.1.4 Labware

Labware, such as beakers, which are used to hold samples for field measurements, water chemistry, etc. will be decontaminated according to the procedures in Section 3.3.1.1.

3.3.1.5 Water Level Indicators

Electric water level indicators, weighted measuring tapes, or piezometers used in the determination of water levels, well depths, and/or non-aqueous phase liquid levels will be decontaminated in accordance with Section 3.3.1.1. Clean laboratory wipes may be substituted for brushes. Tapes, probes, and piezometers should be wiped dry with clean laboratory wipes, and coiled on spools or clean plastic sheeting rather than allowed to air dry.

3.3.2 Solid Materials Samplers

Solid materials samplers include soil sampling probes, augers, trowels, shovels, sludge samplers, and sediment samplers, which will be decontaminated as follows:

- Scrub the sampler to remove gross (visible) contamination, using appropriate brush(es), approved water, and non-phosphate laboratory detergent.
- Rinse off detergent with approved water.
- Rinse sampler with reagent grade alcohol.
- Rinse sampler with HPLC-grade water.
- For non-metallic samplers only, rinse sampler with 0.10N nitric acid solution.
- For non-metallic samplers only, rinse sampler with HPLC-grade water.
- Allow sampler to air dry.
- Wrap sampler in aluminum foil clean plastic sheeting, or store in a new zipseal bag (size permitting) or clean, dedicated polyvinyl chloride or PTFE storage container.
- Dispose used decontamination solutions properly according to the site-specific Health and Safety Plan.
- Rinse sampler with HPLC-grade water immediately prior to re-use.

3.3.3 Other Sampling and Measurement Probes

Soil gas sampling probes will be decontaminated as solids sampling devices.

Temperature, pH, conductivity, redox, and dissolved oxygen probes will be decontaminated according to manufacturer's specifications. If no such specifications exist, remove gross contaminant and triple rinse probe with HPLC-grade water. A summary of the decontamination procedures to be used must be included in the instrument-specific standard operating procedure.

Measuring tapes that become contaminated through contact with soil during field use will be decontaminated as follows:

- Wipe tape with a clean cloth or laboratory wipe that has been soaked with non-phosphate laboratory detergent solution to remove gross contamination. Rinse cloth in the solution and continue wiping until tape is clean.
- Wipe tape with a second clean, wet cloth (or laboratory wipe) to remove soap residues.
- Dry tape with a third cloth (or laboratory wipe) and rewind into case, or re-coil tape.

3.3.4 Drilling Rigs and Other Heavy Equipment

All drilling rigs and associated equipment such as augers, drill casing, rods, samplers, tools, recirculation tank, and water tank (inside and out) will be decontaminated prior to site entry after over-the-road mobilization and immediately upon departure from a site after drilling a hole. Supplementary cleaning will be performed prior to site entry when there is a likelihood that contamination has accumulated on tires and as spatter or dust enroute from one site to the next.

- Place contaminated equipment in an enclosure designed to contain all decontamination residues (water, sludge, etc.).
- Steam clean equipment until all dirt, mud, grease, asphaltic, bituminous, or other encrusting coating materials (with the exception of manufacturer-applied paint) have been removed.
- Water used will be taken from an approved source.
- Containerize in 55-gal drums; sample; characterize; and, based on sample results, dispose of all decontamination residues properly.

Other heavy equipment includes use of backhoes, excavators, skid steers, etc. If heavy equipment is utilized during field activities, i.e., a backhoe for test pitting, the bucket should not come in contact with soil to be sampled. If the bucket contacts the soil to be sampled, then it should be decontaminated between sample locations, following the same procedures as listed above for a drill rig.

3.3.5 High Performance Liquid Chromatography-Grade Water Storage

Dedicated glass storage containers will be used solely for dispensing HPLC-grade water. New HPLC-grade water containers will be decontaminated as follows:

- Clean with tap water from approved source and non-phosphate laboratory detergent while scrubbing the exterior and interior of the container with a stiff-bristled brush.
- Rinse thoroughly with approved water.
- Rinse with 0.01N nitric acid.
- Rinse with approved water.
- Rinse thoroughly with HPLC-grade water.
- Fill clean container with HPLC-grade water. Cap with one layer of PTFE-lined paper and one layer of aluminum foil. Secure cap with rubber band and date the container.

Used HPLC-grade water containers will be decontaminated as follows:

- Clean the exterior with tap water from an approved source, non-phosphate laboratory detergent, and a stiff-bristled brush.
- Rinse the exterior thoroughly with HPLC-grade water.
- Rinse the interior twice with pesticide-grade isopropanol.
- Rinse interior thoroughly with HPLC-grade water.
- Fill clean container with HPLC-grade water. Cap with one layer of PTFE-lined paper and one layer of aluminum foil. Secure cap with rubber band and date the container.

3.3.6 Ice Chests and Reusable Shipping Containers

- Scrub exterior/interior with approved brush and liquinox detergent.
- Rinse off detergent three times with approved water.
- Let air dry and properly store until re-use.

NOTE: If container/ice chest is severely contaminated, clean as thoroughly as possible, render unusable, and properly dispose.

4. MAINTENANCE

HPLC-grade water will be stored only in decontaminated glass containers with aluminum foil lids as stipulated above. The water may not be stored for more than nor used more than 3 days after manufacture.

HPLC-grade water will be manufactured onsite. An approved tap water source will be used as the influent to the system. Procedures for system setup, operation, and maintenance will conform to manufacturer's specifications.

5. PRECAUTIONS

Dispose of all wash water, rinse water, rinsates, and other sampling wastes (tubing, plastic sheeting, etc.) in properly marked, sealable containers, or as directed by the Health and Safety Plan.

Once a piece of equipment has been decontaminated, be careful to keep it in such condition until needed.

Do not eat, smoke, or drink onsite.

6. REFERENCES

Site-specific Health and Safety Plan.

Standard Operating Procedure No. 016
for
Surface Water, Groundwater, and Soil/Sediment Field Logbooks

Prepared by

EA Engineering, Science, and Technology, Inc.
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Revision 0
August 2007

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for recording surface water, groundwater, soil/sediment sampling information, instrument calibration data, and data from hydrologic testing in the field logbooks. Acceptable field logbooks are: bound, unprinted books such as a surveyor's field book, or a federal supply service No. 7530-00-222-3525 record book (or equivalent); or they may be company-proprietary, pre-printed forms bound into a field logbook. Example forms are provided herein. Alternate, equivalent forms are acceptable.

2. MATERIALS

The following material may be required: applicable field logbook and indelible ink pen.

3. PROCEDURE

Information pertinent to soil/sediment, groundwater, or surface water sampling will be recorded in the appropriate logbook. Each page/form of the logbook will be consecutively numbered. Entries will be made in indelible ink. Corrections will consist of line-out deletions that are initialed and dated. If using carbon paper or self-duplicating forms, before entering data in logbook, insert a sheet protector between form sets to isolate first blank form from remaining forms.

3.1 SOIL/SEDIMENT LOGBOOK (Requires Figures SOP016-1 and SOP016-3)

3.1.1 Field Parameter Form (Items on Figures SOP016-1 and SOP016-2)

1. HIGH CONCENTRATION EXPECTED?: Answer "Yes" or "No."

2. HIGH HAZARD?: Answer "Yes" or "No."

3. SITE: Record the complete name of the site.

4. AREA: Record the area designation of the sample site.

5. INST CODE: Record the 2-letter installation code appropriate for the installation or site. Correct abbreviations can be found on Pages 3-6 of the IRDMS User's Guide for chemical data entry.

6. FILE NAME: Record "CSO" for a soil sample or "CSE" for a sediment sample.

7. SITE TYPE: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on Pages 18-21 of the IRDMS User's Guide for chemical data entry. This entry must match the Site Type on the map file form.

8. SITE ID: Record a code up to 10 characters or numbers which is unique to the site.

9. FIELD SAMPLE NUMBER: Record a code specific for the sample.

10. DATE: Enter the date the sample was taken.

11. TIME: Enter the time (12-hour or 24-hour clock acceptable as long as internally consistent) the sample was taken.

12. AM PM: Circle "AM" or "PM" to designate morning or afternoon (12-hour clock).

13. SAMPLE PROG: Record "GQA" (Groundwater Quality Assessment) or other appropriate sample program.

14. DEPTH (TOP): Record the total depth sampled.

15. DEPTH INTERVAL: Record the intervals at which the plug will be sampled.

16. UNITS: Record the units of depth (feet, meters)

17. SAMPLE MEASUREMENTS: Check the appropriate sampling method.

18. CHK: Check off each container released to a laboratory.

19. ANALYSIS: Record the type of analysis to be performed on each sample container.

20. SAMPLE CONTAINER: Record the sample container type and size.

21. NO.: Record the number of containers.

22. REMARKS: Record any remarks about the sample

23. TOTAL NUMBER OF CONTAINERS FOR SAMPLE: Record the total number of containers.

24. SITE DESCRIPTION: Describe the location where the sample was collected.

25. SAMPLE FORM: Record the form of the sample (i.e., clay, loam, etc.) using The Unified Soil Classification System.

26. COLOR: Record the color of the sample as determined from standard Munsell Color Charts.

27. ODOR: Record the odor of the sample or "none." See SOP No. 001 Section 5.

28. PID (HNu): Record the measured PID (HNu) values.

29. UNUSUAL FEATURES: Record anything unusual about the site or sample.

30. WEATHER/TEMPERATURE: Record the weather and temperature.

31. SAMPLER: Record your name.

3.1.2 Map File Form (Figure SOP016-3)

1. The map file logbook form will be located on the reverse of the field parameter logbook form, or on an adjoining page of the field logbook (if level book is used).

2. SITE ID: Record the Site ID from the field parameter form.

3. POINTER: Record the field sample number for the sample being pointed to.

4. DESCRIPTION/MEASUREMENTS: Describe the location where the sample was taken, along with distances to landmarks.

5. SKETCH/DIMENSIONS: Diagram the surroundings and record the distances to landmarks.

6. MAP REFERENCE: Record which U.S. Geological Survey Quad Map references the site.

7. COORDINATE DEFINITION: Write the compass directions the X- and Y-Coordinates of the map run.

8. COORDINATE SYSTEM: Write “UTM” (Universal Transverse Mercator).

9. SOURCE: Record the 1-digit code representing the Map Reference.

10. ACCURACY: Give units (e.g., write “1-M” for 1 meter).

11. X-COORDINATE: Record the X-Coordinate of the sample site location.

12. Y-COORDINATE: Record the Y-Coordinate of the sample site location.

13. UNITS: Record the units map sections are measured in.

14. ELEVATION REFERENCE: Record whether topography was determined from a map or a topographical survey.

15. ELEVATION SOURCE: Record the 1-digit code representing the elevation reference.

16. ACCURACY: Record the accuracy of the map or survey providing the topographical information.

17. ELEVATION: Record the elevation of the sampling site.

18. UNITS: Write the units in which the elevation is recorded.

19. SAMPLER: Write your name.

3.2 SURFACE WATER LOGBOOK (Requires Figures SOP016-2 and SOP016-3)

3.2.1 Field Parameter Form (Items Unique to Figure SOP016-3)

1. CAL REF: Record the calibration reference for the pH meter.

2. pH: Record the pH of the sample.

3. TEMP: Record the temperature of the sample in degrees Celsius.

4. COND: Record the conductivity of the water.

5. For all other sections, see Section 3.2.1.

3.3 GROUNDWATER SAMPLING LOGBOOK (Requires Figures SOP016-2, SOP016-3, and SOP016-4)

3.3.1 Field Parameter Form (Items on Figure SOP016-4)

1. WELL NO. OR ID: Record the abbreviation appropriate for where the sample was taken. Correct abbreviations can be found on Pages 18-21 of the IRDMS User's Guide for chemical data entry.

2. SAMPLE NO.: Record the reference number of the sample.

3. WELL/SITE DESCRIPTION: Describe the location where the sample was taken, along with distances to landmarks.

4. X-COORD and Y-COORD: Record the survey coordinates for the sampling site.

5. ELEV: Record the elevation where the sample was taken.

6. UNITS: Record the units the elevation was recorded in.

7. DATE: Record the date in the form MM/DD/YY.

8. TIME: Record the time, including a designation of AM or PM.

9. AIR TEMP.: Record the air temperature, including a designation of C or F (Celsius or Fahrenheit).

10. WELL DEPTH: Record the depth of the well in feet and inches.

11. CASING HT.: Record the height of the casing in feet and inches.

12. WATER DEPTH: Record the depth (underground) of the water in feet and inches.

13. WELL DIAMETER: Record the diameter of the well in inches.

14. WATER COLUMN HEIGHT: Record the height of the water column in feet and inches.

15. SANDPACK DIAM.: Record the diameter of the sandpack. Generally, this will be the same as the bore diameter.

16. EQUIVALENT VOLUME OF STANDING WATER: Use one of the following equations, to determine one equivalent volume (EV):

1 EV = Volume in casing + volume in saturated sand pack. Or to restate:

$$1\ EV = (BR_w^2 h_w + 0.30B(R_s^2 - R_w^2)h_s) * (0.0043)$$

where

R_s = Radius of sandpack in inches
R_w = Radius of well casing in inches
h_s = Height of sandpack in inches
h_w = Water depth in inches

0.0043 = gal/in.^{3}
and filter pack porosity is assumed as 30 percent

— OR —

$$\text{Volume in casing} = (0.0043\ \text{gal/in.}^3)(B)(12\ \text{in./ft})(R_c^2)(W_h)$$

where

R_c = Radius of casing in inches
W_h = Water column height in feet

$$\text{Vol. in sandpack} = (0.0043\ \text{gal/in.}^3)(B)(12\ \text{in./ft})(R_b^2 - R_c^2)(W_h)(0.30)$$

(if W_h is less than the length of the sandpack),

— PLUS —

Vol. in sandpack = $(0.0043\ gal/in.^3)(B)(12\ in./ft)(R_b^2 - R_c^2)(S_h)(0.30)$

(if W_h is greater than the length of the sandpack).

where
R_b = Radius of the borehole
S_h = Length of the sandpack.

Show this calculation in the comments section.

17. VOLUME OF BAILER OR PUMP RATE: Record bailer volume or pump rate.

18. TOTAL NUMBER OF BAILERS OR PUMP TIME: Record the number of bailers required to remove 3 equivalent volumes (EV) of water from the well or the total purge time and volume as applicable.

19. WELL WENT DRY? Write “YES” OR “NO.”

20. NUMBER OF BAILERS OR PUMP TIME: Record the number of bailers or pump time which made the well go dry.

21. VOLUME REMOVED: Record the volume of water (gal) removed before the well went dry.

22. RECOVERY TIME: Record the time required for the well to refill.

23. PURGE AGAIN?: Answer “YES” or “NO.”

24. TOTAL VOL. REMOVED: Record the total volume of water (in gal) removed from the well.

25. CAL REF.: Record the calibration reference for the pH meter.

26. TIME: Record time started (INITIAL T[0]), 2 times DURING the sampling and the time sampling ended (FINAL).

27. pH: Record the pH at start of sampling (INITIAL), twice DURING the sampling and at the end of sampling (FINAL).

28. TEMP: Record the water temperature (Celsius) at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

29. COND: Record the conductivity of the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

30. D.O.: Record the dissolved oxygen level in the water at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

31. TURBIDITY: Record the readings from the turbidity meter (nephelometer) and units at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

32. ORD: Record the oxidation/reduction (RedOx) potential of the water sample at the start of sampling, twice DURING the sampling and at the end of sampling (FINAL).

33. HEAD SPACE: Record any positive readings from organic vapor meter reading taken in well headspace prior to sampling.

34. NAPL: Record the presence and thickness of any non-aqueous phase liquids (light or dense)

35. COMMENTS: Record any pertinent information not already covered in the form.

36. SIGNATURE: Sign the form.

3.4 FIELD CALIBRATION FORMS (Maintained as a separate logbook, or incorporated into sampling logbooks)

3.4.1 Items on Figure SOP016-5

1. Record time and date of calibration. Note whether 12- or 24-hour clock was used.

2. Record calibration standard reference number.

3. Record meter I.D. number

4. Record initial instrument reading, recalibration reading (if necessary), and final calibration reading on appropriate line.

5. Record value of reference standard (as required).

6. COMMENTS: Record any pertinent information not already covered on form.

7. SIGNATURE: Sign form.

3.5 GROUNDWATER HYDROLOGY TESTS LOGBOOK (Must include Figures SOP016-6 and SOP016-7 and/or SOP016-8, OR SOP016-9 or SOP016-10)

3.5.1 Field Permeability Test Data Sheet (Items on Figures SOP016-6)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

4. PROJECT NO.: Record the contractor assigned project number or the contract number.

5. LOCATION: Specific location

6. CLIENT: Agency or company with the contract under which the work is being performed.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

9. TEST TYPE: Short description of the type of test to be performed.

10. RISING/FALLING HEAD WITH SLUG: Check if the test involved the insertion/removal of and inert object.

11. RISING/FALLING HEAD WITHOUT SLUG: Check if the test involved the addition/removal of a quantity of water.

12. START DATE: Date on which the test was begun.

13. CLOCK TIME: Time each datum (depth to groundwater level) is collected. Note whether 12- or 24-hour clock was used.

14. ELAPSED TIME: Time since the last datum was collected.

15. DEPTH TO GWL (ft): Depth to the top of the groundwater table (Groundwater Level) as measured by manual methods.

16. REC. (ft): Water level as reported by transducer/datalogger (this is the depth of water above the transducer.

17. TIME: Time the discharge rate check was begun (addition or removal of water method). Note whether 12- or 24-hour clock was used.

18. FLOW METER (Addition or removal of water method): The amount of water added or removed as registered by the flowmeter, in gal of liters.

19. DISCHARGE RATE: Flowmeter reading divided by time interval (gal/min or liters/min).

20. SIGNATURE: The person completing this form must sign the form at the end of the test.

21. DATE: Date the form was signed.

3.5.2 Groundwater Levels – Single Well (Items on Figure SOP016-7)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

Well Data

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: Remedial investigation wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

15. DATE: Date of each water level reading

16. TIME: Time of each water level reading. Note whether 12- or 24-hour clock was used.

17. ELAPSED TIME: Time since test was begun.

18. DEPTH TO WATER: Measured depth to the groundwater table.

19. WATER ELEVATION: Elevation of the top of the groundwater table (use datum listed above).

20. MEAS. METH.: Method used to measure the water level in the well (see abbreviation key at the bottom of the data sheet).

21. TAPE NO.: The unique identification number of the traceable standard tape used to calibrate the measuring device.

22. WELL STATUS: Condition of the well at the time of measuring (see abbreviation key at the bottom of the data sheet).

23. REMARKS: Any additional pertinent comments not specifically required above.

24. INITIALS: Initials of person completing this data entry.

25. ABBREVIATION KEYS: Self explanatory.

26. SIGNATURE: The person completing this form must sign the form at the end of the test.

27. DATE: Date the form was signed.

3.5.3 Groundwater Levels – Single Well (Items on Figure SOP016-8)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

WELL DATA

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: All Rhode Island wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

15. DATALOGGER: This section is record of pertinent datalogger information.

16. MANUFACTURER: Record the manufacturer/brand name as stated on the datalogger.

17. MODEL: Enter the model number of the datalogger.

18. S/N: Enter the serial number of this datalogger.

19. TAG PROGRAMMED IN LOGGER: What is the identifier used in the datalogger's program to indicate that this unit was used to record a given data set?

20. TRANSDUCER: This section is a listing of pertinent information about the transducer used.

21. MANUFACTURER: Record the manufacturer/brand name as stated on the transducer.

22. MODEL: Enter the model number of the transducer.

23. S/N: Enter the serial number of this transducer.

24. INPUT/UNITS: What are the units this transducer uses?

25. RANGE: Record the pressure or depth range over which this transducer is certified.

CALIBRATION

26. PRESSURE RATING: This is taken from the manufacturer's specifications for a given transducer. (Usually in psi, or kpa).

27. "SUBMERGENCE = ___ (V) / (MV)": Record the voltage returned by the transducer at a given depth of submergence. Indicate whether the reading is in volts (v), or millivolts (mv).

28. VOLUME WATER ADDED/REMOVED: (Applicable if inert object insertion/removal method was not employed.) Record the volume of water added to or removed from the well.

29. DISCHARGE RATE: If z (above) is filled, enter the rate at which this water was added or removed.

30. INITIAL WATER LEVEL (ft): Enter the water level in the well at the beginning of the test.

31. PRESSURE TRANSDUCER SUBMERGENCE: Record the depth to which the transducer is submerged at the beginning of the test and the depth to the transducer at the end if the test. All depths will be recorded to the nearest 0.01 ft.

32. TIME: Record the time the test is begun and ended. Note whether 12- or 24-hour clock was used.

33. OBSERVED CHANGES IN ADJACENT WELLS: Note any changes in water levels in nearby wells.

34. RESULTS RECORDED ON DISKETTE #: Tracking number of the diskette on which these data are archived.

35. DISKETTE FILE NAME: Name of the file(s).

36. SIGNATURE: The person completing this form must sign the form at the end of the test

37. DATE: Date the form was signed.

3.6 GROUNDWATER LEVELS – MULTIPLE WELLS (Items on Figure SOP016-9)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

5. LOCATION: Specific location.

6. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

7. CLIENT: Agency with the contract under which the work is being performed.

8. REMARKS: Any pertinent observations not specifically required above.

9. WELL: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

10. DATE: Date this measurement was made.

11. TIME: Time this measurement was made. Note whether 12- or 24-hour clock was used.

12. DEPTH TO WATER: Depth from MP to top of groundwater table.

13. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

14. MP ELEV.: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

15. MEAS. METH.: Method used to measure the water level in the well (see abbreviation key at the bottom of the data sheet).

16. REMARKS/MP: Describe the location and nature of the measuring point.

17. INITIALS: Initials of the person completing this form.

18. ABBREVIATION KEYS: Self explanatory.

19. SIGNATURE: The person completing this form must sign the form at the end of the test.

20. DATE: Date the form was signed.

3.7 GROUNDWATER LEVELS – DATALOGGERS (Items on Figure SOP016-10)

1. CONTRACTOR: Organization performing the test.

2. SEQ. #: Enter page number of this set of forms (page # of #).

3. PROJECT NO.: Record the contractor assigned project number or the contract number.

4. WELL #: Record the well number as it appears on the well completion tag, affixed to the protector casing or well completion records.

5. PROJECT NAME: Record the name assigned by the contractor's organization to the project.

6. LOCATION: Specific location.

7. FIELD PARTY CHIEF: Printed name of the person responsible for this particular field test.

8. CLIENT: Agency with the contract under which the work is being performed.

WELL DATA

9. STICKUP: Enter the length of well casing extending above the average ground surface at the base of the protective casing.

10. MEASURED UP(+)/DOWN(-) FROM: Describe the starting point for the previous measurement.

11. MP ELEVATION: Enter the elevation of the measuring point here. NOTE: This datum may require reference to tables and/or maps and may be added after completing the day's field work.

12. DATUM = MSL OR: Is the datum for the previous elevation Mean Sea Level? If not, what? Also tell whether it was derived from a map elevation (write "MAP") or survey data (write "SURVEY").

13. MEASURING POINT DESCRIPTION: Describe the point used as the origin for all down-hole (water table) measurements. NOTE: All Rhode Island wells are required to have a permanently marked reference (measuring) point (refer to SOP No. 019, Section 3.4).

14. REMARKS: Record any pertinent observations about the site/well conditions not specifically required in the preceding.

DATALOGGER (This section is a record of pertinent datalogger information)

15. MANUFACTURER: Record the manufacturer/brand name as stated on the datalogger.

16. MODEL: Enter the model number of the datalogger.

17. S/N: Enter the serial number of this datalogger.

18. TAG PROGRAMMED IN LOGGER: What is the identifier used in the datalogger's program to indicate that this unit was used to record a given data set?

TRANSDUCER (This section is a listing of pertinent information about the transducer used)

19. MANUFACTURER: Record the manufacturer/brand name as stated on the transducer.

20. MODEL: Enter the model number of the transducer.

21. S/N: Enter the serial number of this transducer.

22. INPUT/UNITS: What are the units this transducer uses?

23. RANGE: Record the pressure or depth range over which this transducer is certified.

CALIBRATION

24. PRESSURE RATING: This is taken from the manufacturer's specifications for a given transducer (usually in psi, or kpa).

25. "SUBMERGENCE = ___ (V) / (MV)": Record the voltage returned by the transducer at a given depth of submergence. Indicate whether the reading is in volts (v), or millivolts (mv).

26. DATE: Date of each water level reading

27. TIME: Time of each water level reading. Note whether 12- or 24-hour clock was used.

28. LOGGING TIME INTERVAL: Time since test was begun.

29. WL FEET BELOW MP: Measured depth to the groundwater table from measuring point.

30. SUBMERGENCE: Depth of water above the transducer.

31. MEAS.METHOD: What device/method was used to measure the water level.

32. TAPE NO.: Record the tape identification number.

33. TRANSDUCER MOVED?: Was the transducer moved since the last water level reading?

34. REMARKS: Any pertinent remarks not otherwise specified.

35. INITIALS:

DATA TRANSFER TO DISKETTE:

36. DATE: Date data were archived onto diskette.

37. TIME: Time stamp the computer assigns the data file.

38. FILE NAME: Name assigned the data file.

39. SOFTWARE USED FOR TRANSFER: Any special software, or computer operating system used to write the files to diskette. NOTE: If a "shareware" archiver which compresses files was used, and the archived file is not self-extracting, a copy of the unarchive program should be copied onto the diskette also.

40. OUTPUT FORMAT: What is the format of the output file? (DOS, UNIX, Binary, Compressed?)

41. INITIALS: Initials of the person who copied the data to diskette.

42. ABBREVIATION KEY: Self-explanatory.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

None.

6. REFERENCES

U.S. Environmental Protection Agency. 1984. User's Guide to the Contract Laboratory Program. July.

FIGURE SOP016-1
FIELD PARAMETER LOGBOOK
SOIL AND SEDIMENT SAMPLES

HIGH CONCENTRATION EXPECTED? HIGH HAZARD?

INSTALLATION/SITE _____ AREA _____

INST CODE FILE NAME _____

SITE TYPE SITE ID
FIELD SAMPLE NUMBER

DATE (MM/DD/YY) / / TIME AM PM SAMPLE PROG.

DEPTH (TOP) DEPTH INTERVAL UNIT _____

SAMPLING METHOD:

SPLIT SPOON AUGER SHELBY TUBE SCOOP OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE

DESCRIPTION OF SITE AND SAMPLE CONDITIONS
SITE DESCRIPTION:_____
SAMPLE FORM _____ COLOR _____ ODOR _____
PID (HNu) _____ UNUSUAL FEATURES _____
_____
WEATHER/TEMPERATURE
SAMPLER

HIGH CONCENTRATION EXPECTED? HIGH HAZARD?

FIGURE SOP016-2
FIELD PARAMETER LOGBOOK
GROUNDWATER AND SURFACE WATER SAMPLES

INSTALLATION/SITE AREA
INST CODE FILE NAME SITE TYPE
SITE ID FIELD SAMPLE NUMBER
DATE (MM/DD/YY) / / TIME AM PM SAMPLE PROG.
DEPTH (TOP) DEPTH INTERVAL UNITS

SAMPLING MEASUREMENTS

CAL REF.	pH	TEMPERATURE C	CONDUCTIVITY	OTHER

CHK	ANALYSIS	SAMPLE CONTAINER	NO.	REMARKS

TOTAL NUMBER OF CONTAINERS FOR SAMPLE

DESCRIPTION OF SITE AND SAMPLE CONDITIONS
SITE DESCRIPTION
SAMPLING METHOD
SAMPLE FORM COLOR ODOR
PID (HNu)
UNUSUAL FEATURES
WEATHER/TEMPERATURE _____ SAMPLER _____

FIGURE SOP016-3
MAP FILE LOGBOOK

SITE ID POINTER _____
DESCRIPTION/MEASUREMENTS
SKETCH/DIMENSIONS:

MAP REFERENCE
COORDINATE DEFINITION (X is Y is)
COORDINATE SYSTEM SOURCE ACCURACY
X-COORDINATE Y-COORDINATE UNITS
ELEVATION REFERENCE
ELEVATION SOURCE ACCURACY ELEVATION
UNITS

SAMPLER

FIGURE SOP016-4
MAP FILE AND PURGING LOGBOOK
GROUNDWATER SAMPLES

WELL COORD. OR ID SAMPLE NO._____
WELL/SITE
DESCRIPTION_____
_____
X-COORD. Y-COORD. _____ ELEV. UNITS
DATE ____/____/____ TIME AIR TEMP.

WELL DEPTH _____ ft _____ in. CASING HT. _____ ft _____ in.
WATER DEPTH _____ ft _____ in. WELL DIAMETER _____ in.
WATER COLUMN HEIGHT _____ ft _____ in. SANDPACK DIAM. _____ in.
EQUIVALENT VOLUME OF STANDING WATER _____ (gal) (L)
VOLUME OF BAILER _____ (gal) (L) or PUMP RATE _____ (gpm) (lpm)
TOTAL NO. OF BAILERS (5 EV)_____ or PUMP TIME _____ MIN.
WELL WENT DRY? [Yes] [No] NUM. OF BAILERS _____ or PUMP TIME_____MIN
VOL. REMOVED _____ (gal) (L) RECOVERY TIME _____ MIN
PURGE AGAIN? [Yes] [No] TOTAL VOL. REMOVED _____ (gal) (L)

Date and Time	Quantity Removed	Time Required	pH	Cond	Temp	ORD	Turb	DO	Character of water (color/ clarity/odor/partic.)
(before)									
(during)									
(during)									
(during									
(after)									

COMMENTS:_____
_____
_____
_____

_____
SIGNATURE

FIGURE SOP016-5
FIELD CALIBRATION: pH, CONDUCTIVITY, TEMPERATURE, TURBIDITY, OXIDATION-REDUCTION POTENTIAL, AND DISSOLVED OXYGEN METERS

INITIAL CALIBRATION	FINAL CALIBRATION
DATE:	DATE:
TIME:	TIME:

pH METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

pH STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
7.0			
10.0			
4.0			

CONDUCTIVITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

COND. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

TEMPERATURE METER CALIBRATION

METER ID _____

TEMP. STANDARD	INITIAL READING	RECALIB. READING	FINAL READING
ICE WATER			
BOILING WATER			
OTHER			

FIGURE SOP016-5 *(continued)*

TURBIDITY METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

ORD METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

DISSOLVED OXYGEN METER CALIBRATION

CALIBRATION STANDARD REFERENCE NO: _____

METER ID _____

STANDARD	INITIAL READING	RECALIB. READING	FINAL READING

COMMENTS:_____

_____

_____

_____

_____

SIGNATURE

FIGURE SOP016-6
FIELD PERMEABILITY TEST DATA SHEET

Contractor: **Seq. #** /

Project Name:	Project #:
Location:	Client:
Party Chief:	Contractor:
Observation Well:	
Test Type: Rising/Falling Head w/Slug	Rising/Falling Head w/out Slug
Start Date:	

								Discharge Rate		
Clock Time	Elapsed Time (min)	Depth to GWL (ft)	Rec (ft)	Clock Time	Elapsed Time	Depth to GWL (ft)	Rec (ft)	Time	Flow Meter	Discharge Rate

Signature: _____ **Date:** _____

FIGURE SOP016-7
GROUNDWATER LEVELS – SINGLE WELL

Contractor: **Seq. #** /

Project No.:
Project Name:
Field Party Chief:

WELL DATA:

Stickup: (ft)
MP Elevation:
Area:

Datum = MSL or:

Measuring Point Description:

Datalogger:
Manufacturer: Model: S/N:
Tag No. Programmed in Logger:

Transducer: Manufacturer: Model: S/N:
Input/Units: Range:

Calibration:
Pressure Rating:
0 ft submergence = (v) / (mv) ft submergence = (v) / (mv)

Volume Water Added/Removed:
Discharge Rate:
Initial Water Level (ft):

Pressure Transducer Submergence
Initial (ft): Final(ft): Time:Start: End:
Observed Changes in Adjacent Wells:

Results Recorded on Diskette #:
Diskette File Name:

Signature: _____ **Date:** _____

FIGURE SOP016-8
GROUNDWATER LEVELS – MULTIPLE WELLS

Contractor: **Seq. #** /

Project No.:
Project Name:
Field Party Chief:

WELL DATA:

Stickup: (ft)
MP Elevation:

Measuring Point Description:
Remarks:
Well No.:
up (+)/down (-) from:
Datum = MSL or:

Date	Time	Elapsed Time	Depth to Water	Water Elevation	Meas. Meth.	Tape No.	Well Status	Remarks	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently pumped
NP = Nearby well pumping
NRP = Nearby well recently
X = Obstructed

Signature: _____ **Date:** _____

FIGURE SOP016-8 *(continued)*

Contractor: **Seq. #** /

Project No.:
Project Name: Location:
Field Party Chief: Client:

Well	Date	Time	Depth to Water	Stickup	MP Elev.	Meas. Meth.	Tape No.	Remarks/MP	Initials

Measurement Method:
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Well Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently pumped
NP = Nearby well pumping
NRP = Nearby well recently
X = Obstructed

FIGURE SOP016-9
GROUNDWATER LEVELS DATALOGGERS

Contractor
Project No.: **Well No.:**
Project Name: **Site:**
Field Party Chief: **Area:**

WELL DATA: Stickup: **(ft)** **up (+)/down (-) from:**

Measuring Point Description:
Remarks:

Datalogger:
Manufacturer: Model: S/N:
Tag No. Programmed in Logger:
Transducer: Manufacturer: Model: S/N:
Input/Units: Range:
Calibration: Pressure Rating:
0 ft submergence = (v) / (mv) ft submergence = (v)

Logging	Date	Time	Logging Time Interval	WL, ft Below MP	Submergence (logger reading)	Meas. Method	Tape No.	Well Status	Transducer Moved	Remarks	Initials
Start											
Stop											
Start											
Stop											

Data Transfer to Disk

Date	Time	File Name	Software Used for Transfer	Output Format	Initials

Measurement Method: **Well**
A = Airline
C = Chalk and tape
E = Electric tape
T = Tape with popper
X = Other (describe in remarks)

Status:
D = Dry
F = Flowing
P = Pumping
RP = Recently
NP = Nearby well pumping
NRP = Nearby well recently pumped
X = Obstructed

Signature **Date**

Standard Operating Procedure No. 021
for
Sediment Sampling

Prepared by

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Revision: 1
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1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) delineates protocols for sampling sediments from streams, rivers, ditches, lakes, ponds, lagoons, and marine and estuarine systems.

EA recognizes that other protocols have been developed that meet the criteria of quality and reproducibility. Clients may have their own sediment sampling protocols which may contain methodologies and procedures that address unique or unusual site-specific conditions or may be in response to local regulatory agency requirements. In such cases, EA will compare its and the client's protocols. The goal is to provide the client with the most quality; therefore, if the client's protocols provide as much or more quality assurance than EA's protocols for the particular site or project, EA will adopt those particular protocols and this SOP will be superseded in those respects. If EA is required to implement the client's protocols in lieu of EA's protocols, EA will make the client formally aware of any concerns regarding differences in protocols that might affect data quality and will document such concerns in the project file.

2. PROCEDURES

The water content of sediment varies. Sediments range from soft to dense and fine to rocky. A variety of equipment may be necessary to obtain representative samples, even at a single site. Factors to consider in selecting the appropriate sampling equipment include sample location (edge or middle of the waterbody), depth of water and sediment, grain size, water velocity, and analytes of interest.

3. GENERAL PROCEDURES

1. Surface water and sediment samples are to be collected at the same location (if both are required in the project-specific Sampling and Analysis Plan).

2. Collect the surface water sample first. Sediment sampling usually results in disturbance of the sediments, which may influence the analytical results of the surface water samples.

3. Wear gloves when collecting samples. Comply with the Health and Safety Plan specifications for proper personal protective equipment.

4. If sampling from a boat or near waterbodies with depths of 4 ft or more, the sampling team will wear life jackets.

5. Wading into a waterbody disturbs the sediment. Move slowly and cautiously, approach the sample location from downstream. If flow is not strong enough to move entrained particles away from the sample location, wait for the sediment to resettle before sampling.

6. Collect samples first from areas suspected of being the least contaminated, thus minimizing the risk of cross-contamination.

7. Collecting samples directly into sample containers is not recommended. Sediment samples should be placed in Teflon®, stainless steel, or glass trays, pans, or bowls for sample preparation.

8. Use the proper equipment and material construction for the analytes of interest. For example, for volatile organic compound analysis, the sampling material in direct contact with the sediment or surface water must consist of Teflon, polyethylene, or stainless steel.

9. Refer to EA SOP No. 005 (Field Decontamination) for proper decontamination methods before and after sampling and between samples.

10. Collect samples for volatile organic compound analysis first. Do not mix such samples before placing them in the sample containers. For composite volatile organic compound samples, place equal aliquots of each subsample in the sample container.

11. Sediment that will be analyzed for other than volatile organic compounds should be prepared as follows:

    - Place the sediment in a mixing container.
    - Divide the sediment into quarters.
    - Mix each quarter separately and thoroughly.
    - Combine the quarters and mix thoroughly.
    - For composite samples, mix each subsample as described above. Place equal aliquots of each subsample in a mixing container and follow the procedure described above.

12. Mark the sampling location on a site map. Record sampling location coordinates with a Global Positioning System unit, photograph (optional, recommended) and describe each location, and place a numbered stake above the visible high water mark on the bank closest to the sampling location. The photographs and description must be adequate to allow the sampling station to be relocated at a future date.

13. Dispose of investigation-derived wastes according to applicable rules and regulations.

4. CORERS

A corer provides a vertical profile of the sediment, which may be useful in tracing historical contaminant trends. Because displacement is minimal, a corer is particularly useful when sampling for trace metals and organics. Corers can be constructed out of a variety of materials.

For example, a 2-in. diameter polyvinyl chloride pipe with a Teflon or polyethylene liner can be lowered into the sediment; a 2-in. diameter well cap can be used to form an airtight seal and negative pressure as the pipe is withdrawn.

- Ensure that the corer and (optional) liner are properly cleaned.
- Stand downstream of the sample location.
- Force the corer into the sediment with a smooth continuous motion. Rotate (not rock) the corer if necessary to penetrate the sediment.
- Twist the corer to detach the sample; then withdraw the corer in a single smooth motion. If the corer does not have a nosepiece, place a cap on the bottom to keep the sediment in place.
- Remove the top of the corer and decant the water (into appropriate sample containers for surface water analysis, if required).
- Remove the nosepiece or cap and deposit the sample into a stainless steel, Teflon, or glass tray.
- Transfer the sample into sample containers using a stainless steel spoon (or equivalent device).

5. SCOOPS AND SPOONS

When sampling at the margins of a waterbody or in shallow water, scoops and spoons may be the most appropriate sampling equipment. For collecting samples several feet from shore or in deeper water, the scoop or spoon may be attached to a pole or conduit.

- Stand downstream of the sample location.
- Collect the sample slowly and gradually to minimize disturbing the fine particles.
- Decant the water slowly to minimize loss of fine particles.
- Transfer the sediment to sample containers or mixing trays, as appropriate.

6. DREDGES

Three types of dredges are most frequently used: Peterson, Ponar, and Eckman. Many other dredge types are available; their applicability will depend upon site-specific factors.

6.1 PETERSON AND PONAR DREDGES

These dredges are suitable for hard, rocky substrates, deep waterbodies, and streams with fast currents. Ponars have top screens and side plates to prevent sample loss during retrieval.

- Open the jaws and place the cross bar into the proper notch.
- Lower the dredge to the bottom, making sure it settles flat.
- When tension is removed from the line, the cross bar will drop, enabling the dredge to close as the line is pulled upward during retrieval.
- Pull the dredge to the surface. Make sure the jaws are closed and that no sample was lost during retrieval.
- Open the jaws and transfer the sediment to sample containers or to a mixing tray.

6.2 ECKMAN DREDGE

The Eckman dredge works best in soft substrates in waterbodies with slow or no flow.

- Open the spring-loaded jaws and attach the chains to the pegs at the top of the sampler.
- Lower the dredge to the bottom, making sure it settles flat.
- Holding the line taut, send down the message to close the jaws.
- Pull the dredge to the surface. Make sure the jaws are closed and that no sample was lost during retrieval.
- Open the jaws and transfer the sediment to sample containers or a mixing tray.

7. REFERENCES

None.

Standard Operating Procedure No. 022
for
Sediment and Benthic Macroinvertebrate Sampling with Eckman Grab

Prepared by

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August 2007

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1. SCOPE AND APPLICATION

This Standard Operating Procedure covers the protocol for obtaining qualitative or quantitative samples of soft sediments and macroinvertebrates inhabiting soft sediments in lakes, reservoirs, and other waterbodies. The Eckman grab sampler is well suited to collecting samples in deeper (up to 100 ft) waterbodies.

Use of brand names in this Standard Operating Procedure is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the contractor will provide applicable and comparable standard operating procedures for the maintenance and calibration of same.

2. MATERIAL

The following materials may be required:

Eckman grab sampler: a box-shaped device with two scoop-like jaws	Sample containers
Boat	Sieve – 500 µ (U.S. Standard No. 30)
Personal protective equipment	Stainless steel spoon or trowel
Personal flotation devices	

3. PROCEDURES

The following is a summary of procedures on use of the Eckman grab sampler:

- Cock the sampler by raising each jaw upward into the cocked position using the attached able and secure the cable to the catch pin located at the top of the sampler.
- Once cocked, lift the sampler overboard and lower slowly but steadily to the bottom.
- Once on the bottom, indicated by a slack line, the weighted messenger is sent down the line tripping the catch mechanism, causing the spring loaded jaws to close the bottom of the sampler, containing the sediment.
- Raise the sample at a slow but steady rate to prevent sample loss or washout.
- Once the sample is on board, empty the sample into a stainless steel, polytetrafluoroethelyne, or polytetrafluoroethelyne-lined bowl or tray for processing.
  - If the sediment will be analyzed for volatile organic compounds, transfer the sample into the appropriate sample containers immediately.

- If the sediment will not be analyzed for volatile organic compounds, use Stainless steel spoon to thoroughly homogenize sample, then transfer sample into appropriate containers. Add preservative (if required) and place in ice-filled chest.

  — If benthic macroinvertebrates are to be collected, sieve sample and transfer macroinvertebrates into appropriate container.

- Thoroughly decontaminate the device.

4. MAINTENANCE

Maintain according to manufacturer's suggestions.

5. PRECAUTIONS

The following precautions should be taken while using an Eckman grab sampler:

- Inspect the device for mechanical deficiencies prior to its use.
- This sampler is inefficient in waters deeper than approximately 75-100 ft, under adverse weather conditions, and in waters of moderate to strong currents or wave action.
- Exercise caution at all times once the grab is loaded or cocked because a safety lock is not part of the standard design.
- Operate the sampler from a boat with a winch and cable.
- Wear gloves when collecting sediment samples. Be sure to consult the health and safety plan for the proper dermal and respiratory protection prior to collecting any samples.
- Higher levels of personal protective equipment may be required by the Health and Safety Plan.
- While sampling from a boat in waterbodies with a depth of 5 ft or more, the sampling team will wear personal flotation devices (life jackets).
- Collect samples first from those areas that are suspected of being the least contaminated, thus minimizing the risk of cross-contamination.

6. REFERENCES

American Society for Testing and Materials. Standard 2.1. D4387 Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates.

U.S. Environmental Protection Agency. 1990. Macroinvertebrate Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Waters. Office of Research and Development. EPA/600/4-90/030. November.

Standard Operating Procedure No. 039
for
Sample Preservation and Container Requirements

Prepared by

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August 2007

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6. PRECAUTIONS 3

7. REFERENCES 3

1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure (SOP) is to define the preservatives and techniques to be employed in preserving environmental samples between collection and analysis.

2. MATERIALS

The following materials may be required:

Containers (see Section 3 for description)	NaOH
HNO_3	Ice chests
H_2SO_4	Ice

3. DEFINITION OF CONTAINER TYPES

Type A **Container**: 80 oz amber glass, ring handle bottle/jug, 38-mm neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 38-430 size, 0.015-mm polytetrafluoroethelyne (PTFE) liner.

Type B **Container**: 40-mL glass vial, 24-mm neck finish
Closure: White polypropylene or black phenolic, open top, screw cap, 15-mm opening, 24-400 size.
Septum: 24-mm disc of 0.005-in PTFE bonded to 0.120-in. silicon for total thickness of 0.125-in.

Type C **Container**: 1-L high density polyethylene, cylinder-round bottle, 28-mm neck finish.
Closure: White polyethylene cap, white ribbed, 28-410 size; F217 polyethylene liner.

Type D **Container**: 120-mL wide mouth glass vial, 48-mm neck finish.
Closure: White polyethylene cap, 40-480 size; 0.015-mm PTFE liner.

Type E **Container**: 250-mL boston round glass bottle
Closure: White polypropylene or black phenolic, open top, screw cap.
Septum: Disc of 0.005-in PTFE bonded to 0.120-in silicon for total thickness of 0.125-in.

Type F **Container**: 8-oz short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.030-mm PTFE liner.

Type G **Container**: 4-oz tall, wide mouth, straight -sided, flint glass jar, 48-mm neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm PTFE liner.

Type H **Container**: 1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm PTFE liner.

Type K **Container**: 4-L amber glass ring handle bottle/jug, 38-mm neck finish.
Closure: White polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm PTFE liner.

Type L **Container**: 500-mL high-density polyethylene, cylinder bottle, 28-mm neck finish.
Closure: White polypropylene, white ribbed, 28-410 size; F217 polyethylene liner.

4. PROCEDURE

All containers must be certified clean, with copies of laboratory certification furnished upon request.

Water samples will be collected into pre-preserved containers appropriate to the intended analyte as given in Quality Assurance Project Plan. Samples taken for volatile organic compounds will be collected in accordance with SOP No. 003, Section 3.3.8. Samples taken for metals analysis will be verified in the field to a pH <2. The container should be tightly capped, then swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure this operation may have generated. Samples taken for total phosphorous content will be verified in the field to a pH <2. The container should be tightly capped and swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure this operation may have generated. Samples taken for cyanide will be verified for a pH >12. No preservatives will be added to any other water samples. These samples will be immediately placed on ice and cooled to 4°C.

Soil and sediment samples will be collected into containers appropriate to the intended analyte as given in the Quality Assurance Project Plan. Samples taken for volatile organic compound analysis will collected in accordance with the site-specific SOP. Samples taken for metals

analysis will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Samples taken for total phosphorous content will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Samples taken for cyanide will be alkalized to a pH > 12 by the addition of NaOH. No preservatives will be added to any other soil samples. These samples will be immediately placed on ice and cooled to 4°C.

5. MAINTENANCE

Not applicable.

6. PRECAUTIONS

Note that acidifying a sample containing cyanide may liberate HCN gas.

- Avoid breathing any fumes emanating from acidified samples.
- Acidify samples only in the open, rather than in closed spaces such as a vehicle.
- Hold suspected HCN-generating sample away from body and downwind while manipulating it.
- See the Health and Safety Plan for other safety measures

7. REFERENCES

U.S. Environmental Protection Agency (U.S. EPA). 1986. Test Methods for Evaluating Solid Waste, SW-845.

U.S. EPA. 1987. A Compendium of Superfund Field Operations Methods, EPA 540-P87-001.

U.S. EPA. 1991. A Compendium of ERT Soil Sampling and Surface Geophysics Procedures.

Standard Operating Procedure No. 042
for
Disposal of
Investigation-Derived Material

Prepared by

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to define the required steps for disposing of investigation-derived material (IDM) generated during field activities.

IDM, as used herein, includes soil cuttings, drilling muds, purged groundwater, decontamination fluids, and disposable personal protective equipment. For the sake of clarity and ease in use, this Standard Operating Procedure is subdivided into procedures for disposal of liquid IDM and solid IDM as follows:

- Liquid IDM (Section 3.2) includes the following materials:
  - Water from initial development of new wells and the redevelopment of existing wells.
  - Purge water from groundwater sampling.
  - Decontamination fluids (Section 3.4)
- Solid IDM (Section 3.3) consists of the following materials:
  - Drill cuttings from monitoring well installation
  - Grout, a mixture of cement and bentonite, generated during installation of monitoring wells
  - Disposable personal protective equipment (Section 3.4).

2. MATERIALS

The following materials may be required:

Any additional equipment that may be dictated by project or site-specific plans	Hazardous waste labels
Bar codes	Permanent marker
Chain-of-custody forms	Field Logbook (bound)
Department of Transportation 17C spec. metal containers	Waste identification labels

3. PROCEDURE

3.1 GENERAL

No container will be labeled as a “Hazardous Waste” unless the contents are in fact known to be hazardous as defined by 40 CFR 261.

IDM may be disposed onsite if it is: (1) initially screened, or evaluated to determine whether it is contaminated; (2) not abandoned in an environmentally unsound manner; and (3) not inherently waste-like.

IDM are to be considered contaminated if they: (1) are visually or grossly contaminated, (2) have activated any field monitoring device which indicates that the level exceeds standard Level 1, (3) have previously been found to exhibit levels of contamination above environmental quality standards, and (4) the responsible party and/or appropriate regulator deem(s) that records of historical uses indicate that additional testing of the IDM is needed, or additional caution is warranted handling IDM from a given site.

3.2 PROCEDURES FOR LIQUID INVESTIGATION-DERIVED MATERIAL DISPOSAL

1. All water from initial development of new wells, and purge water generated during the first round of groundwater sampling will be containerized in Department of Transportation approved 55-gal drums. Decontamination fluids may be bulk-containerized until completion of field task.

2. Label all containers as to type of media, the date the container was sealed, the point-of-generation, and the points-of-contact. The well number and container number will be identified on the container.

3. The contractor/support personnel will log all media generated onsite into a bound Field Logbook. Media information should include the following: the date of generation, contents of containers, the number of containers with the same contents (if applicable), location of containers, the well number the media is associated with, personnel sampling the media, sampling dates, and sampling results.

4. Containers of well development water and purge water may be stored at the well site pending first round analytical results.

5. Laboratory turnaround time must be no greater than 30 days. Upon receipt of the analytical results, a copy will be furnished to the client within 3 working days. Both the client and contractor will evaluate the data to determine disposal requirements, per state and local regulations. A disposal decision is required within 10 days of receipt of sampling results. Appropriate disposal must be performed no later than 50 days from the decision date unless prevented by inclement weather (e.g., rain and muddy conditions may preclude site access, freezing weather may freeze media).

---

1. This value is defined as two times background, where “background” values are to be determined as follows: (1) regional background values will be used where they are available; and (2) if regional values are not available, background may be empirically determined at uncontaminated sampling sites using onsite sensors such as organic vapor analyzers (photoionization detector or flame ionization detector), scintillometers, etc.

Dispose of non-hazardous media in accordance with Step 6 et seq. through 8 et seq. of this procedure.

Dispose of hazardous waste in accordance with Step 9 et seq. of this procedure.

6. If the first round analytical data of the liquid media is below the Maximum Contaminant Levels established by the Federal Safe Drinking Water Act, the water may be gradually infiltrated into the ground at least 50 ft downgradient of the well.

   If the well location has no downgradient area, the water will be infiltrated into the ground in an area deemed appropriate by the client and the contractor/support personnel.

   Disposal locations must allow percolation of the water and prohibit "ponding."

   Upon completion of water discharge to ground, enter type of media, amount of media, date of disposal, and discharge point(s) in a bound Field Logbook and provide this information to the client.

   Empty containers are to be properly decontaminated, stored, and reused by the appropriate personnel.

   If the liquid media sampling results do not meet the required Maximum Contaminant Levels and cannot be discharged to the ground, then determine if the waste meets the sanitary sewer discharge criteria (National Pollutant Discharge Elimination System standards).

7. If at any time visual contamination of purge/development water is observed, or if organic vapor monitor readings (HNu, photoionization detector) register more than 5 ppm above background and/or rad meters register more than twice the background mrems, then the liquid will be drummed and a composite sample will be taken that day. A disposal decision will be based on the analytical results of this sample rather than the first round of analytical results.

3.3 PROCEDURE FOR SOLID INVESTIGATION-DERIVED MATERIAL DISPOSAL

1. If the conditions outlined in Section 3.1 are met, proceed to Section 3.3, Step 2; otherwise, go to Section 3.3, Step 7.

2. During drilling operations, the resulting cuttings and mud will be discharged onto the ground near the well if the following conditions are met: (1) no visual contamination is observed, (2) organic vapors are less than 5 ppm above background, (3) rad meter readings (if applicable) are under two times background, and (4) if the potential for metals contamination exists, the medium has been screened and found to be less than two times background.

Proper sediment and erosion control measures will be implemented as follows:

- Drill cuttings will be uniformly spread and contoured to blend with the surroundings of the site.
- If amount of solid IDM exceeds 5,000 ft^2 or 100 yd^3 of material, a Sediment and Erosion Control Plan is required.
- If the amount of solid IDM is under 5,000 ft^2 or 100 yd^3, the site will be stabilized as soon as possible. Stabilization includes mulch, seed, and tack.
- Critical areas require stabilization within 7 days from the date of well completion. Critical areas include swales, water sources, drainage ditches, etc.
- All other disturbed areas require stabilization within 14 days from the date of well completion.

3. If the well location is in or near a wetland, the drill cuttings will be drummed and transported away from the site for spreading.

4. Label all IDM containers that will not be spread on the day of generation. Each container should be labeled with the type of media, the date the container was sealed, the point-of-generation, and the name of the contact person. The well number and container number should be identified on the container.

5. The contractor/support personnel will log all media generated onsite into a bound Field Logbook. Media information should include: the date of generation, contents in containers, the number of containers with the same contents, location of containers, and the well number the media is associated with.

6. Containers will be staged at the well site until contractor/support personnel spread the cuttings in the appropriate locations, using proper sediment and erosion control measures per Section 3.3 et seq.

7. If drilling mud and cuttings show visible contamination, or organic vapor readings are more than 5 ppm above background levels, or rad meter readings (if applicable) show greater than two times background levels, media will immediately be containerized, labeled appropriately (Section 3.2), and sampled on the same day.

8. The solid IDM should be sampled and appropriate Toxicity Characteristic Leaching Procedure analyses conducted prior to determining disposition. Laboratory turn-around time must be no greater than 30 days. Upon receipt of analytical results, a copy will be furnished to the client within 3 working days. The contractor will evaluate the data to determine disposal requirements within 10 days. Appropriate disposal must be performed no later than 50 days after the decision date if weather permits (Section 3.2).

- If the solid IDM are determined to be non-hazardous and uncontaminated, go to Section 3.3.

- If the solid IDM are determined to be non-hazardous but contaminated , go to Section 3.3.

- If the solid IDM are found to be hazardous wastes, go to Section 3.3.

9. If the solid IDM are not a hazardous waste **and** analytical data shows contaminant concentrations below the U.S. Environmental Protection Agency Region 3 (or applicable Region where work is being performed) Risk-Based Concentrations, contact the appropriate federal, state, or local agency for approval to discharge onto the ground near the site of generation.

    - Follow steps detailed in Section 3.3, Step 2 et seq. (above) pertaining to sediment and erosion control.

    - Upon completion of the solid IDM discharge to the ground, enter type of media, amount of media, date of disposal, and discharge point(s) in a bound Field Logbook. This information must be provided to the client.

    - Empty containers are to be properly decontaminated, stored and reused by appropriate personnel.

10. If the intrusive media is not a hazardous waste but analytical data shows concentrations above the screening criteria, dispose of the IDM according to state and local regulations.

    - Ensure that the waste containers are properly labeled as applicable in accordance with Section 3.3, Step 4.

    - Inform the client of the type and amount of waste, and the location of the waste.

    - When the waste is removed, enter the type of waste, amount of waste, date of pickup, and the destination of the waste in a bound Field Logbook. This information must be provided to the client.

3.4 PROCEDURES FOR DECONTAMINATION SOLUTION AND PERSONAL PROTECTIVE EQUIPMENT DISPOSAL

Decontamination solutions include catch water from steam-cleaning operations performed on large sampling equipment, drill rigs, and drums, as well as smaller quantities of soapy water and rinse solutions used in decontaminating field sampling equipment. At the completion of the field event, a composite sample of the decontamination solution will be taken. The decontamination solution will be treated as liquid IDM pending results (Section 3.2 et seq.).

Personal protective equipment will be containerized onsite, appropriately labeled, and disposed of in a designated trash receptacle.

4. MAINTENANCE

Not applicable.

5. REFERENCES

Environment Article Section 7-201(t).

U.S. Environmental Protection Agency. 1991. Management of Investigation-Derived Wastes during Site Inspections PB91-921331, OERR Directive 9345.3-02. Office of Emergency and Remedial Response U.S. Environmental Protection Agency, Washington, D.C. May.

Appendix C

Field Forms

(Included on Electronic Copy Only)

Daily Field Report
State Marine of Port Arthur Superfund Site

EPA Personnel: _____ Site Manager: _____ Date: _____

1.0 SITE INVESTIGATION ACTIVITIES/PROGRESS

2.0 WEATHER:

3.0 HEALTH AND SAFETY:

3.0 ACTIVITIES PLANNED FOR NEXT WORKING DAY

4.0 ISSUES AND CONCERNS

STATE MARINE OF PORT ARTHUR SUPERFUND SITE
SEDIMENT SAMPLING FORM

Project: State Marine of Port Arthur Superfund Site Sample ID.:_____

Date: _____ Time: _____

Sampled By:_____

Weather Conditions:_____

Tide Conditions:_____

Sample Location:_____

Sample Location Description:_____

Sample Depth: _____

Water Depth: _____

Surface Water Type: _____

Sample Condition (i.e. Turbidity, Odor, Etc.):_____

_____

Collection Method: _____

Collection Time:_____

Analysis	Containers	Sample Preparation/Preservation

Comments: _____

_____

_____

Appendix D

Electronic Information

(Included on Electronic Copy Only)

EXHIBIT E

CONTRACT LABORATORY PROGRAM QUALITY ASSURANCE MONITORING PLAN

Exhibit E – Contract Laboratory Program Quality Assurance Monitoring Plan

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1.0 OVERVIEW

Quality Assurance (QA) and Quality Control (QC) are integral parts of the U.S. Environmental Protection Agency's (USEPA's) Contract Laboratory Program (CLP). The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity, and ensures that data provided are of the quality required. The QC process includes those activities required during data collection to produce the data quality desired and to document the quality of the collected data.

1.1 Quality Assurance/Quality Control (QA/QC) Activities

During the planning of an environmental data collection program, QA activities focus on defining data quality criteria and designing a QC system to measure the quality of data being generated. During the implementation of the data collection effort, QA activities ensure that the QC system is functioning effectively, and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data obtained to determine its suitability to support enforcement or remedial decisions.

1.1.1 This Exhibit describes the overall QA/QC operations and the processes by which the CLP meets the QA/QC objectives defined above. This contract requires a variety of QA/QC activities. These contract requirements are the minimum QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These QC operations are designed to facilitate laboratory comparison by providing USEPA with comparable data from all Contractors. These requirements do not release the analytical Contractor from maintaining their own QC checks on method and instrument performance.

1.2 Performance

Samples are distributed routinely to Contractors based on the quality of work performed, as measured by the Performance Scheduling Algorithm (PSA) (see Section G of the contract for details). A Contractor that consistently meets the contract performance requirements as highlighted below will earn a higher PSA score, thereby increasing the likelihood of receiving samples for analyses.

Contract Performance Requirements:

- high quality, technically sound data as stipulated by the ISM01.2 contract;
- on-time or early delivery of the Sample Delivery Group (SDG) Cover Sheet;
- above average Quarterly Blind (QB) Performance Evaluation (PE) sample scores;
- electronic deliverables that pass the initial Contract Compliance Screening (CCS) acceptance criteria; and
- SDGs delivered on-time.

If the Contractor fails to meet the requirements set forth in this Statement of Work (SOW) or elsewhere in the contract, USEPA may take,

but is not limited to, the following actions (see Section E of the contract for details): reduction in the number of samples sent under the contract; suspension of sample shipments; data package audit(s); Remedial QB Audit; electronic data audit(s); on-site laboratory evaluation(s); and/or remedial PE sample(s).

2.0 INTRODUCTION

Appropriate use of data generated under the large range of analytical conditions encountered in environmental analyses requires reliance on the Quality Control (QC) procedures and criteria incorporated into the ISM01.2 Statement of Work (SOW).

The data acquired from QC procedures are used to estimate and evaluate the information content of analytical results and to determine the necessity for, or the effect of, corrective action procedures. The parameters used to estimate information content include precision, accuracy, detection limit, and other quantitative and qualitative indicators. In addition, QC procedures give an overview of the activities required in an integrated program to generate data of known and documented quality required to meet defined objectives.

2.1 Quality Assurance/Quality Control (QA/QC) Program Components

2.1.1 The Contractor’s QA/QC program shall include (1) internal QC criteria that demonstrate compliant levels of performance, as determined by QA review, as well as (2) external review of data and procedures accomplished by the monitoring activities of the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB), Regional Data Users, Sample Management Office (SMO), and the Quality Assurance Technical Support (QATS). Each external review accomplishes a different purpose. These reviews are described in specific sections of this Exhibit. Laboratory evaluation samples, electronic data audits, and data packages provide an external QA reference for the program. A Contractor on-site evaluation system is also part of the external QA monitoring. A feedback loop provides the results of the various review functions to the Contractors through direct communications with the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and the USEPA OSRTI ASB Inorganic Program Manager (ASB PM).

2.1.2 This Exhibit does not provide specific instructions for constructing QA Management Plans, QC systems, or a QA organization. It is, however, an explanation of the QA/QC requirements of CLP. It outlines minimum standards for QA/QC programs. It also includes specific items that are required in a Quality Assurance Plan (QAP) and by the QA/QC documentation detailed in this contract. Delivery of this documentation provides USEPA with a complete data package which will stand alone, and limits the need for contact with the Contractor or with an analyst, at a later date, if some aspect of the analysis is questioned.

2.1.3 In order to assure that the product delivered by the Contractor meets the requirements of the contract, and to improve interlaboratory data comparison, the Contractor shall:

- Prepare, and adhere to, a written approved QAP, as defined in Exhibit E, Section 5;
- Prepare, and adhere to, Standard Operating Procedures (SOPs) as described in Exhibit E, Section 6;

- Adhere to the analytical methods in Exhibit D and associated QC requirements specified within Exhibit E;
- Verify and document analytical standards and retain documentation of the purity of neat materials, as well as the purity and accuracy of solutions obtained from private chemical supply houses;
- Submit all requested raw data and required documentation for USEPA Regional review;
- Submit results of all analyzed laboratory evaluation samples and adhere to corrective action procedures;
- Submit, upon request, electronic instrument data and applicable documentation for tape audits, including a copy of the Complete Sample Delivery Group (SDG) File (CSF);
- Submit to on-site laboratory evaluations and adhere to corrective action procedures; and
- Submit all original documentation generated during sample analyses for USEPA review.

3.0 GENERAL QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) REQUIREMENTS

The Contractor shall adhere to USEPA's Good Laboratory Practices for laboratory cleanliness with regard to glassware and apparatus. The Contractor shall also adhere to good laboratory practices with regard to reagents, solvents, and gases. For additional guidelines regarding these general laboratory procedures, see the Handbook for Analytical Quality Control in Water and Wastewater Laboratories USEPA-600/4-79-019, USEPA Environmental Monitoring Systems Laboratory, Cincinnati, Ohio, September 1982.

4.0 SPECIFIC QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) MONITORING PROCEDURES

4.1 Purpose

4.1.1 The purpose of this document is to provide (1) a uniform set of procedures for the analysis of inorganic constituents of samples; (2) documentation of methods and their performance; and (3) verification of the sample data generated. Although it is impossible to address every analytical situation in one document, this Exhibit defines the minimum requirements for each major step relevant to any inorganic analysis.

4.1.2 The primary function of the Contract Laboratory Program (CLP) QA/QC program is the definition of procedures for the evaluation and documentation of analytical methodologies and the reduction and reporting of data. The location and summary of the QA/QC performance-based contracting methods can be found in Exhibit E, Section 15, Table 1 - Contract Laboratory Program Quality Assurance Monitoring Plan. The objective is to provide a uniform basis for sample handling, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. In many instances where methodologies are available, specific QC procedures are incorporated into the method documentation (see Exhibit D).

4.1.3 The QA/QC procedures defined herein shall be used by the Contractor when performing the methods specified in Exhibit D. When QA/QC procedures are specified in Exhibit D, the Contractor shall follow those procedures, in addition to procedures specified here.

4.2 Laboratory Audit and Intercomparison Study Program

The Contractor is required to participate in the Laboratory Audit and Intercomparison Study Program run by USEPA. The Contractor shall be required to analyze at least one Quarterly Blind (QB) sample per calendar quarter during the contract period for inorganics.

4.3 Annual Determination of Method Detection Limit (MDL)

The Contractor shall update and report the annual determination of MDLs by the method specified in Exhibit D, by type, matrix, and model for each instrument used on this contract, to the Sample Management Office (SMO), Quality Assurance Technical Support (QATS), and the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) as specified in Exhibit B. All MDLs shall meet the requirements specified in Exhibit C.

4.4 Annual Verification of Interelement Correction Factors

For Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) methods, the Contractor shall report, as specified in Exhibit B, all interelement correction factors.

4.5 Quality Assurance/Quality Control Measurements

4.5.1 In this Exhibit, as well as other places within this Statement of Work (SOW), the term "analytical sample" discusses the required frequency or placement of certain QA/QC measurements. The term "analytical sample" is defined in the glossary, Exhibit G.

4.5.2 In order for the QA/QC information to reflect the status of the samples analyzed, all samples and their associated QA/QC analysis shall be analyzed under the same operating and procedural conditions.

4.5.3 If any QC measurement fails to meet contract criteria, the analytical measurement must not be repeated prior to taking the appropriate corrective action as specified in Exhibit D.

4.5.4 The Contractor shall report all QC data in the exact format specified in Exhibits B and H.

4.5.5 MDLs, precision, and interference effects shall be established for each analyte on a particular instrument. All reported measurements shall be within the instrumental calibrated ranges. The Contractor shall maintain QC data confirming instrument performance and analytical results.

5.0 QUALITY ASSURANCE PLAN (QAP)

5.1 Introduction

The Contractor shall establish a Quality Assurance (QA) program with the objective of providing sound analytical chemical measurements. This program shall incorporate the Quality Control (QC) procedures, any necessary corrective action, all documentation required during data collection, and the quality assessment measures performed by management to ensure acceptable data production. The Contractor shall follow the USEPA EPA Requirements for Quality Management Plans (EPA QA/R-2). An electronic version can be found at http://www.epa.gov/quality/qs-docs/r2-final.pdf.

5.1.1 The Contractor shall prepare a written QAP which describes the procedures that are implemented to achieve the following:

- Maintain data integrity, validity, and usability;
- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility;
- Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable; and
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

5.1.2 The QAP must present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA/QC activities designed to achieve the data quality requirements in this contract. Standard Operating Procedures (SOPs) pertaining to each element shall be included or referenced as part of the QAP. The QAP shall be paginated consecutively in ascending order. The QAP shall be available during on-site laboratory evaluations and shall be submitted to the designee within 7 days of written request by the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM). Additional information relevant to the preparation of a QAP can be found in USEPA and ASTM publications.

5.2 Required Elements of a Quality Assurance Plan

The required elements of a laboratory's QAP are outlined in this Section. This outline shall be used as a framework for developing the QAP.

A. Organization and Personnel

1. QA Policy and Objectives (the mission and quality policy of the organization)
2. QA Management (the specific roles, authorities, and responsibilities of management and staff with respect to QA and QC activities)
   a. Organization
   b. Assignment of QA/QC Responsibilities
   c. Reporting Relationships (the means by which effective communications with personnel actually performing the work are assured)

d. QA Document Control Procedures

e. QA Program Assessment Procedures (the process used to plan, implement, and assess the work performed)

3. Personnel

a. Resumes

b. Education and Experience Pertinent to this Contract

c. Training Records and Progress

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives

2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy

2. Sample Tracking/Custody Procedures

3. Logbook Maintenance and Archiving Procedures

4. Sample Delivery Group (SDG) File Organization, Preparation, and Review Procedures

5. Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs

6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Calibration Procedures and Frequency

2. Sample Preparation Procedures

3. Sample Analysis Procedures

4. Standards Preparation Procedures

5. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

E. Data Generation

1. Data Collection Procedures

2. Data Reduction Procedures

3. Data Validation Procedures

4. Data Reporting and Authorization Procedures

F. Quality Assurance (the process which measures the effectiveness of QA will be established and how frequently effectiveness will be measured)

1. Data Quality Assurance

2. Systems/Internal Audits

3. Performance/External Audits

4. Corrective Action Procedures (the continual improvement based on lessons learned from previous experience)

5. QA Reporting Procedures

6. Responsibility Designation

G. Quality Control

1. Solvent, Reagent, and Adsorbent Check Analysis
2. Reference Material Analysis
3. Internal QC Checks
4. Corrective Action and Determination of QC Limit Procedures
5. Responsibility Designation

5.3 Updating and Submitting the Quality Assurance Plan

5.3.1 The revised QAP will become the official QAP under the contract and may be used during legal proceedings. The Contractor shall maintain the QAP on file at the Contractor's facility for the term of the contract. Both the initial submission and the revised QAP shall be paginated consecutively in ascending order. The revised QAP shall include:

- Changes resulting from (1) the Contractor's internal review of their organization, personnel, facility, equipment, policy and procedures; and (2) the Contractor's implementation of the requirements of the contract, and
- Changes resulting from USEPA's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the pre-award on-site laboratory evaluation.

5.3.1.1 The Contractor shall send a copy of the latest version of the QAP within 7 days of a request from a USEPA Regional CLP PO or the USEPA OSRTI ASB PM. The request will designate the recipients.

5.3.2 Subsequent Updates and Submissions. During the term of the contract, the Contractor shall amend the QAP when the following circumstances occur:

- USEPA modifies the technical requirements of the Statement of Work (SOW) or contract;
- USEPA notifies the Contractor of deficiencies in the QAP document;
- USEPA notifies the Contractor of deficiencies resulting from USEPA's review of the Contractor's performance;
- The Contractor's organization, personnel, facility, equipment, policy, or procedures change; or
- The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy, or procedures changes.

5.3.2.1 The Contractor shall amend the QAP within 14 days of when the circumstances listed in Exhibit E, Section 5.3, result in a discrepancy between what was previously described in the QAP and what is presently occurring at the Contractor's facility. When the QAP is amended, all changes in the QAP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, highlighting the change by underlining the change, bold printing the change, or using a different print font) and a copy is sent to the USEPA Regional CLP PO and Quality

Assurance Technical Support (QATS). The amended section pages shall have the date on which the changes were implemented. The Contractor shall incorporate all amendments to the latest version of the QAP document. The Contractor shall archive all amendments to the QAP document for future reference by USEPA.

5.4 Performance

The Contractor shall amend the QAP as specified within this Section. The QAP describes the policies and procedures for ensuring that work processes, products, or services satisfy expectations or specifications in ISM01.2. Failure to comply with the requirements of this Section may result in disincentives as described in the contract.

6.0 STANDARD OPERATING PROCEDURES (SOPS)

6.1 Introduction

In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by USEPA, an SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks. The Contractor shall follow the USEPA Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents (EPA QA/G-6). An electronic version can be found at http://www.epa.gov/quality1/qs-docs/g6-final.pdf.

6.1.1 SOPs prepared by the Contractor shall be functional (i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts). The SOPs shall be paginated consecutively in ascending order.

6.1.2 All SOPs shall reflect Contractor activities as they are currently performed in the laboratory. In addition, all SOPs shall be:

- Consistent with current USEPA regulations, guidelines, and the Contract Laboratory Program (CLP) ISM01.2 contract requirements.
- Consistent with instrument(s) manufacturer’s specific instruction manuals.
- Available to USEPA during an on-site laboratory evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During on-site laboratory evaluations, laboratory personnel shall demonstrate the application of the SOPs if requested.
- Available to the designated recipients within 7 days, upon request by the USEPA Regional CLP Project Officer (CLP PO) or the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch Inorganic Program Manager (ASB PM).
- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol.

- Capable of demonstrating the validity of data reported by the Contractor and explaining the cause of missing or inconsistent results.
- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements.
- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made.
- Archived for future reference in usability or evidentiary situations.
- Available at specific work stations as appropriate.
- Subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.
- Reviewed and signed by all Contractor personnel performing actions identified in the SOP.

6.2 Format

The format for SOPs may vary depending upon the type of activity for which they are prepared; however, at a minimum, the following sections shall be included:

- Title page;
- Document Control;
- Scope and Applicability;
- Summary of Method;
- Definitions (acronyms, abbreviations, and specialized forms used in the SOP);
- Health & Safety;
- Personnel Qualifications;
- Interferences;
- Apparatus & Materials (list or specify; note also designated locations where found);
- Handling & Preservation;
- Instrument or Method Calibration;
- Sample Preparation and Analysis;
- Data Calculations;
- Quality Control (QC) limits;
- Corrective action procedures, including procedures for secondary review of information being generated;
- Data Management and Records Management;
- Miscellaneous notes and precautions; and
- References.

6.3 Required SOPs

The Contractor shall maintain the following SOPs:

6.3.1 Evidentiary SOPs for required chain-of-custody and document control are discussed in Exhibit F.

6.3.2 Sample Receipt and Storage

- Sample receipt and identification logbooks,
- Refrigerator temperature logbooks, and
- Security precautions.

6.3.3 Sample Preparation

6.3.3.1 Metals

6.3.3.2 Cyanide

6.3.4 Glassware Cleaning

6.3.5 Calibration (Balances, etc.)

- Procedures;
- Frequency requirements;
- Preventative maintenance schedule and procedures;
- Acceptance criteria and corrective actions; and
- Logbook maintenance authorization.

6.3.6 Analytical Procedures (for each analytical system)

- Instrument performance specifications;
- Instrument operating procedures;
- Data acquisition system operation;
- Procedures when automatic quantitation algorithms are overridden;
- QC required parameters;
- Analytical run/injection logbooks;
- Instrument error and editing flag descriptions and resulting corrective actions; and
- Method Detection Limit (MDL) determinations and Interelement Correction (IEC) determinations.

6.3.7 Maintenance Activities (for each analytical system)

- Preventative maintenance schedule and procedures;
- Corrective maintenance determinants and procedures; and
- Maintenance authorization.

6.3.8 Analytical Standards

- Standard coding/identification and inventory system;
- Standards preparation logbook(s);
- Standard preparation procedures;

- Procedures for equivalency/traceability analyses and documentation;
- Standard Certificate of Analysis;
- Purity logbook (primary standards and solvents);
- Storage, replacement, and labeling requirements; and
- QC and corrective action measures.

6.3.9 Data Reduction Procedures

- Data processing systems operation;
- Outlier identification methods;
- Identification of data requiring corrective action; and
- Procedures for format and/or forms for each operation.

6.3.10 Documentation Policy/Procedures

- Contractor/analyst's notebook policy, including review policy;
- Complete Sample Delivery Group (SDG) File (CSF) contents;
- Complete SDG File organization and assembly procedures, including review policy; and
- Document inventory procedures, including review policy.

6.3.11 Data Validation/Self-Inspection Procedures

- Data flow and chain-of-command for data review;
- Procedures for measuring precision and accuracy;
- Evaluation parameters for identifying systematic errors;
- Procedures to assure that hardcopy and electronic deliverables are complete and compliant with the requirements in the Statement of Work (SOW) Exhibits B and H;
- Procedures to assure that hardcopy deliverables are in agreement with their comparable electronic deliverables;
- Demonstration of internal Quality Assurance (QA) inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal laboratory evaluation samples, etc.);
- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);
- Demonstration of problem identification, corrective actions, and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback); and
- Documentation of audit reports (internal and external), response, corrective action, etc.

6.3.12 Data Management and Handling

- Procedures for controlling and estimating data entry errors;
- Procedures for reviewing changes to data and deliverables and ensuring traceability of updates;

- Lifecycle management procedures for testing, modifying, and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems;
- Database security, backup, and archival procedures including recovery from system failures;
- System maintenance procedures and response time;
- Individual(s) responsible for system operation, maintenance, data integrity, and security; and
- Specifications for staff training procedures.

6.4 Updating and Submitting SOP Requirements

6.4.1 The revised SOPs will become the official SOPs under the contract and may be used during legal proceedings. The Contractor shall maintain the complete set of SOPs on file at the Contractor's facility for the term of the contract. Both the initial submission and the revised SOPs shall be paginated consecutively in ascending order. The revised SOPs shall include:

- Changes resulting from (1) the Contractor's internal review of their procedures; and (2) the Contractor's implementation of the requirements of the contract, and
- Changes resulting from USEPA's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the pre-award on-site laboratory evaluation.

6.4.1.1 The Contractor shall send a complete set of the latest version of SOPs or individually requested SOPs within 7 days of a request from an USEPA Regional CLP PO or the USEPA OSRTI ASB PM. The request will designate the recipients.

6.4.2 Subsequent Updates and Submissions. During the term of the contract, the Contractor shall amend the SOPs when the following circumstances occur:

- USEPA modifies the technical requirements of the SOW or contract;
- USEPA notifies the Contractor of deficiencies in the SOP documentation;
- USEPA notifies the Contractor of deficiencies resulting from USEPA's review of the Contractor's performance;
- The Contractor's procedures change;
- The Contractor identifies deficiencies resulting from the internal review of the SOP documentation; or
- The Contractor identifies deficiencies resulting from the internal review of their procedures.

6.4.2.1 Existing SOPs shall be amended or new SOPs shall be written within 14 days of when the circumstances listed in Exhibit E, Section 6.4, result in a discrepancy between what was previously described in the SOPs and what is presently occurring at the Contractor's facility. All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is

in the document, highlighting the change by underlining the change, bold printing the change, or using a different print font) and a copy is sent to the USEPA Regional CLP PO and Quality Assurance Technical Support (QATS). The amended/new SOPs shall have the date on which the changes were implemented.

6.4.2.2 When existing SOPs are amended or new SOPs are written, the Contractor shall document the reasons for the changes and maintain the amended SOPs or new SOPs on file. Documentation of the reasons for the changes shall be maintained on file with the amended SOPs or new SOPs.

6.4.2.3 Documentation of the reason(s) for changes to the SOPs shall also be submitted along with the SOPs.

6.5 Performance

The Contractor shall amend SOPs as specified within this Section. The SOPs specify analytical procedures in greater detail than appear in Exhibit D. Adherence to these requirements will ensure that the procedure is conducted in a standard, reliable, and reproducible process described in ISM01.2. Failure to comply with the requirements specified herein may result in disincentives as described in the contract.

7.0 CONTRACT COMPLIANCE SCREENING (CCS) PERFORMANCE STANDARDS

7.1 Overview

7.1.1 CCS is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the Complete Sample Delivery Group (SDG) File (CSF) delivered to USEPA.

7.1.2 CCS is performed by the Sample Management Office (SMO) under the direction of USEPA. To assure a uniform review, a set of standardized procedures has been developed to evaluate the CSF submitted by a Contractor against the technical and completeness requirements of the contract. USEPA reserves the right to add and/or delete individual checks.

7.2 CCS Results

CCS results are distributed to the Contractor and other data recipients. The Contractor has 6 business days to correct deficiencies and shall send all corrections to the USEPA Regional client and SMO. CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

7.3 CCS Trend Report

USEPA will periodically generate a CCS trend report which summarizes CCS results over a given period of time. USEPA will send the CCS trend report or discuss the CCS trend report during an on-site laboratory evaluation. In a detailed letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and USEPA Contracting Officer, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation.

7.4 Performance

7.4.1 The Contractor shall correct deficiencies and resubmit the data within 6 business days, as specified within this Section. Resubmission and correction of the data will ensure that the end user is reviewing contractually-compliant data described in ISM01.2. Correct resubmission of the data may also result in a reduction in overall disincentives. Specific details on disincentives can be found in the contract. If the Contractor fails to adhere to the requirements listed in this Section, the Contractor will be in noncompliance with the contract and may be subjected to disincentives as described in the contract.

7.4.2 If new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

8.0 ANALYTICAL PERFORMANCE STANDARDS REQUIREMENTS AND REAGENTS

8.1 Overview

USEPA will not supply analytical reference standards used to prepare calibration standards either for direct analytical measurements or for the purpose of traceability. All contract laboratories shall be required to prepare from materials or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol. At this time, USEPA will supply reference materials.

8.2 Preparation of Chemical Standards from the Neat High Purity Bulk Material

8.2.1 If the laboratory cannot obtain analytical reference standards, the laboratory may prepare their own chemical standards. Laboratories shall obtain the highest purity possible when purchasing chemical standards; standards purchased at less than 97% purity shall be documented as to why a higher purity could not be obtained.

8.2.2 The chemical standards shall be kept at manufacturer recommended conditions when not being used in the preparation of standard solutions. Proper storage of chemicals is essential in order to safeguard them from decomposition.

8.2.3 The Contractor shall be responsible for having analytical documentation proving the purity of each compound as stated. Purity confirmation, when performed, shall use appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

EQ. 1 Weight of Impure Compound

$$\text{weight of impure compound} = \frac{\text{weight of pure compound}}{(\text{percent purity}/100)}$$

WHERE, "weight of pure compound" is that required to prepare a specific volume of a solution standard of a specified concentration.

8.2.4 The Contractor is responsible for obtaining analytical documentation proving that all compounds used in the preparation of solution standards are correctly identified.

8.2.5 Logbooks shall be kept for all weighing and dilutions of standards and reagents. All subsequent dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person. All solution standards shall be refrigerated, if required, when not in use. All solution standards shall be clearly labeled as to the identity of the analyte or analytes, the standard ID number of the solution, concentration, date prepared, solvent, expiration date of the solution, special storage requirements (if any), and initials of the preparer.

8.3 Purchase of Chemical Standards Already in Solution

Solutions of analytical reference standards can be purchased by Contractors provided the solutions meet the following criteria.

8.3.1 Reference standards shall be accompanied by documentation of the purity confirmation of the material to verify the integrity of the standard solutions.

8.3.2 The quality of reference standards purchased shall be demonstrated statistically and analytically by a method of the supplier's choice.

8.3.3 Solutions of analytical reference materials can be ordered from the USEPA Chemical Standards Repository, depending on availability. The Contractor may place an order for standards only after demonstrating that these standards are not available from commercial vendors, either in solution or as a neat material.

8.4 Documentation of the Verification and Preparation of Chemical Standards

It is the responsibility of the Contractor to maintain the necessary documentation to show that the chemical standards it has used in the performance of Contract Laboratory Program (CLP) analysis conform to the requirements previously listed.

8.4.1 Weighing logbooks, calculations, raw data, etc., whether produced by the Contractor or purchased from chemical supply houses, shall be maintained by the Contractor and may be subject to review during on-site inspection visits. In those cases where the documentation is supportive of the analytical results of data packages sent to USEPA, such documentation is to be kept on file by the Contractor for a period of one year.

8.4.2 Upon request by the USEPA Regional CLP Project Officer (CLP PO), the Contractor shall submit their most recent previous year's documentation (12 months) for the verification and preparation of chemical standards within 14 days of the receipt of request to the designated recipients.

8.4.3 USEPA will periodically generate a report discussing deficiencies in the Contractor's documentation for the verification and preparation of chemical standards. USEPA will send the report or discuss the deficiencies during an on-site laboratory evaluation. In a detailed letter to the USEPA Regional CLP PO and CLP Quality Assurance Coordinator, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation.

8.4.4 If new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

8.5 Performance

The Contractor shall obtain the highest purity possible when purchasing chemical standards specified within this Section. The use of high purity standards will ensure a more accurate identification and quantitation of analytes described in the ISM01.2 Statement of Work (SOW). Failure to meet the requirements set forth in this Section may result in disincentives as described in the contract.

9.0 DATA PACKAGE MONITORING AUDITS

9.1 Overview

Data package audits are performed by USEPA for program overview and specific Regional concerns. Standardized procedures have been established to assure uniformity of the auditing process. Data packages are periodically selected from recently received Cases. They are evaluated for the technical quality of hardcopy raw data, Quality Assurance (QA), and adherence to contractual requirements. This function provides external monitoring of program Quality Control (QC) requirements. Data package audits are used to assess the technical quality of the data and evaluate overall laboratory performance. Audits provide USEPA with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. A thorough review of the raw data is completed, including all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements; a check for transcription and calculation errors; a review of the qualifications of the laboratory personnel involved with the Case; and a review of the latest version of all Standard Operating Procedures (SOPs) on file.

9.2 Responding to the Data Package Audit Report

9.2.1 After completion of the data package audit, USEPA will send a copy of the data package audit report to the Contractor or discuss the data package audit report on an on-site laboratory evaluation. In a detailed letter to the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) and the USEPA designated recipient, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the report.

9.2.2 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

9.3 Performance

The Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within

14 days of receipt of the comments from USEPA, as specified within this Section. The data package audits ensure that the policies and procedures identified in this Statement of Work (SOW) meet the requirements of this contract. Failure to meet the requirements set forth in this Section may result in disincentives as described in the contract.

10.0 REGIONAL DATA REVIEW MONITORING

10.1 Overview

Contractor data are generated to meet the specific needs of USEPA Regions. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of the end user, based on functional guidelines for data review which have been developed jointly by the Regions and the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB). Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problem under investigation and the Regional response appropriate to the specific circumstances.

10.1.1 Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process. They complement the review done at the Sample Management Office (SMO), which is designed to identify contractual discrepancies, and the review done by the USEPA OSRTI ASB, which is designed to evaluate Contractor and method performance.

11.0 QUALITY ASSURANCE (QA) PROFICIENCY MONITORING

As a means of measuring and evaluating both the Contractor's and the method's analytical performance, the Contractor shall participate in USEPA's Proficiency Testing Program. USEPA's Proficiency Testing Program involves the analysis of Case-specific Performance Evaluation (PE) samples and Quarterly Blind (QB) Audits. The Contractor's analytical PE samples and QB results will be used by USEPA to assess and verify the Contractor's continuing ability to produce acceptable analytical data in accordance with the contractual requirements. The Contractor shall receive a passing score of 75% to be in compliance with the contract.

11.1 Performance Evaluation (PE) Samples

11.1.1 PE sample(s) may be scheduled with the Contractor as frequently as on a Sample Delivery Group (SDG)-by-SDG basis. PE samples may be sent either by the USEPA Regional client or the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB). PE samples assist USEPA in monitoring Contractor performance.

11.1.2 PE samples will be provided as either single-blinds (recognizable as a PE sample but of unknown composition), or as double-blinds (not recognizable as a PE sample and of unknown composition). The Contractor will not be informed of either the analytes/parameters or the concentrations in the PE samples.

11.1.3 The Contractor may receive the PE samples as either full volume samples or ampulated/bottled concentrates from USEPA or a designated USEPA Contractor. The PE samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the PE samples (i.e., the required dilution of the PE sample concentrate). PE samples are to be digested and analyzed with the rest of the routine samples in the SDG. The Contractor shall prepare and analyze the PE sample using the procedure described in the sample preparation and method analysis sections of Exhibit D. All contract required Quality Control (QC) shall be met. The PE sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

11.1.4 In addition to PE sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes included in each PE sample. When PE sample results are received by USEPA, the PE sample results will be evaluated for correct analytical identification and quantitation. The PE sample evaluation will be provided to the Contractor via coded evaluation sheets, by analyte. USEPA will notify the Contractor of unacceptable performance.

11.2 Quarterly Blind (QB) Audits

11.2.1 A QB Audit is a unique analytical Case containing only PE samples (i.e., referred to as QB samples). The QB samples will be scheduled by the USEPA OSRTI ASB through the Sample Management Office (SMO). QB samples assist USEPA in monitoring Contractor performance.

11.2.2 QB samples will be provided as single-blinds (recognizable as a PE sample but of unknown composition). The Contractor will not be informed of either the analytes or the concentrations in the PE samples.

11.2.3 The Contractor may receive the QB samples as either full volume samples or ampulated/bottled concentrates from USEPA or a designated USEPA Contractor. The QB samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the QB samples (i.e., the required dilution of the QB sample concentrate). The Contractor shall prepare and analyze the QB samples using the procedure described in the sample preparation and method analysis sections of Exhibit D. All contract required QC shall be met, including spike and duplicate analyses. The QB sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B.

11.2.4 In addition to QB sample preparation and analysis, the Contractor shall be responsible for correctly identifying and quantitating the analytes included in each QB sample. When QB sample results are received by USEPA, the QB sample results will be scored for correct analytical identification, quantitation, and timeliness. The QB sample scoring will be provided to the Contractor via coded evaluation sheets, by analyte. USEPA will notify the Contractor of unacceptable performance. The Contractor's QB sample performance will be assessed into one of the following three categories:

11.2.4.1 Acceptable, No Response Required: Score greater than or equal to 90%. The data meets most or all of the scoring criteria. No response is required.

11.2.4.2 Acceptable, Response Explaining Deficiencies Required: Score greater than or equal to 75%, but less than 90%. Deficiencies exist in the Contractor's performance. Corrective action response required.

11.2.4.3 Unacceptable Performance, Response Explaining Deficiencies Required: Score less than 75%. Corrective action response required.

11.2.5 In the case of Section 11.2.4.2 or 11.2.4.3, the Contractor shall describe the deficiency(ies) and the action(s) taken in a corrective action letter to the USEPA Contracting Officer, USEPA Regional Contract Laboratory Program Project Officer (CLP PO), the Analytical Services Branch Inorganic Program Manager (ASB PM), and the CLP Quality Assurance (QA) Coordinator within 14 days of receipt of notification from USEPA.

11.2.6 In the case of Section 11.2.4.2 or 11.2.4.3, if new Standard Operating Procedures (SOPs) are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

11.2.7 A Remedial QB Audit is a unique analytical Case containing only QB samples. A Remedial QB Audit may be scheduled by the USEPA OSRTI ASB with the Contractor(s) for any of the following reasons: unacceptable PE sample performance, unacceptable QB sample performance, and/or major change in the laboratory (e.g., relocation, new owner, or high turnover of key personnel). The Contractor shall be on Contracting Officer (CO) Hold until the Remedial QB Audit package is submitted. Sections 11.2.2 through 11.2.7 apply to the Remedial QB Audit process.

11.2.8 The Contractor shall be notified by the USEPA Contracting Officer concerning agreement or disagreement with the proposed remedy for unacceptable performance.

11.3 Performance

The Contractor shall analyze PE and QB samples with acceptable analytical results in accordance with the contractual requirements as described in this Section. Failure to meet the requirements set forth in this Section may result in disincentives as described in the contract.

12.0 ON-SITE LABORATORY QUALITY ASSURANCE (QA) MONITORING EVALUATIONS

12.1 Overview

The USEPA Regional Contract Laboratory Program Project Officer (CLP PO), the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch Inorganic Program Manager (ASB PM), or the USEPA Contracting Officer's authorized representative will conduct an on-site laboratory evaluation. On-site laboratory evaluations are carried out to monitor the Contractor's ability to meet selected terms and conditions specified in the contract. The evaluation process incorporates two separate categories: Quality Assurance (QA) Evaluation and Evidentiary Audit.

12.2 Quality Assurance On-Site Evaluation

QA evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation; the continuity, experience and education of personnel; and the acceptable performance of analytical and Quality Control (QC) procedures for adherence to the contract requirements.

12.2.1 The Contractor shall expect that items to be monitored will include, but are not limited to, the following:

- Size, cleanliness, and organization of the facility;
- Quantity, age, availability, scheduled maintenance, and performance of instrumentation;
- Availability, appropriateness, and utilization of the Quality Assurance Plan (QAP) and Standard Operating Procedures (SOPs);
- Staff qualifications, experience, and personnel training programs;
- Analysis of Performance Evaluation (PE) sample(s);
- Reagents, standards, and sample storage facilities;
- Standard preparation logbooks, pipette calibration logbooks, balance check logbooks, and raw data;
- Bench sheets and analytical logbook maintenance and review; and
- Review of the Contractor's sample analysis/data package inspection (hardcopy and electronic)/data management procedures.

12.2.2 Prior to an on-site evaluation, various documentation pertaining to performance of the specific Contractor is integrated into a profile package for discussion during the evaluation. Items that may be included are previous on-site reports; Quarterly Blind (QB) and/or

PE sample scores results; USEPA Regional review of data; Contractor performance information provided by the Region; data audit reports; results of Contract Compliance Screening (CCS); and data trend reports.

12.3 Evidentiary Audit

Evidence auditors conduct an on-site laboratory evaluation to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements as stated in Exhibit F. The evidence audit comprises a procedural audit, an audit of written SOPs, and an audit of analytical project file documentation.

12.3.1 Procedural Audit. The Contractor shall perform analysis of PE sample(s) in the presence of the USEPA-designated team during the procedural audit. The procedural audit will be comprised of everything from sample receipt to data package assembly and completion. This includes the review and examination of actual SOPs and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), analytical project file organization and assembly, and proper disposal of samples and cogenerated wastes.

12.3.2 Written SOPs Audit. The written SOPs audit consists of the review and examination of the written SOPs to determine if they are accurate and complete for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

12.3.3 Analytical Project File Evidence Audit. The analytical project file evidence audit consists of the review and examination of the analytical project file documentation. The auditors review the files to determine:

- The accuracy of the document inventory;
- The completeness of the file;
- The adequacy and accuracy of the document numbering system;
- Traceability of sample activity;
- Identification of activity recorded on the documents; and
- Error correction methods.

12.4 Discussion of the On-Site Team's Findings

The QA and evidentiary auditors discuss their findings with the USEPA Regional CLP PO prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel. A report which discusses deficiencies found during the on-site audit will be sent to the Contractor to provide further clarification of findings. In a detailed letter to the USEPA Regional CLP PO and CLP Quality Assurance Coordinator, the Contractor shall discuss the deficiencies and the subsequent corrective actions implemented by the Contractor to resolve the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation.

12.4.1 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section 6.

12.5 Performance

The Contractor shall submit to on-site evaluations, as specified within this Section. The on-site evaluations ensure that the policies and procedures identified in this Statement of Work (SOW) meet the requirements of this contract. Failure to meet the requirements set forth in this Section may result in disincentives as described in the contract.

13.0 ELECTRONIC DATA QUALITY ASSURANCE (QA) MONITORING AUDITS

13.1 Overview

Periodically, USEPA requests the instrument electronic data from Contractors for a specific Case in order to accomplish electronic data audits. Generally, electronic data submissions and audits are requested for the following reasons:

- Program overview;
- Indication of data quality problems;
- Support for on-site audits; and
- Specific USEPA Regional requests.

13.1.1 Depending upon the reason for an audit, the instrument electronic data from a recent Case, a specific Case, or a laboratory evaluation sample may be requested. Electronic data audits provide a mechanism to assess adherence to contractual requirements and to ensure the consistency of data reported on the hardcopy/electronic deliverables with that generated on analytical instruments. This function provides external monitoring of Program Quality Control (QC) requirements and checks adherence of the Contractor to internal Quality Assurance (QA) procedures. In addition, electronic data audits enable USEPA to evaluate the utility, precision, and accuracy of the analytical methods.

13.1.2 The Contractor shall store <u>all</u> raw and processed electronic analytical data in the appropriate instrument manufacturer's format, uncompressed, and with no security codes. The data shall include all necessary data files for a complete reconstruction of the previously submitted hardcopy and electronic deliverable data package. All associated raw data files in the instrument manufacturer proprietary software format must be submitted if those files contain data or instrumental parameters regarding any analysis and or correction applied to an instrument or analytical result. This instrument electronic data shall include data for all samples and all QC samples, including but not limited to: blanks, matrix spikes, post-digestion spikes, analytical spikes, duplicates, serial dilutions, Laboratory Control Samples (LCSs), Interference Check Samples (ICSs), tunes, initial calibrations and verifications, and Continuing Calibration Verifications (CCVs). In addition, the Contractor shall supply raw data for the Method Detection Limit (MDL) studies which are used to set the MDL values for the year in which the Sample Delivery Group (SDG) was analyzed. The Contractor

shall maintain a reference logbook of data files of EPA sample number, calibration data, standards, blanks, spikes, and duplicates. The logbook shall include EPA sample numbers, identified by Case and SDG.

13.1.3 The Contractor is required to retain the instrument electronic data for 3 years after submission of the reconciled Complete SDG File (CSF). Electronic media shipped to the USEPA designated recipient must be fully usable by the recipient. Compact Discs (CDs) or DVDs may be used. Alternative means for delivery of electronic data may be utilized by the Contractor upon prior written approval by USEPA. When submitting electronic instrument data to USEPA, the following materials shall be delivered in response to the request:

13.1.3.1 All associated raw data files for all analytical samples and all QC samples. For example, files for ICP should include raw or background corrected intensities, and mercury and cyanide files should include raw absorbances or integrated areas.

13.1.3.2 All processed data files and quantitation output files associated with the raw data files described in Section 13.1.3.1.

13.1.3.3 All associated identification and calculation files used to generate the data submitted in the data package. This includes, but is not limited to, result files, acquisition files, calibration files, and method files.

13.1.3.4 All Contractor-generated Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES)/ICP - Mass Spectrometer (ICP-MS) interference correction files must be submitted.

13.1.3.5 A copy of the Contractor’s reference logbook relating data files to EPA sample number, calibration data, standards, blanks, spikes, and duplicates. The logbook shall include EPA sample numbers and laboratory file identifiers for all samples, blanks, and standards, identified by Case and SDG.

13.1.3.6 A printout of the directory of all files in each directory, including all subdirectories and the files contained therein.

13.1.3.7 A copy (hardcopy) of the completed CSF.

13.1.3.8 A statement attesting to the completeness of the electronic instrument data submission signed and dated by the Contractor’s laboratory manager. The Contractor shall also provide a statement attesting that the data reported have not been altered in any way. These statements shall be part of a Cover Sheet that includes the following information relevant to the data submission:

- Contractor name;
- Date of submission;
- Case number;
- SDG number;
- Instrument make and model number for each instrument;
- Instrument operating software name and version number;
- Data software name and version used for acquisition, re-quantitation, and hardcopy/report generation;
- Data system computer;

- System operating software;
- Data system network;
- Data backup software;
- Data backup hardware;
- Media type and volume of data (in MB) backed up; and
- Names and telephone numbers of two Contractor contacts for further information regarding the submission.

13.2 Submission of the Instrument Electronic Data

Upon request of the USEPA Regional Contract Laboratory Program Project Officer (CLP PO), the Contractor shall send the required instrument electronic data and all necessary documentation to the USEPA designated recipient [e.g., Quality Assurance Technical Support (QATS)] within 7 days of notification.

NOTE: The instrument electronic data shall be shipped according to the procedures in Exhibit F.

13.3 Responding to the Electronic Data Audit Report

After completion of the electronic data audit, USEPA will send a copy of the electronic data audit report to the Contractor or may discuss the electronic data audit report at an on-site laboratory evaluation. In a detailed letter to the USEPA Regional CLP PO, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the electronic data audit report within 14 days of receipt of the report or the on-site laboratory evaluation.

13.3.1 If new Standard Operating Procedures (SOPs) are required to be written or SOPs are required to be amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend and submit the SOPs per the requirements listed in Exhibit E, Section 6.

13.4 Performance

The Contractor shall submit to electronic data audits and adhere to the requirements specified in this Section. Resubmission and correction of electronic data will ensure that the end user is reviewing contractually compliant data described in the ISM01.2 contract. Failure to meet the requirements set forth in this Section may result in disincentives as described in the contract.

14.0 DATA MANAGEMENT PERFORMANCE REQUIREMENTS

14.1 Overview

14.1.1 Data management procedures are defined as procedures specifying the acquisition or entry, update, correction, deletion, storage, and security of computer readable data and files. These procedures shall be in written form and contain a clear definition for all databases and files used to generate or resubmit deliverables. Key areas of concern include system organization (including personnel and security), documentation operations, traceability, and Quality Control (QC).

14.1.2 Data manually entered from hardcopy shall be subject to QC checks and the error rates estimated. Systems should prevent entry of incorrect or out-of-range data and alert data entry personnel of errors. In addition, data entry error rates shall be estimated and recorded on a monthly basis by re-entering a statistical sample of the data entered and calculating discrepancy rates by data element.

14.2 Documenting Data Changes

The record of changes in the form of corrections and updates to data originally generated, submitted, and/or resubmitted shall be documented to allow traceability of updates. Documentation shall include the following for each change:

- Justification or rationale for the change.
- Initials of the person making the change(s). Data changes shall be implemented and reviewed by a person or group independent of the source generating the deliverable.
- Documentation of changes shall be retained according to the schedule of the original deliverable.
- Resubmitted electronic data or other deliverables shall be re-inspected as a part of the laboratory’s internal inspection process prior to resubmission. The entire deliverable, not just the changes, shall be inspected.
- The Laboratory Manager shall approve changes to originally submitted deliverables.
- Documentation of data changes may be requested by laboratory auditors.

14.3 Lifecycle Management Procedures

Lifecycle management procedures shall be applied to computer software systems developed by the Contractor to be used to generate and edit contract deliverables. Such systems shall be thoroughly tested and documented prior to utilization.

14.3.1 A software test and acceptance plan including test requirements, test results, and acceptance criteria shall be developed, followed, and available in written form.

14.3.2 System changes shall not be made directly to production systems generating deliverables. Changes shall be made first to a development system and tested prior to implementation.

14.3.3 Each version of the production system will be given an identification number, date of installation, and date of last operation and will be archived.

14.3.4 System and operations documentation shall be developed and maintained for each system. Documentation shall include a user's manual and an operations and maintenance manual.

14.3.5 This documentation shall be available for on-site review and/or upon written request by the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM).

14.4 Personnel Responsibilities

Individual(s) responsible for the following functions shall be identified.

- System operation and maintenance including documentation and training.
- Database integrity, including data entry, data updating, and QC.
- Data and system security, backup, and archiving.

15.0 TABLES

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit A: Summary of Requirements	Summary of Program Requirements	Performance standards are summarized in Exhibit A, Sections 1.0 through 4.0.	QA monitoring plan is outlined in Exhibit E.
Exhibit B: Reporting and Deliverables Requirements	Reporting and Deliverable Requirements	Performance standards are outlined in Exhibit B, Sections 1.0 through 4.0.	CCS in Exhibit E, Section 7.0, and SMO data review will be used to monitor reporting electronic deliverables.
Exhibit C: Inorganic Target Analyte List with Contract Required Quantitation Limits	Target Analyte List with Contract Required Quantitation Limits	Performance standards are outlined in Exhibit C, Section 1.0.	QA monitoring plan is outlined in Exhibit E.
Exhibit D: Analytical Methods	ICP-AES requirements are outlined in Exhibit D, Part A, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Part A, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Exhibit D, Part A, Section 12.0, and Exhibit E.
	ICP-MS requirements are outlined in Exhibit D, Part B, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Part B, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Exhibit D, Part B, Section 12.0, and Exhibit E.
	Mercury requirements are outlined in Exhibit D, Part C, Sections 1.0 through 8.0, 14.0 and 15.0.	Performance standards are outlined in Exhibit D, Part C, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Exhibit D, Part C, Section 12.0, and Exhibit E.
	Cyanide requirements are outlined in Exhibit D, Part D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Part D, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Exhibit D, Part D, Section 12.0, and Exhibit E.

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit E: Contract Laboratory Program Quality Assurance Monitoring Plan	General QA/QC Requirements	As outlined in Exhibit D, Quality Control Sections.	QA Management Plan is outlined in Exhibit E, Section 5.0.
	Quality Assurance Plan (QAP)	As outlined in Exhibit E, Sections 5.1.1 and 5.1.2, a written QAP shall be used to ensure acceptable data production of known and documented quality.	USEPA will review and approve the QA Management Plan.
	Standard Operating Procedures	Performance standards are outlined in Exhibit E, Sections 6.0 through 6.4, and must be performed as stated.	SOPs will be reviewed by USEPA during Pre-Award, on-site audits, after modifications are made, and randomly, as deemed appropriate.
	Contract Compliance Screening	Performance standards are outlined in the contract and must be performed as stated.	The Complete Sample Delivery Group (SDG) File (CSF) will be evaluated against the technical and completeness requirements of the contract.
	Analytical Standards	Performance standards are outlined in Exhibit E, Sections 8.0 through 8.5, and must be performed as stated.	Randomly, USEPA will review analytical standards verification and preparation documentation, as deemed appropriate.
	Data Package Audits	Performance standards are outlined in Exhibit E, Sections 9.0 through 9.2.	Data package audits are performed by USEPA to evaluate technical quality of the hardcopy raw data, QA, and adherence to contractual requirements.
	Regional Data Review	Analytical data is reviewed by each Region from the perspective of the end user to determine the usability of the data, as outlined in Exhibit E, Section 10.0.	USEPA Regional validation and/or SMO data review reports are generated for all data packages.

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit E: Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)	Proficiency Testing	Performance standards are outlined in Exhibit E, Sections 11.0 through 11.2, and must be performed as stated.	Acceptable QB scores will assist in monitoring contractor performance as defined in Exhibit E, Sections 11.2.4.1 through 11.2.4.3, and 11.2.8.
	On-Site Laboratory Evaluations	Performance standards are outlined in Exhibit E, Sections 12.0 through 12.4.	USEPA will evaluate the results from quality assurance and evidentiary on-site audits as defined in Exhibit E, Sections 12.2.1 through 12.3.3, to assist in monitoring the contractor.
	Electronic Data Audits	Performance standards are outlined in Exhibit E, Sections 13.0 through 13.3.	CCS in Exhibit E, Section 7.0, will be used to monitor electronic deliverables.
	Data Management	Performance standards are outlined in Exhibit E, Sections 14.0 through 14.4, and must be performed as stated.	USEPA will monitor data management practices during quality assurance and evidentiary on-site audits.
Exhibit F: Chain-of-Custody, Document Control, and Written Standard Operating Procedures	Standard Operating Procedures	Performance standards are outlined in Exhibit F, Sections 2.0 through 2.7.	SOPs will be reviewed by USEPA during Pre-Award, on-site audits, after modifications are made, and randomly as deemed appropriate.
	Written Standard Operating Procedures	Performance standards are outlined in Exhibit F, Sections 3.0 through 3.7.	SOPs will be reviewed by USEPA during Pre-Award, on-site audits, after modifications are made, and randomly as deemed appropriate.
Exhibit G: Glossary of Terms	Glossary of Terms	Contractors shall adhere to interpretation of SOW terms as defined within Exhibit G.	N/A
Exhibit H: Format for Electronic Data Deliverables	Data Dictionary and Format	Performance standards are outlined in Exhibit H.	CCS in Exhibit E, Section 7.0, will be used to monitor electronic deliverables.

EXHIBIT F

CHAIN-OF-CUSTODY, DOCUMENT CONTROL, AND WRITTEN STANDARD OPERATING PROCEDURES

Exhibit F – Chain-of-Custody, Document Control, and
Written Standard Operating Procedures

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1.0 INTRODUCTION

A sample is physical evidence collected from a facility or from the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. To ensure that U.S. Environmental Protection Agency's (USEPA's) sample data and records supporting sample-related activities are admissible and have weight as evidence in future litigation, Contractors are required to maintain USEPA samples under chain-of-custody and to account for all samples and supporting records of sample handling, preparation, and analysis. Contractors shall maintain sample identity, sample custody, and all sample-related records according to the requirements in this Exhibit.

1.1 Purpose of Evidence Requirements

The purpose of the evidence requirements includes:

- Ensuring traceability of samples while in possession of the Contractor;
- Ensuring custody of samples while in possession of the Contractor;
- Ensuring the integrity of sample identity while in possession of the Contractor;
- Ensuring sample-related activities are recorded on documents or in other formats for USEPA sample receipt, storage, preparation, analysis, and disposal;
- Ensuring that all laboratory records for each specified Sample Delivery Group will be accounted for when the project is completed; and
- Ensuring that all laboratory records directly related to USEPA samples are assembled and delivered to USEPA or, prior to delivery, are available upon USEPA's request.

2.0 STANDARD OPERATING PROCEDURES

The Contractor shall implement the following Standard Operating Procedures (SOPs) for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and Complete Sample Delivery Group (SDG) File (CSF) organization and assembly to ensure accountability of USEPA sample chain-of-custody as well as control of all USEPA sample-related records.

2.1 Sample Receiving

2.1.1 The Contractor shall designate a sample custodian responsible for receiving USEPA samples.

2.1.2 The Contractor shall designate a representative to receive USEPA samples in the event that the sample custodian is not available.

2.1.3 Upon receipt, the condition of shipping containers and sample containers shall be inspected and recorded on Form DC-1 by the sample custodian or a designated representative.

2.1.4 Upon receipt, the condition of the custody seals (intact/broken) shall be inspected and recorded on Form DC-1 by the sample custodian or a designated representative.

2.1.5 The sample custodian or a designated representative shall verify and record on Form DC-1 the agreement or disagreement of information recorded on all documents received with samples and information recorded on sample containers.

2.1.6 The sample custodian or a designated representative shall verify and record the following information on Form DC-1 as samples are received and inspected:

- Presence or absence and condition of custody seals on shipping and/or sample containers;
- Custody seal numbers when present;
- Presence or absence of Traffic Reports/Chain of Custody Records or Packing Lists;
- Presence or absence of airbills or airbill stickers;
- Airbill or airbill sticker numbers;
- Presence or absence of sample tags;
- Sample tags listed/not listed on Traffic Reports/Chain of Custody Records;
- Condition of the sample bottles;
- Presence or absence of cooler temperature indicator bottle;
- Cooler temperature;
- Date of receipt;
- Time of receipt;
- EPA sample numbers;
- pH of all aqueous/water samples;
- Sample tag numbers;
- Assigned laboratory numbers;
- Remarks regarding condition of sample shipment, etc.;
- Samples delivered by hand; and
- Problems and discrepancies.

2.1.7 The sample custodian or a designated representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (e.g., Traffic Reports/Chain of Custody Records or packing lists, and airbills).

NOTE: Initials are not acceptable.

2.1.8 The Contractor shall contact the Sample Management Office (SMO) to: resolve problems and discrepancies including, but not limited to: absent documents; conflicting information; absent or broken custody seals; insufficient sample volume; unsatisfactory sample condition (e.g., leaking sample container); and samples not preserved to the proper pH.

2.1.9 The Contractor shall record the resolution of all problems and discrepancies communicated through SMO.

2.2 Sample Identification

2.2.1 The Contractor shall maintain the identity of USEPA samples and prepared samples (including extracted samples, digested samples, and distilled samples) throughout the laboratory.

2.2.2 Each sample and sample preparation container shall be labeled with the EPA sample number or a unique laboratory sample identification number.

2.3 Sample Security

2.3.1 The Contractor shall demonstrate that USEPA sample custody is maintained from receiving through retention or disposal. A sample is in custody if:

- It is in your possession; or
- It is in your view after being in your possession; or
- It is locked in a secure area after being in your possession; or
- It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel.)

2.3.2 The Contractor shall demonstrate security of designated secure areas.

2.3.3 The Contractor shall demonstrate the capability to track sample location and transfers within the laboratory facility.

2.4 Sample Storage

The Contractor shall designate storage areas for USEPA samples and prepared samples.

2.5 Sample Tracking and Document Control

2.5.1 The Contractor shall record all activities performed on USEPA samples.

2.5.2 Titles which identify the activities recorded shall be printed on each page of all laboratory documents. (Activities include, but are not limited to: sample receipt; sample storage; sample preparation, and sample analysis.) When a document is a record of analysis, the instrument type and parameter group [e.g., Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES) (metals)] shall be included in the title.

2.5.3 Reviewers' signatures shall be identified on laboratory documents when reviews are conducted.

NOTE: Individuals recording review comments on computer-generated raw data are not required to be identified unless the written comments address data validity.

2.5.4 The laboratory name shall be identified on preprinted laboratory documents.

2.5.5 Each laboratory document entry shall be dated with the month/day/year (e.g., 01/01/2007) and signed by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.

2.5.6 Notations on laboratory documents shall be recorded in ink.

2.5.7 Corrections to laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.5.8 Unused portions of laboratory documents shall be lined-out, signed (or initialed), and dated.

2.5.9 Pages in bound and unbound logbooks shall be sequentially numbered.

2.5.10 Instrument-specific run logs shall be maintained to enable the reconstruction of run sequences.

2.5.11 Logbook entries shall be in chronological order.

2.5.12 Logbook entries shall include only one SDG per page, except in the events where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs).

2.5.13 Each page in bound and unbound logbooks shall be dated (month/day/year) and signed (no initials) at the bottom by the individual recording the activity (if a single entry is made on a page) or by the last individual recording information on the page (if multiple entries are on the same page).

2.5.14 Information inserted into laboratory documents shall be affixed permanently in place. The individual responsible for inserting information shall sign and date across the insert and logbook page at the time information is inserted.

2.5.15 The Contractor shall document disposal or retention of USEPA samples, remaining portions of samples, and prepared samples.

2.6 Computer-Resident Sample Data Control

2.6.1 Contractor personnel responsible for original data entry shall be identified at the time of data input.

2.6.2 The Contractor shall make changes to electronic data in a manner which ensures that the original data entry is preserved, the editor is identified, and the revision date is recorded.

2.6.3 The Contractor shall routinely verify the accuracy of manually entered data, electronically entered data, and data acquired from instruments.

2.6.4 The Contractor shall routinely verify documents produced by the electronic data collection system to ensure accuracy of the information reported.

2.6.5 The Contractor shall ensure that the electronic data collection system is secure.

2.6.5.1 The electronic data collection system shall be maintained in a secure location.

2.6.5.2 Access to the electronic data collection system functions shall be limited to authorized personnel through utilization of software security techniques (e.g., log-ons or restricted passwords).

2.6.5.3 Electronic data collection systems shall be protected from the introduction of external programs or software (e.g., viruses).

2.6.6 The Contractor shall designate archive storage areas for electronic data and the software required to access the data.

2.6.7 The Contractor shall designate an individual responsible for maintaining archives of electronic data including the software.

2.6.8 The Contractor shall maintain the archives of electronic data and necessary software in a secure location. (Secure areas shall be accessible only to authorized personnel.)

2.7 Complete SDG File (CSF) Organization and Assembly

2.7.1 The Contractor shall designate a document control officer responsible for the organization and assembly of the CSF.

2.7.2 The Contractor shall designate a representative responsible for the organization and assembly of the CSF in the event that the document control officer is not available.

2.7.3 The Contractor shall maintain documents relating to the CSF in a secure location.

2.7.4 All original laboratory forms and copies of SDG-related logbook pages shall be included in the CSF.

2.7.5 Copies of laboratory documents in the CSF shall be photocopied in a manner to provide complete and legible replicates.

2.7.6 Documents relevant to each SDG including, but not limited to, the following shall be included in the CSF for Stage 2b deliverables:

- logbook pages;
- bench sheets;
- receiving records;
- airbill receipts;
- sample condition at receipt (pH, temperature, physical);
- screening records;
- preparation records;
- repreparation records;
- sample storage;
- sample temperature;
- storage temperature records;
- analytical records;
- reanalysis records;
- records of failed or attempted analysis;
- custody records;
- sample tracking records;
- raw data summaries;
- computer printouts;
- correspondence;
- FAX originals;
- library search results; and
- other.

2.7.7 The document control officer or a designated representative shall ensure that sample tags are encased in clear plastic bags before placing them in the CSF.

2.7.8 CSF documents shall be organized and assembled on an SDG-specific basis.

2.7.9 Original documents which include information relating to more than one SDG (e.g., Traffic Reports/Chain of Custody Records, calibration logs) shall be filed in the CSF of the lowest SDG number, and copies of these originals shall be placed in the other CSF(s). The document control officer or a designated representative shall record the following statement on the copies in (indelible) dark ink:

COPY
ORIGINAL DOCUMENTS ARE INCLUDED IN CSF _____

_____
Signature

_____
Date

2.7.10 All CSFs shall be submitted with a completed Form DC-2. All resubmitted CSFs shall be submitted with a new or revised Form DC-2.

2.7.11 Each item in the CSF and resubmitted CSFs shall be inventoried and assembled in the order specified on Form DC-2. Each page of the CSF shall be stamped with a sequential number. Page number ranges shall be recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence shall be recorded in the "Comments" section on Form DC-2. When inserting new or inadvertently omitted documents, the Contractor shall identify them with unique accountable numbers. The unique accountable numbers and the locations of the documents shall be recorded in the "Other Records" section on Form DC-2.

2.7.12 Before shipping each CSF, the document control officer or a designated representative shall verify the agreement of information recorded on all documentation and ensure that the information is consistent and the CSF is complete.

2.7.13 The document control officer or a designated representative shall document the shipment of deliverable packages including what was sent, to whom, the date, and the carrier used.

2.7.14 Shipments of deliverable packages, including resubmittals, shall be sealed with custody seals by the document control officer or a designated representative in a manner such that opening the packages would break the seals.

2.7.15 Custody seals shall be signed and dated by the document control officer or a designated representative when sealing deliverable packages.

3.0 WRITTEN STANDARD OPERATING PROCEDURES

The Contractor shall develop and implement the following written Standard Operating Procedures (SOPs) for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and Complete Sample Delivery Group (SDG) File (CSF) organization and assembly to ensure accountability for USEPA sample chain-of-custody and control of all USEPA sample-related records.

3.1 Sample Receiving

3.1.1 The Contractor shall have written SOPs for sample receiving which accurately reflect the procedures used by the laboratory.

3.1.2 The written SOPs for sample receiving shall ensure that the procedures listed below are in use at the laboratory.

3.1.2.1 The condition of shipping containers and sample containers are inspected and recorded on Form DC-1 upon receipt by the sample custodian or a designated representative.

3.1.2.2 The condition of custody seals are inspected and recorded on Form DC-1 upon receipt by the sample custodian or a designated representative.

3.1.2.3 The presence or absence of the following documents/items accompanying the sample shipment is verified and recorded on Form DC-1 by the sample custodian or a designated representative:

- Custody seals;
- Traffic Reports/Chain of Custody Records or Packing Lists;
- Airbills or airbill stickers;
- Sample tags; and
- Cooler temperature indicator bottle.

3.1.2.4 The agreement or disagreement of information recorded on shipping documents with information recorded on sample containers is verified and recorded on Form DC-1 by the sample custodian or a designated representative.

3.1.2.5 The following information is recorded on Form DC-1 by the sample custodian or a designated representative as samples are received and inspected:

- Custody seal numbers, when present;
- Airbill or airbill sticker numbers;
- Sample tag numbers listed/not listed on Traffic Reports/Chain of Custody Records;
- Condition of sample bottles;
- Cooler temperature;
- Date of receipt;
- Time of receipt;
- EPA sample numbers;

- pH of all aqueous/water samples;
- Sample tag numbers;
- Assigned laboratory numbers;
- Remarks regarding condition of sample shipment, etc.;
- Samples delivered by hand; and
- Problems and discrepancies.

3.1.2.6 All accompanying forms are signed, dated, and the time is recorded, when applicable, at the time of sample receipt (e.g., Traffic Reports/Chain of Custody Records or packing lists, and airbills) by the sample custodian or a designated representative.

3.1.2.7 The Sample Management Office (SMO) is contacted to resolve problems and discrepancies including, but not limited to: absent documents; conflicting information; absent or broken custody seals; insufficient sample volume; unsatisfactory sample condition (e.g., leaking sample container); and samples not preserved to the proper pH.

3.1.2.8 The resolution of all problems and discrepancies communicated through SMO is recorded.

3.2 Sample Identification

3.2.1 The Contractor shall have written SOPs for sample identification which accurately reflect the procedures used by the laboratory.

3.2.2 The written SOPs for sample identification shall ensure that the procedures listed below are in use at the laboratory.

3.2.2.1 The identity of USEPA samples and prepared samples is maintained throughout the laboratory when:

- The Contractor assigns unique laboratory sample identification numbers, the written SOPs shall include a description of the procedure used to assign these numbers;
- The Contractor uses prefixes or suffixes in addition to laboratory sample identification numbers, the written SOPs shall include their definitions; and
- The Contractor uses methods to uniquely identify fractions/parameter groups and matrix type, the written SOPs shall include a description of these methods.

3.2.2.2 Each sample and sample preparation container is labeled with the EPA Sample Number, or a unique laboratory sample identification number.

3.3 Sample Security

3.3.1 The Contractor shall have written SOPs for sample security which accurately reflect the procedures used by the laboratory.

3.3.2 The written SOPs for sample security shall include the items listed below.

3.3.2.1 Procedures which ensure the following:

- Sample custody is maintained; and

- The security of designated secure areas is maintained.

3.3.2.2 A list of authorized personnel who have access to locked storage areas.

3.4 Sample Storage

3.4.1 The Contractor shall have written SOPs for sample storage which accurately reflect the procedures used by the laboratory.

3.4.2 The written SOPs for sample storage shall describe the locations, contents, and identities of all storage areas for USEPA samples and prepared samples in the laboratory.

3.5 Sample Tracking and Document Control

3.5.1 The Contractor shall have written SOPs for sample tracking and document control which accurately reflect the procedures used by the laboratory.

3.5.2 The written SOPs for sample tracking and document control shall include the items listed below.

3.5.2.1 Examples of all laboratory documents used during sample receiving, sample storage, sample transfer, sample analyses, CSF organization and assembly, and sample retention or disposal.

3.5.2.2 Procedures which ensure the following:

- All activities performed on USEPA samples are recorded;
- Titles which identify the activities recorded are printed on each page of all laboratory documents;
- Information recorded in columns is identified with column headings;
- Reviewers' signatures are identified on laboratory documents;
- The laboratory name is included on preprinted laboratory documents;
- Laboratory document entries are signed and dated with the month/day/year (e.g., 01/01/2007);
- Entries on all laboratory documents are recorded in ink;
- Corrections and additions to laboratory documents are made by drawing single lines through the errors, entering the correct information, and initialing and dating the new information;
- Unused portions of laboratory documents are lined-out, signed (or initialed), and dated;
- Pages in bound and unbound logbooks are sequentially numbered;
- Instrument-specific run logs are maintained to enable the reconstruction of run sequences;
- Logbook entries are recorded in chronological order;
- Entries are recorded for only one SDG on a page, except in the event where SDGs "share" Quality Control (QC) samples (e.g., instrument run logs and extraction logs);

- Each page in bound and unbound logbooks shall be dated (month/day/year) and signed (no initials) at the bottom by the individual recording the activity (if a single entry is made on a page) or by the last individual recording information on the page (if multiple entries are on the same page);
- Information inserted in laboratory documents is affixed permanently, signed, and dated across the insert; and
- The retention or disposal of USEPA samples, remaining portions of samples, and prepared samples is documented.

3.6 Computer-Resident Sample Data Control

3.6.1 The Contractor shall have written SOPs for computer-resident sample data control which accurately reflect the procedures used by the laboratory.

3.6.2 The written SOPs for computer-resident sample data control shall include the items listed below.

3.6.2.1 Procedures which ensure the following:

- Contractor personnel responsible for original data entry are identified;
- Changes to electronic data are made such that the original data entry is preserved, the editor is identified, and the revision date is recorded;
- The accuracy of manually entered data, electronically entered data, and data acquired from instruments is verified;
- Report documents produced by the electronic data collection system are routinely verified to ensure the accuracy of the information reported;
- Electronic data collection system security is maintained;
- Archives of electronic data and accompanying software are maintained in a secure location; and
- Off-site backup and storage of electronic data is maintained.

3.6.2.2 Descriptions of archive storage areas for the electronic data and the software required to access data archives.

3.6.2.3 A list of authorized personnel who have access to electronic data collection system functions and to archived data.

3.7 CSF Organization and Assembly

3.7.1 The Contractor shall have written SOPs for CSF organization and assembly which accurately reflect the procedures used by the laboratory.

3.7.2 The written SOPs for CSF organization and assembly shall ensure that the procedures listed below are in use at the laboratory.

- Documents relating to the CSF are maintained in a secure location.
- All original laboratory forms and copies of SDG-related logbook pages are included in the CSF.

- Laboratory documents are photocopied in a manner to provide complete and legible replicates.
- All documents relevant to each SDG are included in the CSF.
- Sample tags are encased in clear plastic bags by the document control officer or a designated representative before placing them in the CSF.
- The CSF is organized and assembled on an SDG-specific basis.
- Original documents which contain information relating to more than one SDG are filed in the CSF of the lowest SDG and copies are referenced to originals in the event that an original document contains information relating to more than one SDG.
- Each CSF is submitted with a completed Form DC-2, and resubmitted CSFs are submitted with a new or revised Form DC-2.
- Each page of the CSF is stamped with a sequential number and the page number ranges are recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence are recorded in the "Comments" section of Form DC-2. Inserted documents are recorded in the "Other Records" section of Form DC-2.
- Consistency and completeness of the CSF are verified by the document control officer or a designated representative.
- Shipments of deliverable packages are documented by the document control officer or a designated representative.
- Deliverable packages are shipped by the document control officer or a designated representative using custody seals in a manner such that opening the packages would break the seals.
- Custody seals are signed and dated by the document control officer or a designated representative before placing them on deliverable packages.

EXHIBIT G

GLOSSARY OF TERMS

ABSORBANCE - A measure of the decrease in incident light passing through a sample into a detector. It is defined mathematically as:

Absorbance

$$A = -\log \frac{I}{I_O}$$

WHERE, I = Radiation intensity of a sample.
I_o = Radiation intensity of a blank.

ALIQUOT - A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

ANALYSIS DATE/TIME - The date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE - The element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL REFERENCE STANDARD - Standards purchased from private chemical supply houses used to prepare calibration standards, and Continuing Calibration Verification (CCV) standards.

ANALYTICAL SAMPLE - Any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), Continuing Calibration Blank (CCB), and tunes. Note the following are all defined as analytical samples: undiluted and diluted samples [U.S. Environmental Protection Agency (USEPA) and non-USEPA], matrix spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, Interference Check Samples (ICSs), Laboratory Control Samples (LCSs), Performance Evaluation (PE) samples, Preparation Blanks, and cyanide MIDRANGE samples.

ANALYTICAL SEQUENCE - The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB). All sample analyses during the analytical sequence are subject to the Quality Control (QC) protocols set forth in Exhibits D and E of this contract unless otherwise specified in the individual methods.

ANALYTICAL SERVICES BRANCH (ASB) - The division of the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

ANALYTICAL SPIKE - A spike that is fortified just prior to analysis by adding a known quantity of the analyte to an aliquot of the prepared sample. (Also called post-digestion spike).

ASTM - ASTM International. A developer and provider of voluntary consensus standards.

AUTOZERO - Zeroing the instrument at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

BACKGROUND CORRECTION - A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - A group of samples prepared at the same time in the same location using the same method.

BLANK - An analytical sample designed to assess specific sources of contamination. See the individual definitions for types of blanks.

CALIBRATED MASS - 1) A mass whose apparent mass has been adjusted from the uncalibrated mass by the instrumental mass calibration software routine. 2) An analyte mass whose intensity counts have been calibrated against standards of known analyte concentration.

CALIBRATION - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation. This blank is not subjected to the preparation method for Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma—Mass Spectrometry (ICP-MS), but is digested for mercury and cyanide.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

CASE - A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

CONCENTRATION LEVEL (low or medium) - For inorganics analysis, low or medium level is defined by the appropriate designation by the sampler on the Traffic Report/Chain of Custody Record.

CONTAMINATION - A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents laboratory environment, or analytical instruments.

CONTINUING CALIBRATION VERIFICATION (CCV) - A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples. The CCV should have a concentration in the middle of the calibration range and shall be run at the beginning of the day prior to the analysis of samples, and every 2 hours (1 hour for mercury and cyanide).

CONTRACT COMPLIANCE SCREENING (CCS) - A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is done under USEPA direction by the Sample Management Office (SMO) Contractor.

CONTRACT LABORATORY PROGRAM (CLP) - Supports USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) of USEPA.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

CYANIDE (Total) - Cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

DATE - The date format for all reporting forms is MM/DD/YYYY - Where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YYYY = 2007, 2008, 2009, etc.

DAY - Unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - An official record of the sample preparation (digestion or distillation).

DIRECT ANALYSIS - Analysis of a sample, standard, or blank that has not been taken through a preparation procedure (digestion or distillation).

DISSOLVED METALS - Analyte elements in an aqueous/water sample which will pass through a 0.45 micrometer (µm) filter.

DRY WEIGHT - The weight of a sample based on percent solids. The weight after drying in an oven.

DUPLICATE - A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

FIELD BLANK - This is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

FIELD QC - Any Quality Control (QC) sample submitted from the field to the laboratory. Examples include, but are not limited to field blanks, field duplicates, and field spikes.

FIELD SAMPLE - A portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique EPA Sample Number.

HOLDING TIME - Contractual holding time is the elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analysis.

Holding time = (sample analysis date - sample receipt date)

INDEPENDENT STANDARD - A Contractor-prepared standard solution that is composed of analytes from a different source than those used in the standards for the calibration.

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICP-AES) – A technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio-frequency inductively coupled plasma.

INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS) - A technique for the multi-element determination of elements in solution. The basis of the technique is the detection of atomic ions produced by an ICP and sorted by mass-to-charge (m/z) ratio.

IN-HOUSE - At the Contractor's facility.

INITIAL CALIBRATION - Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INITIAL CALIBRATION VERIFICATION (ICV) - Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to NIST or other certified standard sources when USEPA ICV solutions are not available.

INJECTION - Introduction of the analytical sample into the instrument excitation system to measure absorbance, emission, or concentration of an analyte. May also be referred to as exposure.

INSUFFICIENT QUANTITY - When there is not enough volume (aqueous/water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis, percent solids, etc. Exhibit D provides guidance for addressing this problem.

INTERFERENCE CHECK SAMPLE (ICS) - A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INTERFERENTS - Substances which affect the analysis for the element of interest.

INTERNAL STANDARD - A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

LABORATORY - Synonymous with Contractor as used herein.

LABORATORY CONTROL SAMPLE (LCS) – A sample spiked at a known concentration. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the U.S. Environmental Protection Agency (USEPA) samples received.

LABORATORY RECEIPT DATE - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report/Chain of Custody Record. Also referred to as VTSR (Validated Time of Sample Receipt).

MATRIX - The predominant material of which the sample to be analyzed is composed. For the purpose of this Statement of Work (SOW), a sample matrix is either aqueous/water, soil/sediment, wipe, or small (e.g., 37 mm) air filter. Matrix is not synonymous with phase (liquid or solid).

MATRIX EFFECT - In general, the effect of particular matrix constituents.

MATRIX SPIKE - Aliquot of a sample (aqueous/water or soil/sediment) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD DETECTION LIMIT (MDL) - The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

MONITORED MASS - A mass that counts are collected from during analysis that may be subsequently used in isobaric correction equations or for the interpretation of possible interferences in analyte mass results.

PERCENT DIFFERENCE (%D) - As used in this Statement of Work (SOW) and elsewhere to compare two values. The difference between the two values divided by one of the values.

PERCENT SOLIDS (%S) - The proportion of solid in a soil/sediment sample determined by drying an aliquot of the sample.

PERFORMANCE EVALUATION (PE) SAMPLE - A sample of known composition provided by USEPA for Contractor analysis. Used by USEPA to evaluate Contractor performance.

PREPARATION BLANK - An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

PREPARATION LOG - An official record of the sample preparation (digestion or distillation).

QUALITY ASSURANCE TECHNICAL SUPPORT (QATS) LABORATORY - A Contractor-operated facility operated under the QATS contract, awarded and administered by USEPA.

REAGENT WATER - The purity of this water must be equivalent to ASTM Type II reagent water of Specification D1193-06, "Standard Specification for Reagent Water".

REFERENCE MATERIAL - Standards, typically provided by USEPA, used to verify method and instrument performance. Examples include Initial Calibration Verification (ICV) standards and Interference Check Solution (ICS) standards.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this Statement of Work (SOW) and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

REPRESENTATIVE - Alternate or designee who has the knowledge and authority to perform a specific task.

ROUNDING RULES - If the figure is greater than or equal to 5, round up, otherwise round down. As an example, 11.443 is rounded down to 11.44 and 11.455 is rounded up to 11.46. If a series of multiple operations is to be performed (i.e., add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures. See Forms Instructions (Exhibit B) for exceptions.

RUN - A continuous analytical sequence consisting of prepared samples and all associated Quality Assurance (QA) measurements as required by the contract Statement of Work (SOW). A run begins with the instrument calibration or tune.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE DELIVERY GROUP (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- All samples scheduled with the same level of deliverables.
- In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG.

Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory. Laboratories shall take all precautions to meet the 20 sample per SDG criteria.

SAMPLE MANAGEMENT OFFICE (SMO) - A Contractor-operated facility operated under the SMO contract, awarded and administered by USEPA.

SAMPLE NUMBER (EPA SAMPLE NUMBER) - A unique identification number designated by USEPA for each sample. The EPA Sample Number appears on the Sample Traffic Report/Chain of Custody Record which documents information on that sample.

SDG NARRATIVE - Portion of the data package which includes laboratory, contract, Case, sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. Complete SDG Narrative specifications are included in Exhibit B.

SENSITIVITY - The slope of the analytical curve (i.e., functional relationship between instrument response and concentration).

SERIAL DILUTION - The dilution of a sample by a factor of five. 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.

SOIL - Synonymous with soil/sediment as used herein.

SOP - Standard Operating Procedure.

SOW - Statement of Work.

STANDARD ANALYSIS - An analytical determination made with known quantities of target analytes.

STOCK SOLUTION - A standard solution which can be diluted to derive other standards.

TARGET ANALYTE LIST (TAL) - A list of Inorganic Analytes (metals and cyanide) as designated in Exhibit C.

TIME - HH:mm:ss - When required to record time on any deliverable item, time shall be expressed as Military Time [i.e., a 24-hour clock (0000-2359)].

TRAFFIC REPORT/CHAIN OF CUSTODY RECORD (TR/COC) – A USEPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and is used for documenting sample identity, sample chain-of-custody, and sample receipt by the laboratory.

TUNE - Analysis of a solution containing a range of isotope masses to establish Inductively Coupled Plasma—Mass Spectrometry (ICP-MS) accuracy, resolution, and precision prior to calibration.

USEPA OSRTI ASB INORGANIC PROGRAM MANAGER (ASB PM) – The U.S. Environmental Protection Agency (USEPA), Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Official who manages the Contract Laboratory Program (CLP) Inorganic Program.

USEPA REGIONAL CLP PROJECT OFFICER (CLP PO) - The Regional U.S. Environmental Protection Agency (USEPA) official responsible for monitoring laboratory performance and/or requesting analytical data or services from a Contract Laboratory Program (CLP) laboratory.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the Contractor’s facility, as recorded on the shipper’s delivery receipt and Traffic Report/Chain of Custody Record.

WET WEIGHT - The weight of a sample aliquot including moisture (undried).

EXHIBIT H

FORMAT FOR ELECTRONIC DATA DELIVERABLES

Table of Contents

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1.0 FORMAT CHARACTERISTICS

1.1 This constitutes an implementation of the Staged Electronic Data Deliverable (SEDD) based on analytical results and ancillary information required by the contract. Because this implementation is specific to the contract, not all data elements listed in the cross-program Document Type Definition (DTD) are required. This implementation is based on SEDD Specification 5.2 that can be found at:

http://www.epa.gov/superfund/programs/clp/sedd.htm

1.1.1 The SEDD deliverable consists of an eXtensible Markup Language (XML) file(s) compliant with the XML specification 1.0 of the World Wide Web Consortium (W3C). The deliverable must be well-formed based on the W3C XML specification and must be valid based on the DTD.

1.1.2 The Contractor shall create the deliverable using the UTF-8 (Unicode Transformation Format - 8 bit) character set.

1.1.3 The initial line of the deliverable shall be: <?xml version="1.0" encoding="UTF-8"?>.

1.1.4 The second line of the deliverable shall be a DOCTYPE line that contains the filename of the DTD. The DOCTYPE line shall be <!DOCTYPE Header SYSTEM "SEDD_5-2_GENERAL_2b_3.dtd"> or <!DOCTYPE Header SYSTEM “SEDD_5-2_GENERAL_2a_2.dtd”> where "Header" denotes the name of the root element, and "SEDD_5-2_GENERAL_2b_3.dtd" (for a Level 2b deliverable) or “SEDD_5-2_GENERAL_2a_2.dtd” (for a Level 2a deliverable) denotes the filename of the DTD.

1.1.5 The use of XML comment lines is permitted at any position in the file after the first two lines.

1.2 This implementation includes detailed specifications for the required format of the content of each data element for each fraction. The content of each data element is specified as either literal (contained in quotes) which must appear exactly as shown (without quotes), or as a variable for which descriptions and formats are listed. Exhibit H, Section 2.0 describes requirements for each data element.

1.2.1 For this implementation, numeric data elements may contain numeric digits, a decimal place, and a leading minus sign. Values without a leading minus sign are assumed to be positive. Values must be reported to the specified precision or significance.

1.2.2 The values reported by the Contractor are used for data assessment. The Contractor shall not use rounded intermediate values in calculating the final result, and no rounding shall be performed until reaching the final result.

1.2.3 The completeness of analytical data provided in the EDD will be verified against the analytical data requested on the Traffic Report/Chain of Custody (TR/COC). The laboratory code, Case number, contract number, Sample Delivery Group (SDG) number, sample number, and fraction shall be identical in the EDD and the TR/COC and the SDG coversheet submitted by the Contractor for the SDG.

1.2.4 The following variables must be present where required and correct: QC Type; instrument ID; correlation coefficient; intercept; analysis date and time; method ID; collected date; matrix; run batch; analysis batch; analysis group ID; client analysis ID; client analyte ID; preparation batch; percent recovery; Relative Percent Difference (RPD); Percent Difference (%D), Percent Relative Standard Deviation (%RSD).

2.0 DATA ELEMENTS

2.1 The Staged Electronic Data Deliverable (SEDD) consists of data elements arranged hierarchically by data nodes (parent elements). Figures 1 and 2 depict the data node hierarchy. Each data element consists of a start tag, content, and an end tag. An element may contain other elements (child elements).

NOTE: There shall be no more than one occurrence of each child element within a node, unless the child element also behaves as a parent element. For example, in each SamplePlusMethod node, there may be only one occurrence of the element ClientSampleID, but there may be more than one occurrence of the element Analysis.

The tags, nodes, and hierarchy are specified in the Document Type Definition against which the deliverable will be validated (see Exhibit H, Section 5.0). The frequency requirements for each of the data nodes applicable to this implementation are described below.

2.1.1 Header Node (Required for All Deliverable Levels)

One Header node must be reported for each fraction.

2.1.2 SamplePlusMethod Node (Required for All Deliverable Levels)

Each Header node must contain one SamplePlusMethod node for each field sample, field blank (including rinse, equipment, and trip blanks), Performance Evaluation sample, Matrix Spike sample, any required post-digestion spike sample, Duplicate sample, Serial Dilution sample, Preparation Blank, Laboratory Control Sample, and non-client sample analyzed.

2.1.3 ReportedResult Node (Required for All Deliverables Levels)

Each SamplePlusMethod node must contain a ReportedResult node for each target analyte.

2.1.4 ContactInformation Node (Required for All Deliverable Levels)

Each Header node must contain one ContactInformation node.

2.1.5 InstrumentQC Node (Required for Level 2b Deliverables Only)

Each Header node must contain one InstrumentQC node for each instrument performance check (ICP-MS Tune), initial calibration sequence, Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Interference Check Sample (ICSA and ICSAB).

2.1.6 AnalysisGroup Node (Required for Level 2b Deliverables Only)

Each initial calibration InstrumentQC node for multi-point calibration must contain one AnalysisGroup node containing summary data for the initial calibration. Each AnalysisGroup node must contain one Analyte node for each target analyte.

2.1.7 Analysis Node (Required for All Deliverable Levels)

Each SamplePlusMethod node must contain at least one Analysis node and each InstrumentQC node (other than Initial Calibration) must contain one Analysis node.

2.1.8 Analyte Node (Required for All Deliverable Levels)

Each Analysis node under a SamplePlusMethod node must contain one Analyte node for each target analyte, monitored interferent, and internal standard. Each Analysis node under an InstrumentQC node must contain one Analyte node for each target compound, monitored interferent, and internal standard. Each Analysis node under an InstrumentQC node for tune must contain one Analyte node for each tune analyte. Each AnalysisGroup node must contain one Analyte node for each target analyte.

2.1.9 PreparationPlusCleanup Node (Required for All Deliverable Levels)

Each Analysis node under a SamplePlusMethod node must contain one PreparationPlusCleanup node with a PreparationPlusCleanupType equal to "Preparation". For Serial Dilution and Post-Digestion Spike samples, the associated PreparationPlusCleanup node shall contain data for the preparation of the original sample. For those methods requiring digested Quality Control (QC), each InstrumentQC node must contain one PreparationPlusCleanup node with a PreparationPlusCleanupType equal to "Preparation".

2.1.10 Peak Node (Required for Level 2b Deliverables Only)

Each Analyte node under InstrumentQC must contain at least one Peak node. Within a RunBatch, a peak must be consistently identified.

2.1.11 PeakComparison Node (Required for Level 2b Deliverables Only)

For Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), each Peak node under an InstrumentQC must contain a PeakComparison node for each applicable internal standard.

2.1.12 Characteristic Node (Required for All Deliverable Levels)

Each SamplePlusMethod and PreparationPlusCleanup node may contain one or more Characteristic nodes, one for each sample characteristic that must be reported for a sample at time of receipt, or after preparation.

2.1.13 Handling and AnalyteGroup Nodes

These nodes are not required for this implementation.

2.2 Detailed instructions for the content of each data element are provided in Tables 1 and 2. The following is an explanation of the data fields contained in each table.

2.2.1 Node and Data Elements

This field reports each node in bold text, followed by its data elements. If an entire node is not required, then none of its data elements are listed.

2.2.2 Applicability

This field reports the samples, blanks, and standards for which each node and data element is required. An "X" in a column indicates that the node or element is required. Sample refers to field samples, field blanks, and Performance Evaluation samples unless otherwise noted. Abbreviations used in this field are defined in Table 3.

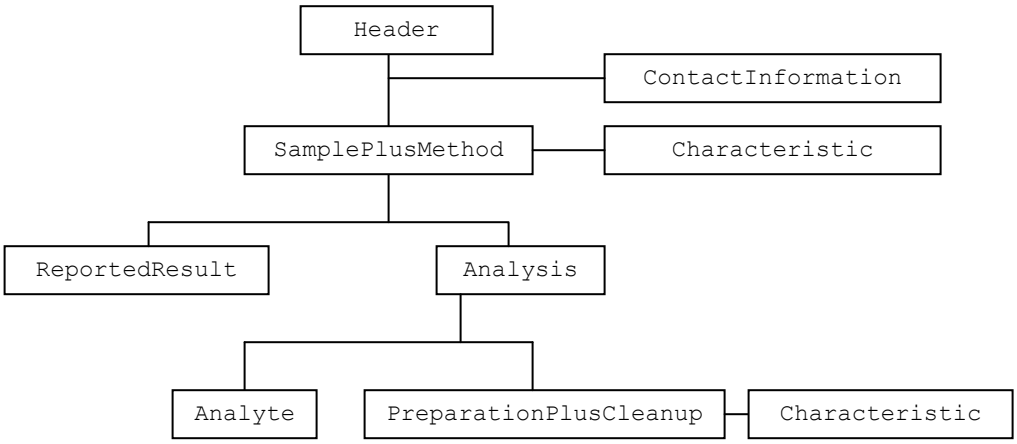

Figure 1: Data Node Hierarchy for
Level 2a Deliverable

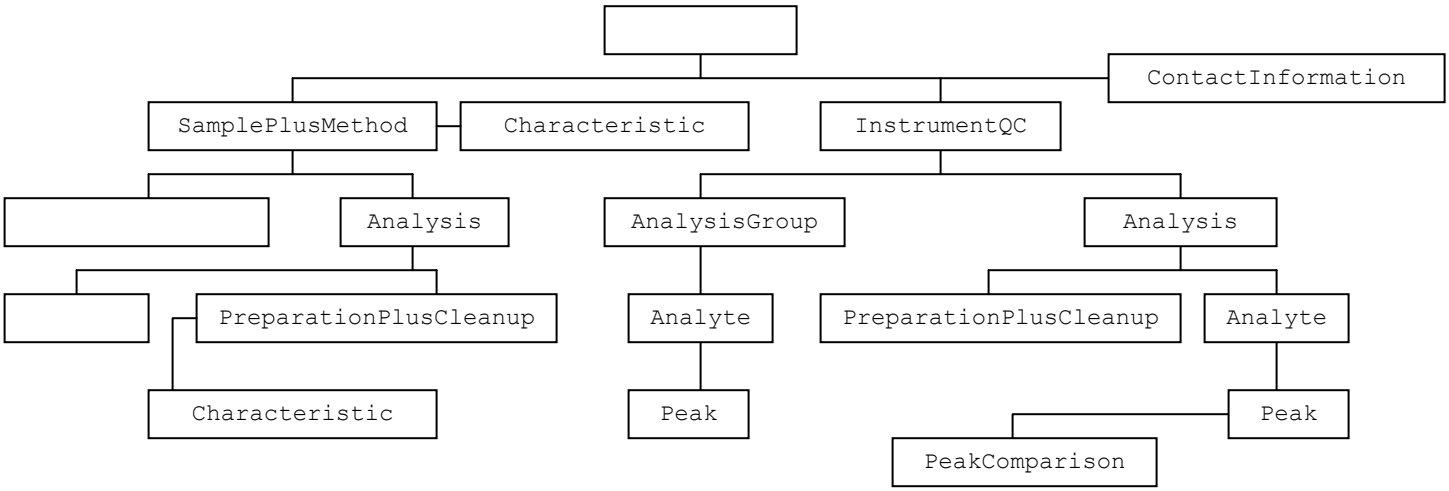

Figure 2: Data Node Hierarchy for
Level 2b Deliverable

2.2.3 Instructions

This field describes the required format and content of each data element. The content of each data element is specified as either literal (contained in quotes), or as a variable for which description and format is listed. Abbreviations used in this field are defined in Table 3.

3.0 BATCHES

3.1 This implementation requires the use of the following batches from the Staged Electronic Data Deliverable (SEDD) Specification: "LabReportingBatch"; "RunBatch"; "AnalysisBatch"; and "PreparationBatch".

3.1.1 The "LabReportingBatch" links all samples reported in the same Sample Delivery Group (SDG). Report the SDG Number.

3.1.2 The "RunBatch" links all analyses performed under the same initial calibration. All analyses performed under an initial calibration must have the same content for the "RunBatch" element as the initial calibration from which their results are calculated.

3.1.3 The "AnalysisBatch" and "AnalysisBatchEnd" link all analyses performed within the same analytical sequence [1- or 2-hour period and Quality Control (QC)]. All analyses performed within the same analytical sequence must have the same content for the "AnalysisBatch" element as the tune or standard(s) that began the analytical sequence, and the same content for the "AnalysisBatchEnd" as the standard(s) that ends the analytical sequence.

3.1.4 The "PreparationBatch" links all samples of the same matrix prepared at the same time by the same preparation method. All samples analyzed, including Preparation Blanks, Matrix Spikes, Duplicates, and Laboratory Control Samples (LCSs) that are prepared together must have the same content for the "PreparationBatch" element. For those methods using digested/distilled QC, all QC that are prepared together must have the same content for the "PreparationBatch" element.

4.0 DELIVERABLE

4.1 Each fraction in a Sample Delivery Group (SDG) shall be submitted as a separate compressed (zipped) file. For reporting requirements, the fractions are "ICP_AES"; "ICP_MS"; "Hg"; and "CN". All fractions within an SDG shall be submitted at the same time [i.e., the file for the second fraction in an SDG shall be submitted in a single file upload with the first fraction].

4.2 The laboratory will utilize a designated website (provided in their Laboratory Welcome Package) to electronically submit their Electronic Data Deliverable (EDD) to the Sample Management Office (SMO). The U.S. Environmental Protection Agency (USEPA) may approve alternative electronic means of file delivery. Written permission must be obtained from the USEPA Analytical Services Branch (ASB) prior to the use of any alternative means.

4.3 The laboratory must follow the delivery instructions in Exhibit B of this Statement of Work (SOW) and deliver their hardcopy and EDD to SMO concurrently. If one of these items is delivered on a later date, the Data Receipt Date (DRD) for the SDG will be the later of the two dates.

4.4 Information in the electronic deliverable must correspond to information submitted in the hardcopy raw data package and on Quality Control (QC) summary forms. If information in the raw data or on the forms is changed, the information in the electronic deliverable shall be changed accordingly. An electronic deliverable containing the changed information for the SDG shall be resubmitted along with the hardcopy at no additional cost to the USEPA.

4.5 The format for the file name shall be Case number_SDG number_contract number_submission number_DTD used_Fraction.zip. For example, the first submission of the ICP-AES fraction from SDG number MABC12, Case number 12345, contract 68-W-0000 would be named 12345_MABC12_68-W-0000_1_SEDD_5-2_GENERAL_2b_3_ICP_AES.zip.

5.0 DOCUMENT TYPE DEFINITION (DTD)

5.1 Introduction

The deliverable will be validated against DTD SEDD_5-2_GENERAL_2b_3 or DTD SEDD_5-2_GENERAL_2a_2. The deliverable must not contain any tags not included in the DTD and must conform to the hierarchical structure modeled in the DTD.

5.2 General Stage 2b DTD

```
<?xml version="1.0" encoding="UTF_8"?>
<!—SEDD_5-2_GENERAL_2b_3.dtd 10/22/2009 Based on SEDD Specification 5.2 -->
<!-- Acronym Description -->
<!-- Coeff - Coefficient -->
<!-- EDD   - Electronic Data Deliverable -->
<!-- ID    - Identity -->
<!-- Lab   - Laboratory -->
<!-- QC    - Quality Control -->
<!-- RPD   - Relative Percent Difference -->
<!-- RRF   - Relative Response Factor -->
<!-- RSD   - Relative Standard Deviation -->
<!ELEMENT Header (
                ClientID|
                ClientName|
                Comment|
                DateFormat|
                EDDID|
                EDDImplementationID|
                EDDImplementationVersion|
                EDDVersion|
                GeneratingSystemID|
                GeneratingSystemVersion|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
                LabDataPackageID|
                LabDataPackageName|
                LabDataPackageVersion|
                LabID|
                LabName|
                LabNarrative|
                LabQualifiersDefinition|
                LabReportedDate|
                ProjectID|
                ProjectName|
                SiteID|
                SiteName|
                ContactInformation|
                SamplePlusMethod|
                InstrumentQC
                        )*>
<!ELEMENT Analysis (
                AliquotAmount|
                AliquotAmountUnits|
                AnalysisBatch|
                AnalysisBatchEnd|
                AnalysisDuration|
```

```
                AnalysisDurationUnits|
                AnalysisGroupID|
                AnalysisType|
                Analyst|
                AnalyzedAmount|
                AnalyzedAmountUnits|
                AnalyzedDate|
                ClientAnalysisID|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Column|
                ColumnInternalDiameter|
                ColumnInternalDiameterUnits|
                ColumnLength|
                ColumnLengthUnits|
                Comment|
                ConfirmationAnalysisID|
                Counts|
                CountsUncertainty|
                CountsUncertaintyConfidenceLevel|
                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
                CountsUncertaintyLimitHigh|
                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
                DetectorID|
                DetectorType|
                DilutionFactor|
                Efficiency|
                HeatedPurge|
                Inclusion|
                InjectionVolume|
                InjectionVolumeUnits|
                InstrumentID|
                LabAnalysisID|
                LabFileID|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                ProcedureID|
                ProcedureName|
                ReferenceDate|
                ResultBasis|
                RunBatch|
```

```
                Temperature|
                TemperarureUnits|
                Wavelength|
                WavelengthUnits|
                Yield|
                PreparationPlusCleanup|
                Analyte|
                AnalyteGroup
                         )*>
<!ELEMENT AnalysisGroup (
                AnalysisGroupID|
                AnalysisType|
                Comment|
                Analyte|
                AnalyteGroup
                         )*>
<!ELEMENT Analyte (
                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                BiasErrorRatio|
                CalibrationBasis|
                CalibrationFactor|
                CalibrationFactorUnits|
                CalibrationType|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Coeffa0|
                Coeffa1|
                Coeffa2|
                Coeffa3|
                CoeffOfDetermination|
                CoeffOfDeterminationLimitLow|
                CoeffOfDeterminationLimitType|
                Comment|
                CorrelationCoeff|
                CorrelationCoeffLimitLow|
                CorrelationCoeffLimitType|
                Counts|
                CountsUncertainty|
                CountsUncertaintyConfidenceLevel|
                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
                CountsUncertaintyLimitHigh|
                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
                DetectionLimit|
                DetectionLimitType|
                DetectionLimitUnits|
                DifferenceErrorRatio|
                Efficiency|
                ExpectedResult|
                ExpectedResultUncertainty|
                ExpectedResultUncertaintyConfidenceLevel|
                ExpectedResultUncertaintyDetermination|
                ExpectedResultUncertaintyIntervalType|
```

```
                ExpectedResultUncertaintyLimitHigh|
                ExpectedResultUncertaintyLimitLow|
                ExpectedResultUncertaintyType|
                ExpectedResultUncertaintyUnits|
                ExpectedResultUnits|
                Inclusion|
                LabAnalyteID|
                LabQualifiers|
                LotNumber|
                Mass|
                MassUnits|
                MeanCalibrationFactor|
                MeanCalibrationFactorUnits|
                MeanRRF|
                MeanRRFLimitLow|
                MeanRRFLimitType|
                PeakID|
                PercentBreakdown|
                PercentBreakdownLimitHigh|
                PercentBreakdownLimitType|
                PercentDifference|
                PercentDifferenceLimitHigh|
                PercentDifferenceLimitLow|
                PercentDifferenceLimitType|
                PercentRecovery|
                PercentRecoveryLimitHigh|
                PercentRecoveryLimitLow|
                PercentRecoveryLimitType|
                PercentRecoveryType|
                PercentRSD|
                PercentRSDLimitHigh|
                PercentRSDLimitLow|
                PercentRSDLimitType|
                QuantitationBasis|
                QuantitationLimit|
                QuantitationLimitType|
                QuantitationLimitUnits|
                ReportingLimit|
                ReportingLimitType|
                ReportingLimitUnits|
                Result|
                ResultLimitHigh|
                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUncertaintyConfidenceLevel|
                ResultUncertaintyDetermination|
                ResultUncertaintyIntervalType|
                ResultUncertaintyLimitHigh|
                ResultUncertaintyLimitLow|
                ResultUncertaintyType|
                ResultUncertaintyUnits|
                ResultUnits|
                RPD|
                RPDLimitHigh|
                RPDLimitType|
                RPDType|
                RRF|
```

```
                RRFLimitLow|
                RRFLimitType|
                StandardSource|
                TailingFactor|
                TailingFactorLimitHigh|
                TailingFactorLimitType|
                Wavelength|
                WavelengthUnits|
                WeightingFactor|
                Peak
                         )*>
<!ELEMENT AnalyteGroup (
                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Comment|
                LabAnalyteID|
                LabQualifiers|
                Result|
                ResultType|
                ResultUncertainty|
                ResultUnits
                         )*>
<!ELEMENT Characteristic (
                CharacteristicType|
                CharacteristicValue|
                CharacteristicUnits|
                Comment
                        )*>
<!ELEMENT ContactInformation (
                LabAddress1|
                LabAddress2|
                LabCity|
                LabCountry|
                LabID|
                LabName|
                LabPointOfContact|
                LabPointOfContactElectronicAddress|
                LabPointOfContactTitle|
                LabPointOfContactType|
                LabState|
                LabTelephoneNumber|
                LabType|
                LabZipCode
                        )*>
<!ELEMENT Handling (
                Analyst|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
```

```
                HandledDate|
                HandlingBatch|
                HandlingType|
                InitialAmount|
                InitialAmountUnits|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                ProcedureID|
                ProcedureName|
                SampleAmount|
                SampleAmountUnits|
                Characteristic
                         )*>
<!ELEMENT InstrumentQC (
                ClientInstrumentQCType|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                LabID|
                LabInstrumentQCID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                QCLinkage|
                QCType|
                AnalysisGroup|
                Analysis
                         )*>
<!ELEMENT Peak (
                CalibrationFactor|
                CalibrationFactorUnits|
                CalibrationType|
                Coeffa0|
                Coeffa1|
                Coeffa2|
                Coeffa3|
                CoeffOfDetermination|
                CoeffOfDeterminationLimitLow|
```

```
CoeffOfDeterminationLimitType|
Comment|
CorrelationCoeff|
CorrelationCoeffLimitLow|
CorrelationCoeffLimitType|
DifferenceErrorRatio|
Efficiency|
Inclusion|
LabQualifiers|
Mass|
MassLimitHigh|
MassLimitLow|
MassLimitType|
MassUnits|
MeanCalibrationFactor|
MeanCalibrationFactorUnits|
MeanRetentionTime|
MeanRetentionTimeLimitHigh|
MeanRetentionTimeLimitLow|
MeanRetentionTimeLimitType|
MeanRetentionTimeUnits|
MeanRRF|
MeanRRFLimitLow|
MeanRRFLimitType|
PeakID|
PercentDifference|
PercentDifferenceLimitHigh|
PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentRecovery|
PercentRecoveryLimitHigh|
PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
PercentRSD|
PercentRSDLimitHigh|
PercentRSDLimitLow|
PercentRSDLimitType|
Resolution|
ResolutionLimitHigh|
ResolutionLimitLow|
ResolutionLimitType|
ResolutionType|
ResolutionUnits|
Result|
ResultLimitHigh|
ResultLimitLow|
ResultLimitType|
ResultType|
ResultUncertainty|
ResultUnits|
RRF|
RRFLimitLow|
RRFLimitType|
TailingFactor|
TailingFactorLimitHigh|
TailingFactorLimitType|
Wavelength|
WavelengthUnits|
WeightingFactor|
```

```
                PeakComparison
                         )*>
<!ELEMENT PeakComparison (
                Comment|
                PeakID|
                PercentRatio|
                PercentRatioLimitHigh|
                PercentRatioLimitLow|
                PercentRatioLimitType
                         )*>
<!ELEMENT PreparationPlusCleanup (
                AliquotAmount|
                AliquotAmountUnits|
                Analyst|
                CleanedUpDate|
                CleanupBatch|
                CleanupType|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                FinalAmount|
                FinalAmountUnits|
                InitialAmount|
                InitialAmountUnits|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                LotNumber|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                PreparationPlusCleanupType|
                PreparationType|
                PreparedDate|
                ProcedureID|
                ProcedureName|
                Solvent|
                Characterisitic
                         )*>
<!ELEMENT ReportedResult (
                AnalysisGroupID|
                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                BiasErrorRatio|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
```

```
ClientDetectionLimit|
ClientDetectionLimitUnits|
ClientQuantitationLimit|
ClientQuantitationLimitUnits|
Comment|
DetectionLimit|
DetectionLimitType|
DetectionLimitUnits|
DifferenceErrorRatio|
ExpectedResult|
ExpectedResultUncertainty|
ExpectedResultUncertaintyConfidenceLevel|
ExpectedResultUncertaintyDetermination|
ExpectedResultUncertaintyIntervalType|
ExpectedResultUncertaintyLimitHigh|
ExpectedResultUncertaintyLimitLow|
ExpectedResultUncertaintyType
ExpectedResultUncertaintyUnits|
ExpectedResultUnits|
LabAnalysisID|
LabAnalyteID|
LabQualifiers|
LabResultStatus|
PeakID|
PercentDifference|
PercentDifferenceLimitHigh|
PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentRecovery|
PercentRecoveryLimitHigh|
PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
QuantitationLimit|
QuantitationLimitType|
QuantitationLimitUnits|
ReportingLimit|
ReportingLimitType|
ReportingLimitUnits|
Result|
ResultLimitHigh|
ResultLimitLow|
ResultLimitType|
ResultType|
ResultUncertainty|
ResultUncertaintyConfidenceLevel|
ResultUncertaintyDetermination|
ResultUncertaintyIntervalType|
ResultUncertaintyLimitHigh|
ResultUncertaintyLimitLow|
ResultUncertaintyType|
ResultUncertaintyUnits|
ResultUnits|
RetentionTime|
RetentionTimeUnits|
RPD|
RPDLimitHigh|
RPDLimitType|
RPDType
         )*>
```

```
<!ELEMENT SamplePlusMethod (
                ClientID|
                ClientMethodCategory|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodType|
                ClientMethodVersion|
                ClientName|
                ClientSampleID|
                CollectedDate|
                CollectedEndDate|
                Comment|
                Composite|
                CoolerID|
                CustodyID|
                EquipmentBatch|
                Filtered|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
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                LabMethodName|
                LabName|
                LabReceiptDate|
                LabReportingBatch|
                LabSampleID|
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                LocationName|
                MatrixID|
                MatrixMedium|
                MethodBatch|
                MethodCategory|
                MethodCode|
                MethodID|
                MethodLevel|
                MethodModificationDescription|
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                MethodName|
                MethodSource|
                MethodType|
                MethodVersion|
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                OriginalLabSampleID|
                PhaseAnalyzed|
                Preservative|
                ProjectID|
                ProjectName|
                QCCategory|
                QCLinkage|
                QCType|
                Quarantine|
                SamplingBatch|
                ShippingBatch|
                SiteID|
```

```
                SiteName|
                StorageBatch|
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                Handling|
                AnalysisGroup
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<!ELEMENT WeightingFactor (#PCDATA)>
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5.3 General Stage 2a DTD

```
<?xml version="1.0" encoding="UTF-8"?>
<!—SEDD_5-2_GENERAL_2a_2.dtd 07/21/2008 Based on SEDD Specification 5.2 -->
<!-- Acronym Description -->
<!-- EDD   - Electronic Data Deliverable -->
<!-- ID    - Identity -->
<!-- Lab   - Laboratory -->
<!-- QC    - Quality Control -->
<!-- RPD   - Relative Percent Difference -->
<!ELEMENT Header (
               ClientID|
               ClientName|
               Comment|
               DateFormat|
               EDDID|
               EDDImplementationID|
               EDDImplementationVersion|
               EDDVersion|
               GeneratingSystemID|
               GeneratingSystemVersion|
               LabContract|
               LabContractModificationDescription|
               LabContractModificationID|
               LabDataPackageID|
               LabDataPackageName|
               LabDataPackageVersion|
               LabID|
               LabName|
               LabNarrative|
               LabQualifiersDefinition|
               LabReportedDate|
               ProjectID|
               ProjectName|
               SiteID|
               SiteName|
               ContactInformation|
               SamplePlusMethod
                        )*>
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               AliquotAmountUnits|
               AnalysisDuration|
               AnalysisDurationUnits|
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               AnalysisType|
               Analyst|
               AnalyzedAmount|
               AnalyzedAmountUnits|
               AnalyzedDate|
               ClientAnalysisID|
               ClientMethodCode|
               ClientMethodID|
               ClientMethodModificationDescription|
               ClientMethodModificationID|
               ClientMethodName|
               ClientMethodSource|
               ClientMethodVersion|
               Column|
```

```
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                ColumnLength|
                ColumnLengthUnits|
                Comment|
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                CountsUncertainty|
                CountsUncertaintyConfidenceLevel|
                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
                CountsUncertaintyLimitHigh|
                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
                DetectorID|
                DetectorType|
                DilutionFactor|
                Efficiency|
                HeatedPurge|
                Inclusion|
                InjectionVolume|
                InjectionVolumeUnits|
                InstrumentID|
                LabAnalysisID|
                LabFileID|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                ProcedureID|
                ProcedureName|
                ReferenceDate|
                ResultBasis|
                Temperature|
                TemperatureUnits|
                Wavelength|
                WavelengthUnits|
                Yield|
                PreparationPlusCleanup|
                Analyte|
                AnalyteGroup
                          )*>
<!ELEMENT AnalysisGroup (
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                Comment|
                Analyte|
                AnalyteGroup
                          )*>
<!ELEMENT Analyte (
```

```
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               AnalyteName|
               AnalyteNameContext|
               AnalyteType|
               CASRegistryNumber|
               ClientAnalyteID|
               ClientAnalyteName|
               Comment|
               Counts|
               CountsUncertainty|
               CountsUncertaintyConfidenceLevel|
               CountsUncertaintyDetermination|
               CountsUncertaintyIntervalType|
               CountsUncertaintyLimitHigh|
               CountsUncertaintyLimitLow|
               CountsUncertaintyType|
               CountsUnits|
               DetectionLimit|
               DetectionLimitType|
               DetectionLimitUnits|
               DifferenceErrorRatio|
               Efficiency|
               ExpectedResult|
               ExpectedResultUncertainty|
               ExpectedResultUncertaintyConfidenceLevel|
               ExpectedResultUncertaintyDetermination|
               ExpectedResultUncertaintyIntervalType|
               ExpectedResultUncertaintyLimitHigh|
               ExpectedResultUncertaintyLimitLow|
               ExpectedResultUncertaintyType|
               ExpectedResultUncertaintyUnits|
               ExpectedResultUnits|
               Inclusion|
               LabAnalyteID|
               LabQualifiers|
               LotNumber|
               PeakID|
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               PercentRecoveryLimitHigh|
               PercentRecoveryLimitLow|
               PercentRecoveryLimitType|
               PercentRecoveryType|
               QuantitationLimit|
               QuantitationLimitType|
               QuantitationLimitUnits|
               ReportingLimit|
               ReportingLimitType|
               ReportingLimitUnits|
               Result|
               ResultLimitHigh|
               ResultLimitLow|
               ResultLimitType|
               ResultType|
               ResultUncertainty|
               ResultUncertaintyConfidenceLevel|
               ResultUncertaintyDetermination|
               ResultUncertaintyIntervalType|
               ResultUncertaintyLimitHigh|
               ResultUncertaintyLimitLow|
```

```
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                ResultUnits|
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                Wavelength|
                WavelengthUnits
                          )*>
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                ClientAnalyteName|
                Comment|
                LabAnalyteID|
                LabQualifiers|
                Result|
                ResultType|
                ResultUncertainty|
                ResultUnits
                          )*>
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                LabCountry|
                LabID|
                LabName|
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                LabPointOfContactType|
                LabState|
                LabTelephoneNumber|
                LabType|
                LabZipCode
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                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                HandledDate|
                HandlingBatch|
                HandlingType|
                InitialAmount|
```

```
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               LabID|
               LabMethodID|
               LabMethodName|
               LabName|
               MethodCode|
               MethodID|
               MethodModificationDescription|
               MethodModificationID|
               MethodName|
               MethodSource|
               MethodVersion|
               ProcedureID|
               ProcedureName|
               SampleAmount|
               SampleAmountUnits|
               Characteristic
                         )*>
<!ELEMENT PreparationPlusCleanup (
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               AliquotAmountUnits|
               Analyst|
               CleanedUpDate|
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               CleanupType|
               ClientMethodCode|
               ClientMethodID|
               ClientMethodModificationDescription|
               ClientMethodModificationID|
               ClientMethodName|
               ClientMethodSource|
               ClientMethodVersion|
               Comment|
               FinalAmount|
               FinalAmountUnits|
               InitialAmount|
               InitialAmountUnits|
               LabID|
               LabMethodID|
               LabMethodName|
               LabName|
               LotNumber|
               MethodCode|
               MethodID|
               MethodModificationDescription|
               MethodModificationID|
               MethodName|
               MethodSource|
               MethodVersion|
               PreparationBatch|
               PreparationPlusCleanupType|
               PreparationType|
               PreparedDate|
               ProcedureID|
               ProcedureName|
               Solvent|
               Characteristic
                         )*>
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```

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AnalyteGroupID|
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AnalyteNameContext|
AnalyteType|
BiasErrorRatio|
CASRegistryNumber|
ClientAnalyteID|
ClientAnalyteName|
ClientDetectionLimit|
ClientDetectionLimitUnits|
ClientQuantitationLimit|
ClientQuantitationLimitUnits|
Comment|
DetectionLimit|
DetectionLimitType|
DetectionLimitUnits|
DifferenceErrorRatio|
ExpectedResult|
ExpectedResultUncertainty|
ExpectedResultUncertaintyConfidenceLevel|
ExpectedResultUncertaintyDetermination|
ExpectedResultUncertaintyIntervalType|
ExpectedResultUncertaintyLimitHigh|
ExpectedResultUncertaintyLimitLow|
ExpectedResultUncertaintyType|
ExpectedResultUncertaintyUnits|
ExpectedResultUnits|
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LabAnalyteID|
LabQualifiers|
LabResultStatus|
PeakID|
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PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentRecovery|
PercentRecoveryLimitHigh|
PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
QuantitationLimit|
QuantitationLimitType|
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ResultUncertaintyConfidenceLevel|
ResultUncertaintyDetermination|
ResultUncertaintyIntervalType|
ResultUncertaintyLimitHigh|
ResultUncertaintyLimitLow|
```

```
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               RPDLimitType|
               RPDType
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               ClientMethodID|
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               ClientMethodModificationID|
               ClientMethodName|
               ClientMethodSource|
               ClientMethodType|
               ClientMethodVersion|
               ClientName|
               ClientSampleID|
               CollectedDate|
               CollectedEndDate|
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               Composite|
               CoolerID|
               CustodyID|
               EquipmentBatch|
               Filtered|
               LabContract|
               LabContractModificationDescription|
               LabContractModificationID|
               LabID|
               LabMethodID|
               LabMethodName|
               LabName|
               LabReceiptDate|
               LabReportingBatch|
               LabSampleID|
               LocationID|
               LocationName|
               MatrixID|
               MatrixMedium|
               MethodBatch|
               MethodCategory|
               MethodCode|
               MethodID|
               MethodLevel|
               MethodModificationDescription|
               MethodModificationID|
               MethodName|
               MethodSource|
               MethodType|
               MethodVersion|
               OriginalClientSampleID|
               OriginalLabSampleID|
               PhaseAnalyzed|
```

```
                Preservative|
                ProjectID|
                ProjectName|
                QCCategory|
                QCLinkage|
                QCType|
                Quarantine|
                SamplingBatch|
                ShippingBatch|
                SiteID|
                SiteName|
                StorageBatch|
                Analysis|
                Characteristic|
                ReportedResult|
                Handling|
                AnalysisGroup
                           )*>
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<!ELEMENT ResultUncertaintyIntervalType (#PCDATA)>
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<!ELEMENT WavelengthUnits (#PCDATA)>
<!ELEMENT Yield (#PCDATA)>
```

6.0 DATA ELEMENT INSTRUCTION TABLES

6.1 Stage 2b

Table 1

Inorganics Data Element Instructions

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
Header	X	X	X	X	X	X	X	X	
ClientID	X	X	X	X	X	X	X	X	Report “1” for Region 1, “2” for Region 2, etc. For samples received from QATS, report “91”.
ClientName									Not required.
Comment									Not Required.
DateFormat	X	X	X	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report “00” for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	X	X	X	Report “SEDD”.
EDDImplementationID	X	X	X	X	X	X	X	X	Report “SEDD_5-2_GENERAL_2b” (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	X	X	X	Report “3” (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	X	X	X	Report “5.2”.
GeneratingSystemID	X	X	X	X	X	X	X	X	Report name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	X	X	X	Report software version number.
LabContract	X	X	X	X	X	X	X	X	Report the Contract number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabDataPackageID	X	X	X	X	X	X	X	X	Report the Sample Delivery Group (SDG).
LabDataPackageName	X	X	X	X	X	X	X	X	Report “ICP_AES”, “ICP_MS”, “Hg”, or “CN” as applicable.
LabDataPackageVersion	X	X	X	X	X	X	X	X	Report “1”, then increment with each resubmission.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabNarrative									Not required.
LabQualifiersDefinition	X	X	X	X	X	X	X	X	Use the format ‘Qualifier:Definition’ to report each qualifier used. Use a ‘;’ to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	X	X	X	Report the Case Number.
ProjectName									Not required.
SiteID									Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
SiteName									Not required.
SamplePlusMethod	X	X	X	X	X	X	X	X	
ClientID	X	X	X						Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM01.2".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID	X	X	X	X	X	X	X		Report the Modified Analysis number, if applicable
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "USEPA_CLP".
ClientMethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
ClientMethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
ClientName									Not required.
ClientSampleID	X	X	X	X	X	X	X	X	Report the EPA Sample Number.
CollectedDate	X	X	X						Report the date and time the sample was collected.
CollectedEndDate									Not required.
Comment									Not required.
Composite									Not required.
CoolerID									Not required.
CustodyID	X								Report the Traffic Report/Chain of Custody Form number.
EquipmentBatch									Not required.
Filtered	X								Report "Yes" for dissolved metals, or "No" for total metals.
LabContract	X	X	X	X	X	X	X		Report the Contract number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID									Not required.
LabMethodName									Not required.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X	X						Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG number.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
LabSampleID	X	X	X	X	X	X	X	X	Report the Lab Sample ID as assigned by the lab.
LocationID									Not required.
LocationName									Not required.
MatrixID	X	X	X	X	X	X	X	X	Report "Water", "Soil", "Wipe", or "Filter" as applicable.
MatrixMedium	X	X	X	X	X	X	X	X	Report "Aqueous" or "Solid" as applicable. Use "Solid" for wipes and filters.
MethodBatch									Not required.
MethodCategory									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM01.2".
MethodLevel	X	X	X			X	X		Report "Low" or "Medium".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "USEPA_CLP".
MethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
MethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
OriginalClientSampleID		X	X			X	X		Report the EPA Sample Number of the original sample this sample was derived from.
OriginalLabSampleID									Not required.
PhaseAnalyzed									Not required.
Preservative	X	X	X						Report any chemical or physical preservative used.
ProjectID	X	X	X	X	X	X	X		Report the Case Number.
ProjectName									Not required.
QCCategory		X	X	X	X	X	X		Report "Blank" for PB, "Spike" for MS and post-digestion spike, "Blank_Spike" for LCS, "Duplicate" for duplicate, or "Serial_Dilution" for SD.
QCLinkage		X	X	X	X	X	X		Report "LabReportingBatch" for MS, post-digestion spike, Dup, and SD; or "PreparationBatch" for PB and LCS.
QCType	X	X	X	X	X	X	X		Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "PT_Sample" for Performance Evaluation Samples; "Method_Blank" for PB; "Matrix_Spike" for MS; "Duplicate" for dup; "Laboratory_Control_Sample" for LCS; "Post_Digestion_Spike" for post-digestion spikes; or "Serial_Dilution" for SD.
Quarantine	X								Report "Yes" or "No" based on sampling information.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
SamplingBatch									Not required.
ShippingBatch									Not required.
SiteID									Not required.
SiteName									Not required.
StorageBatch									Not required.
InstrumentQC									Not required.
Characteristic	X	X	X	X	X	X	X		
CharacteristicType	X	X	X	X	X	X	X		Report "Artifacts"; "Clarity"; "Color"; "Percent_Solids"; "pH"; "Temperature"; and "Texture" for each SamplePlusMethod. Report "Clarity" and "Color" for each PreparationPlusCleanup.
CharacteristicValue	X	X	X	X	X	X	X		Report any sample artifacts for "Artifact"; the clarity (at receipt and after preparation)for aqueous/water samples for "Clarity"; the color (at receipt and after preparation) for "Color"; the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples for "Percent_Solids"; the pH (at receipt) to the nearest whole unit for aqueous/water samples for "pH"; the temperature at receipt to the nearest degree for "Temperature"; and the texture for soil/sediment samples for "Texture".
CharacteristicUnits	X	X	X	X	X	X	X		Report "C" for "Temperature".
Comment									Not required.
ContactInformation	X	X	X	X	X	X	X	X	
LabAddress1	X	X	X	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType									Not required.
LabState	X	X	X	X	X	X	X	X	Report the state or province in which the laboratory is located.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
LabTelephoneNumber	X	X	X	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType									Not required.
LabZipCode	X	X	X	X	X	X	X	X	Report the Zip or postal code.
Analysis	X	X	X	X	X	X	X	X	
AliquotAmount									Not required.
AliquotAmountUnits									Not required.
AnalysisBatch	X	X	X	X	X	X	X	X	Links this analysis to the instrument QC sample(s) that begins this sequence. Report the Lab Analysis ID of the CCV that starts the sequence.
AnalysisBatchEnd	X	X	X	X	X	X	X	X	Links this analysis to the instrument QC sample(s) that ends this sequence. Report the Lab Analysis ID of the CCV that ends this sequence.
AnalysisDuration									Not required.
AnalysisDurationUnits									Not required.
AnalysisGroupID									Not required.
AnalysisType	X	X	X	X	X	X	X		Report "Initial", "Dilution-01", or "Reanalysis-01"; then increment as necessary.
Analyst	X	X	X	X	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount									Not required.
AnalyzedAmountUnits									Not required.
AnalyzedDate	X	X	X	X	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM01.2".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "USEPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
Column									Not required.
ColumnInternalDiameter									Not required.
ColumnInternalDiameterUnits									Not required.
ColumnLength									Not required.
ColumnLengthUnits									Not required.
Comment									Not required.
ConfirmationAnalysisID									Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectorID									Not required.
DetectorType									Not required.
DilutionFactor	X	X	X	X	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency									Not required.
HeatedPurge									Not required.
Inclusion									Not required.
InjectionVolume									Not required.
InjectionVolumeUnits									Not required.
InstrumentID	X	X	X	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	X	X	X	X	Report a unique identifier.
LabFileID	X	X	X	X	X	X	X	X	Report the lab file ID.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM01.2".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "USEPA_CLP".
MethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
PreparationBatch									Not required.
ProcedureID									Not required.
ProcedureName									Not required.
ReferenceDate									Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
ResultBasis	X	X	X		X				Report "Dry" for soil/sediment samples. For water/aqueous sample, report "Dissolved" if sample field-filtered; otherwise report "Total".
RunBatch	X	X	X	X	X	X	X	X	Links this analysis to an initial calibration. Report the Lab Analysis ID of the standard (Tune or calibration standard) that started the ICAL sequence.
Temperature									Not required.
TemperatureUnits									Not required.
WaveLength									Not required.
WaveLengthUnits									Not required.
Yield									Not required.
AnalysisGroup									Not required.
Handling									Not required.
ReportedResult	X	X	X	X	X	X	X		
AnalysisGroupID									Not required.
AnalyteGroupID									Not required.
AnalyteName	X	X	X	X	X	X	X		Report analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target compounds, "Spike" for all target analytes designated as spike compounds for Matrix Spike analysis.
BiasErrorRatio									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report CAS Numbers as they appear in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report analytes as they appear in the SOW.
ClientDetectionLimit									Not required.
ClientDetectionLimitUnits									Not required.
ClientQuantitationLimit	X	X	X	X	X	X	X		Report the CRQL.
ClientQuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
Comment									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the current Method Detection Limit (MDL), adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL_sa".

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
DifferenceErrorRatio									Not required.
ExpectedResult		X		X		X			Report the theoretical final calculated concentration (the spike added) for the spiked analytes or the true value for LCS.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits		X		X		X			Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
LabAnalysisID	X	X	X	X	X	X	X		Report the unique identifier from the analysis this reported result was derived from.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report flags as specified in the SOW. Includes both the C and Q qualifiers from Form I.
LabResultStatus	X	X	X						Report "Preliminary" or "Final" as applicable.
PeakID									Not required.
PercentDifference							X		Report the Percent Difference.
PercentDifferenceLimitHigh							X		Report the upper limit for the Percent Difference.
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType							X		Report "Method".
PercentRecovery		X		X		X			Report the Percent Recovery.
PercentRecoveryLimitHigh		X		X					Report the upper limit for the Percent Recovery.
PercentRecoveryLimitLow		X		X					Report the lower limit for the Percent Recovery.
PercentRecoveryLimitType		X		X					Report "Method".
PercentRecoveryType									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the Contract Required Quantitation Limit adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Result	X	X	X	X	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
RetentionTime									Not required.
RetentionTimeUnits									Not required.
RPD			X						Report the RPD.
RPDLimitHigh			X						Report the upper limit for the RPD.
RPDLimitType			X						Report "Method".
RPDType									Not required.
PreparationPlusCleanup	X	X	X	X	X	X	X		
AliquotAmount	X	X	X	X	X	X	X		Report the sample amount in grams for soil/sediment or mL for aqueous/water to at least three significant figures. Not required for wipes or filters.
AliquotAmountUnits	X	X	X	X	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water. Not required for wipes or filters.
Analyst	X	X	X	X	X	X	X		Report the Analyst's initials.
CleanedUpDate									Not required.
CleanUpBatch									Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
CleanUpType									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X		Report the sample preparation ID as given in Exhibit B.
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X		Report "USEPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X		Report month and year the SOW was issued.
Comment									Not required.
FinalAmount	X	X	X	X	X	X	X		Report the volume of digestate produced by the preparation method in mL.
FinalAmountUnits	X	X	X	X	X	X	X		Report "mL".
InitialAmount									Not required.
InitialAmountUnits									Not required.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
LotNumber									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X		Report "ISM01.2".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X		Report "USEPA_CLP".
MethodVersion	X	X	X	X	X	X	X		Report month and year the SOW was issued.
PreparationBatch	X	X	X	X	X	X	X		Links all samples that were prepared together. Report a unique identifier for each batch.
PreparationPlusCleanupType	X	X	X	X	X	X	X		Report "Preparation".
PreparationType	X	X	X	X	X	X	X		Report "Automated" or "Manual".
PreparedDate	X	X	X	X	X	X	X		Report the date and time the sample was prepared.
ProcedureID									Not required.
ProcedureName									Not required.
Solvent									Not required.
Analyte	X	X	X	X	X	X	X		
AnalyteGroupID									Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
AnalyteName	X	X	X	X	X	X	X		Report analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target compounds, "Spike" for all target analytes designated as spike analytes, "Internal_Standard" for internal standards, or "Monitor" for non-target interferences and masses requiring monitoring.
BiasErrorRatio									Not required.
CalibrationBasis									Not required.
CalibrationFactor									Not required.
CalibrationFactorUnits									Not required.
CalibrationType									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
Coeffa0									Not required.
Coeffa1									Not required.
Coeffa2									Not required.
Coeffa3									Not required.
CoeffOfDetermination									Not required.
CoeffOfDeterminationLimitLow									Not required.
CoeffOfDeterminationLimitType									Not required.
Comment									Not required.
CorrelationCoeff									Not required.
CorrelationCoeffLimitLow									Not required.
CorrelationCoeffLimitType									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the Method Detection Limit (MDL).
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL".

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
DifferenceErrorRatio									Not required.
Efficiency									Not required.
ExpectedResult									Not required.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits									Not required.
Inclusion									Not required.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X	X	X	X		Report the vendor/manufacturer assigned lot number for this standard (Internal Standards and spiking compounds only).
Mass									Not required.
MassUnits									Not required.
MeanCalibrationFactor									Not required.
MeanCalibrationFactorUnits									Not required.
MeanRRF									Not required.
MeanRRFLimitLow									Not required.
MeanRRFLimitType									Not required.
PeakID	X	X	X	X	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown									Not required.
PercentBreakdownLimitHigh									Not required.
PercentBreakdownLimitType									Not required.
PercentDifference									Not required.
PercentDifferenceLimitHigh									Not required.
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType									Not required.
PercentRecovery									Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
PercentRecoveryLimitHigh									Not required.
PercentRecoveryLimitLow									Not required.
PercentRecoveryLimitType									Not required.
PercentRecoveryType									Not required.
PercentRSD									Not required.
PercentRSDLimitHigh									Not required.
PercentRSDLimitLow									Not required.
PercentRSDLimitType									Not required.
QuantitationBasis									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the Contract Required Quantitation Limit (CRQL).
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Result	X	X	X	X	X	X	X		For target and spike analyte detects, and for monitored masses, report the final calculated result.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintylimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, and "ug" for wipe and filter samples.
RPD									Not required.
RPDLimitHigh									Not required.
RPDLimitType									Not required.
RPDType									Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
RRF									Not required.
RRFLimitLow									Not required.
RRFLimitType									Not required.
StandardSource	X	X	X	X	X	X	X		Report the vendor/manufacturer for this standard.
TailingFactor									Not required.
TailingFactorLimitHigh									Not required.
TailingFactorLimitType									Not required.
Wavelength									Not required.
WavelengthUnits									Not required.
WeightingFactor									Not required.
AnalyteGroup									Not required.
Peak									Not required.
PeakComparison									Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
Header	X	X	X	X	X	
ClientID	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2 etc. For samples received from QATS, report "91".
ClientName						Not required.
Comment						Not required.
DateFormat	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	Report "SEDD_5-2_GENERAL_2b" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	Report name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	Report software version number.
LabContract	X	X	X	X	X	Report the Contract number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabDataPackageID	X	X	X	X	X	Report the Sample Delivery Group (SDG).
LabDataPackageName	X	X	X	X	X	Report "ICP_AES", "ICP_MS", "Hg", or "CN" as applicable.
LabDataPackageVersion	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabNarrative						Not required.
LabQualifiersDefinition	X	X	X	X	X	Use the format 'Qualifer:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	Report the Case Number.
ProjectName						Not required.
SiteID						Not required.
SiteName						Not required.
SamplePlusMethod						Not required.
InstrumentQC	X	X	X	X	X	
ClientInstrumentQCType						Not required.
ClientMethodCode						Not required.
ClientMethodID	X	X	X	X	X	Report "ISM01.2".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "USEPA_CLP".
ClientMethodVersion	X	X	X	X	X	Report month and year the SOW was issued.
Comment						Not required.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabInstrumentQCID	X	X	X	X	X	Report a unique ID for each QC.
LabMethodID						Not required.
LabMethodName						Not required.
LabName	X	X	X	X	X	Report the Lab Name.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "ISM01.2".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "USEPA_CLP".
MethodVersion	X	X	X	X	X	Report month and year the SOW was issued.
QCLinkage	X	X	X	X	X	Report "RunBatch" for IPC, calibration, ICV, ICB, and ICS. Report "AnalysisBatch" for CCV and CCB.
QCType	X	X	X	X	X	Report "Instrument_Performance_Check_Tune" for Tune; "Initial_Calibration" for calibration; "Initial_Calibration_Verification" for ICV; "Initial_Calibration_Blank" for ICB; "Continuing_Calibration_Verification" for CCV; "Continuing_Calibration_Blank" for CCB; "Interference_Check_Standard_A" for ICSA;or "Interference_Check_Standard_A/B" for ICSAB.
ContactInformation	X	X	X	X	X	
LabAddress1	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	Report the name of person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType						Not required.
LabState	X	X	X	X	X	Report the state or province in which the laboratory is located.

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Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
LabTelephoneNumber	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType						Not required.
LabZipCode	X	X	X	X	X	Report the Zip or postal code.
Analysis	X	X	X	X	X	
AliquotAmount						Not required.
AliquotAmountUnits						Not required.
AnalysisBatch			X	X		Links this analysis to the instrument QC sample that begins this sequence. Report the Lab Analysis ID of the CCV that starts the sequence.
AnalysisBatchEnd			X	X		Links this analysis to the instrument QC sample that ends this sequence. Report the Lab Analysis ID of the CCV that ends this sequence.
AnalysisDuration						Not required.
AnalysisDurationUnits						Not required.
AnalysisGroupID		X				Links a group of analyses together that are used for the initial calibration. Report the Lab Analysis ID of the standard that starts this calibration sequence.
AnalysisType	X	X	X	X	X	Report "Initial", "Dilution-01", or "Reanalysis-01"; then increment as necessary.
Analyst	X	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount						Not required.
AnalyzedAmountUnits						Not required.
AnalyzedDate	X	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID						Not required.
ClientMethodCode						Not required.
ClientMethodID	X	X	X	X	X	Report "ISM01.2".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "USEPA_CLP".
ClientMethodVersion	X	X	X	X	X	Report month and year the SOW was issued.
Column						Not required.
ColumnInternalDiameter						Not required.
ColumnInternalDiameterUnits						Not required.
ColumnLength						Not required.
ColumnLengthUnits						Not required.
Comment						Not required.
ConfirmationAnalysisID						Not required.
Counts						Not required.
CountsUncertainty						Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectorID						Not required.
DetectorType						Not required.
DilutionFactor	X	X	X	X	X	Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency						Not required.
HeatedPurge						Not required.
Inclusion						Not required.
InjectionVolume						Not required.
InjectionVolumeUnits						Not required.
InstrumentID	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	X	Report a unique identifier.
LabFileID	X	X	X	X	X	Report the lab file ID.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "ISM01.2".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "USEPA_CLP".
MethodVersion	X	X	X	X	X	Report month and year the SOW was issued.
PreparationBatch						Not required.
ProcedureID						Not required.
ProcedureName						Not required.
ReferenceDate						Not required.
ResultBasis						Not required.
RunBatch	X	X	X	X	X	Links this analysis to an initial calibration. Report the Lab Analysis ID of the standard (Tune or calibration standard) that started the ICAL sequence.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
Temperature						Not required.
TemperatureUnits						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
Yield						Not required.
AnalysisGroup		X				
AnalysisGroupID		X				This links a group of analyses together that are used for the initial calibration. Report the lab analysis ID of the standard that starts this calibration sequence.
AnalysisType		X				Report "Initial_Calibration".
Comment						Not required.
Handling						Not required.
ReportedResult						Not required.
PreparationPlusCleanup		X	X	X		
AliquotAmount		X	X	X		Report the actual amount of standard digested/distilled in mL to at least three significant figures.
AliquotAmountUnits		X	X	X		Report "mL".
Analyst		X	X	X		Report the Analyst's initials.
CleanedUpDate						Not required.
CleanupBatch						Not required.
CleanupType						Not required.
ClientMethodCode						Not required.
ClientMethodID		X	X	X		Enter the sample preparation ID as described in Exhibit B.
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource		X	X	X		Report "USEPA_CLP".
ClientMethodVersion		X	X	X		Report month and year the SOW was issued.
Comment						Not required.
FinalAmount		X	X	X		Report the volume of digestate produced by the preparation method in mL.
FinalAmountUnits		X	X	X		Report "mL".
InitialAmount						Not required.
InitialAmountUnits						Not required.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
LabName						Not required.
LotNumber						Not required.
MethodCode						Not required.
MethodID		X	X	X		Report "ISM01.2".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource		X	X	X		Report "USEPA_CLP".
MethodVersion		X	X	X		Report month and year the SOW was issued.
PreparationBatch		X	X	X		Links all samples that were prepared together. Report a unique identifier for each batch.
PreparationPlusCleanupType		X	X	X		Report "Preparation".
PreparationType		X	X	X		Report "Automated" or "Manual".
PreparedDate		X	X	X		Report the date and time the sample was prepared.
ProcedureID						Not required.
ProcedureName						Not required.
Solvent						Not required.
Characteristic						Not required.
Analyte	X	X	X	X	X	
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X	X	Report analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	Report "CAS".
AnalyteType	X	X	X	X	X	Report "Target" for all target compounds, "Spike" for all target analytes designated as spike compounds for MS analysis, "Internal_Standard" for internal standards, "Monitor" for non-target interferences and masses requiring monitoring, or "Instrument_Performance" for tune analytes.
BiasErrorRatio						Not required.
CalibrationBasis		X				Report "Peak" under the AnalysisGroup node.
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType						Not required.
CASRegistryNumber	X	X	X	X	X	Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	Report CAS number.
ClientAnalyteName	X	X	X	X	X	Report the analytes as they appear in the SOW.
Coeffa0						Not required.
Coeffa1						Not required.
Coeffa2						Not required.
Coeffa3						Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDtereminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff						Not required.
CorrelationCoeffLimitLow						Not required.
CorrelationCoeffLimitType						Not required.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectionLimit		X	X	X	X	Report the current Method Detection Limit from the default aqueous preparation method or other appropriate method.
DetectionLimitType		X	X	X	X	Report "MDL".
DetectionLimitUnits		X	X	X	X	Report "ug/L".
DifferenceErrorRatio						Not required.
Efficiency						Not required.
ExpectedResult		X	X		X	Report the concentration of the standard in ug/L.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDeterminatio n						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.
ExpectedResultUnits		X	X		X	Report "ug/L".
Inclusion		X				Report "No" if an analyte in a standard is not to be included in the calibration curve; otherwise report "Yes".
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X	X	Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X	X	Report the vendor/manufacturer assigned lot number for this standard.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
Mass						Not required.
MassUnits						Not required.
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID			X	X	X	If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown						Not required.
PercentBreakdownLimitHigh						Not required.
PercentBreakdownLimitType						Not required.
PercentDifference		X				Report the percent difference.
PercentDifferenceLimitHigh		X				Report the upper limit for the percent difference.
PercentDifferenceLimitLow		X				Report the lower limit for the percent difference.
PercentDifferenceLimitType		X				Report "Method".
PercentRecovery			X		X	Report the percent recovery. Not required for ICS when true value equals 0.
PercentRecoveryLimitHigh			X		X	Report the upper limit for the percent recovery. Not required when ResultLimitHigh applies.
PercentRecoveryLimitLow			X		X	Report the lower limit for the percent recovery. Not required when ResultLimitLow applies.
PercentRecoveryLimitType			X		X	Report "Method".
PercentRecoveryType						Not required.
PercentRSD						Not required.
PercentRSDLimitHigh						Not required.
PercentRSDLimitLow						Not required.
PercentRSDLimitType						Not required.
QuantitationBasis		X				Report "External_Standard" under the AnalysisGroup node.
QuantitationLimit		X	X	X	X	Report the Contract Required Quantitation Limit.
QuantitationLimitType		X	X	X	X	Report "CRQL".
QuantitationLimitUnits		X	X	X	X	Report "ug/L".
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Result		X	X	X	X	For detected target and spike analytes, and for monitored masses, report the final calculated result (in ug/L).
ResultLimitHigh					X	For analytes and interferents with true values less than 5x (10x for ICP-MS) CRQL.
ResultLimitLow					X	For analytes and interferents with true values less than 5x (10x for ICP-MS) CRQL.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
ResultLimitType					X	Report "Method".
ResultType		X	X	X	X	Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.
ResultUnits		X	X	X	X	Report "ug/L".
RPD						Not required.
RPDLimitHigh						Not required.
RPDLimitType						Not required.
RPDType						Not required.
RRF						Not required.
RRFLimitLow						Not required.
RRFLimitType						Not required.
StandardSource	X	X	X	X	X	Report the vendor/manufacturer for this standard.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
WeightingFactor						Not required.
AnalyteGroup						Not required.
Peak	X	X	X	X	X	
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType		X				Report "Linear_Regression"; "Linear_Regression_With_Blank_Force"; "Weighted_Linear_Regression"; or "Weighted_Linear_Regression_With_Blank_Force" under the AnalysisGroup node.
Coeffa0		X				Report the y-intercept of the calibration curve under the AnalysisGroup node.
Coeffa1		X				Report the slope of the calibration curve under the AnalysisGroup node.

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Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
Coeffa2						Not required.
Coeffa3						Not required.
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDeterminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff		X				Report the correlation coefficient (r) of the calibration curve to at least 4 significant figures under the AnalysisGroup node.
CorrelationCoeffLimitLow		X				Report the lower limit for the correlation coefficient to at least 4 significant figures under the AnalysisGroup node.
CorrelationCoeffLimitType		X				Report "Method" under the AnalysisGroup node.
DifferenceErrorRatio						Not required.
Efficiency						Not required.
Inclusion		X				Report "No" if a peak in a standard is not to be included in the calibration curve; otherwise report "Yes".
LabQualifiers						Not required.
Mass	X					Report the Average Measured Mass.
MassLimitHigh	X					Report the upper limit for the mass.
MassLimitLow	X					Report the lower limit for the mass.
MassLimitType	X					Report "method".
MassUnits	X					Report "u".
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRetentionTime						Not required.
MeanRetentionTimeLimitHigh						Not required.
MeanRetentionTimeLimitLow						Not required.
MeanReterntionTimeLimitType						Not required.
MeanRetentionTimeUnits						Not required.
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID	X	X	X	X	X	Report a unique identifier.
PercentDifference						Not required.
PercentDifferenceLimitHigh						Not required.
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType						Not required.
PercentRecovery						Not required.
PercentRecoveryLimitHigh						Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability IPC	Cal	ICV CCV	ICB CCB	ICS	Instructions
PercentRecoveryLimitLow						Not required.
PercentRecoveryLimitType						Not required.
PercentRecoveryType						Not required.
PercentRSD	X	X	X	X	X	For ICP, report the %RSD of the replicates.
PercentRSDLimitHigh	X	X	X	X	X	Report the upper limit for the %RSD.
PercentRSDLimitLow						Not required.
PercentRSDLimitType	X	X	X	X	X	Report "Method".
Resolution	X					Report the Average Peak Width.
ResolutionLimitHigh	X					Report the upper limit from the manufacturer specifications.
ResolutionLimitLow	X					Report the lower limit from the manufacturer specifications.
ResolutionLimitType	X					Report "Laboratory".
ResolutionType						Not required.
ResolutionUnits	X					Report "u".
Result						Not required.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType						Not required.
ResultUncertainty						Not required.
ResultUnits						Not required.
RRF						Not required.
RRFLimitLow						Not required.
RRFLimitType						Not required.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength	X	X	X	X	X	For ICP-AES, Hg, and CN, report the wavelength of the peak in nm.
WavelengthUnits	X	X	X	X	X	Report "nm".
WeightingFactor		X				Report "Inverse_Of_Concentration"; "Inverse_Square_Of_Concentration"; or "None" as applicable under the AnalysisGroup node.
PeakComparison		X	X	X	X	
Comment						Not required.
PeakID		X	X	X	X	Report the unique peak identifier of the associated internal standard.
PercentRatio						Not required.

Table 1

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability					Instructions
	IPC	Cal	ICV CCV	ICB CCB	ICS	
PercentRatioLimitHigh						Not required.
PercentRatioLimitLow						Not required.
PercentRatioLimitType						Not required.

6.2 Stage 2a

Table 2

Inorganics Data Element Instructions

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
Header	X	X	X	X	X	X	X	X	
ClientID	X	X	X	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName									Not required.
Comment									Not Required.
DateFormat	X	X	X	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	X	X	X	Report "SEDD_5-2_GENERAL_2a" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	X	X	X	Report "2" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	X	X	X	Report name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	X	X	X	Report software version number.
Lab Contract	X	X	X	X	X	X	X	X	Report the Contract number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabDataPackageID	X	X	X	X	X	X	X	X	Report the Sample Delivery Group (SDG).
LabDataPackageName	X	X	X	X	X	X	X	X	Report "ICP_AES", "ICP_MS", "Hg", or "CN" as applicable.
LabDataPackageVersion	X	X	X	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID									Report the Agency-assigned Lab Code.
Lab Name	X	X	X	X	X	X	X	X	Report the Lab Name.
LabNarrative									Not required.
LabQualifiersDefinition	X	X	X	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	X	X	X	Report the Case Number.
ProjectName									Not required.
SiteID									Not required.
SiteName									Not required.
SamplePlusMethod	X	X	X	X	X	X	X	X	
ClientID	X	X	X						Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory									Not required.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM01.2".
ClientMethodModificationDescription									Not Required.
ClientMethodModificationID	X	X	X	X	X	X	X		Report the Modified Analysis number, if applicable.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "USEPA_CLP".
ClientMethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
ClientMethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
ClientName									Not required.
ClientSampleID	X	X	X	X	X	X	X	X	Report the EPA Sample Number.
CollectedDate	X	X	X						Report the date and time the sample was collected.
CollectedEndDate									Not required.
Comment									Not required.
Composite									Not required.
CoolerID									Not required.
CustodyID	X								Report the Traffic Report/Chain of Custody Form number.
EquipmentBatch									Not required.
Filtered	X								Report "Yes" for dissolved metals, or "No" for total metals.
LabContract	X	X	X	X	X	X	X		Report the Contract number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID									Not required.
LabMethodName									Not required.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X	X						Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG number.
LabSampleID	X	X	X	X	X	X	X	X	Report the Lab Sample ID as assigned by the lab.
LocationID									Not required.
LocationName									Not required.
MatrixID	X	X	X	X	X	X	X	X	Report "Water", "Soil", "Wipe", or "Filter" as applicable.
MatrixMedium	X	X	X	X	X	X	X	X	Report "Aqueous" or "Solid" as applicable. Use "Solid" for wipes and filters.
MethodBatch									Not required.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
MethodCategory									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM01.2".
MethodLevel	X	X	X			X	X		Report "Low" or "Medium".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "USEPA_CLP".
MethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
MethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
OriginalClientSampleID		X	X			X	X		Report the EPA Sample Number of the original sample this sample was derived from.
OriginalLabSampleID									Not required.
PhaseAnalyzed									Not required.
Preservative	X	X	X						Report any chemical or physical preservative used.
ProjectID	X	X	X	X	X	X	X		Report the Case Number.
ProjectName									Not required.
QCCategory		X	X	X	X	X	X		Report "Blank" for PB; "Spike" for MS and post-digestion spike; "Blank_Spike" for LCS; "Duplicate" for duplicate; or "Serial_Dilution" for SD.
QCLinkage		X	X	X	X	X	X		Report "LabReportingBatch" for MS, post-digestion spike, Dup, and SD; or "PreparationBatch" for PB and LCS.
QCType	X	X	X	X	X	X	X		Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "PT_Sample" for Performance Evaluation Samples; "Method_Blank" for PB; "Matrix_Spike" for MS; "Duplicate" for dup; "Laboratory_Control_Sample" for LCS; "Post_Digestion_Spike" for post-digestion spikes; or "Serial_Dilution" for SD.
Quarantine	X								Report "Yes" or "No" based on sampling information.
SamplingBatch									Not required.
ShippingBatch									Not required.
SiteID									Not required.
SiteName									Not required.
StorageBatch									Not required.
Characteristic	X	X	X	X	X	X	X		
CharacteristicType									Report "Artifacts"; "Clarity"; "Color"; "Percent_Solids"; "pH"; "Temperature"; and "Texture" for each SamplePlusMethod. Report "Clarity" and "Color" for each PreparationPlusCleanup.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
CharacteristicValue									Report any sample artifacts for "Artifact"; the clarity (at receipt and after preparation)for aqueous/water samples for "Clarity"; the color (at receipt and after preparation) for "Color"; the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples for "Percent_Solids"; the pH (at receipt) to the nearest whole unit for aqueous/water samples for "pH"; the temperature at receipt to the nearest degree for "Temperature"; and the texture for soil/sediment samples for "Texture".
CharacteristicUnits									Report "C" for "Temperature".
Comment									Not required.
ContactInformation	X	X	X	X	X	X	X	X	
LabAddress1	X	X	X	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType									Not required.
LabState	X	X	X	X	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType									Not required.
LabZipCode	X	X	X	X	X	X	X	X	Report the Zip or postal code.
Analysis	X	X	X	X	X	X	X	X	
AliquotAmount									Not required.
AliquotAmountUnits									Not required.
AnalysisDuration									Not required.
AnalysisDurationUnits									Not required.
AnalysisGroupID									Not required.
AnalysisType	X	X	X	X	X	X	X		Report "Initial", "Dilution-01", or "Reanalysis-01"; then increment as necessary.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
Analyst	X	X	X	X	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount									Not required.
AnalyzedAmountUnits									Not required.
AnalyzedDate	X	X	X	X	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM01.2".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "USEPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
Column									Not required.
ColumnInternalDiameter									Not required.
ColumnInternalDiameterUnits									Not required.
ColumnLength									Not required.
ColumnLengthUnits									Not required.
Comment									Not required.
ConfirmationAnalysisID									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectorID									Not required.
DetectorType									Not required.
DilutionFactor	X	X	X	X	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency									Not required.
HeatedPurge									Not required.
Inclusion									Not required.
InjectionVolume									Not required.
InjectionVolumeUnits									Not required.
InstrumentID	X	X	X	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
LabAnalysisID	X	X	X	X	X	X	X	X	Report a unique identifier.
LabFileID	X	X	X	X	X	X	X	X	Report the lab file ID.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM01.2".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "USEPA_CLP".
MethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
PreparationBatch									Not required.
ProcedureID									Not required.
ProcedureName									Not required.
ReferenceDate									Not required.
ResultBasis	X	X	X		X				Report "Dry" for soil/sediment samples. For water/aqueous samples, report "Dissolved" if field-filtered; otherwise report "Total".
Temperature									Not required.
TemperatureUnits									Not required.
WaveLength									Not required.
WaveLengthUnits									Not required.
Yield									Not required.
AnalysisGroup									Not required.
Handling									Not required.
ReportedResult	X	X	X	X	X	X	X		
AnalysisGroupID									Not required.
AnalyteGroupID									Not required.
AnalyteName	X	X	X	X	X	X	X		Report analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target compounds or "Spike" for all target analytes designated as spike compounds for Matrix Spike analysis.
BiasErrorRatio									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report CAS Numbers as they appear in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
ClientAnalyteName	X	X	X	X	X	X	X		Report analytes as they appear in the SOW.
ClientDetectionLimit									Not required.
ClientDetectionLimitUnits									Not required.
ClientQuantitationLimit	X	X	X	X	X	X	X		Report the CRQL.
ClientQuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
Comment									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the current Method Detection Limit (MDL), adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
DifferenceErrorRatio									Not required.
ExpectedResult		X		X		X			Report the theoretical final calculated concentration (the spike added) for the spiked analytes or the true value for LCS.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits		X		X		X			Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
LabAnalysisID	X	X	X	X	X	X	X		Report the unique identifier from the analysis this reported result was derived from.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report flags as specified in the SOW. Includes the C and Q qualifiers from Form I.
LabResultStatus	X	X	X						Report "Preliminary" or "Final" as applicable.
PeakID									Not required.
PercentDifference							X		Report the Percent Difference.
PercentDifferenceLimitHigh							X		Report the upper limit for the Percent Difference.
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType							X		Report "Method".
PercentRecovery		X		X		X			Report the Percent Recovery.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
PercentRecoveryLimitHigh		X		X					Report the upper limit for the Percent Recovery.
PercentRecoveryLimitLow		X		X					Report the lower limit for the Percent Recovery.
PercentRecoveryLimitType		X		X					Report "Method".
PercentRecoveryType									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Result	X	X	X	X	X	X	X		Report the final calculated result for detects.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
RetentionTime									Not required.
RetentionTimeUnits									Not required.
RPD			X						Report the RPD to the nearest whole percent.
RPDLimitHigh			X						Report the upper limit for the RPD to the nearest whole percent.
RPDLimitType			X						Report "Method".
RPDType									Not required.
PreparationPlusCleanup	X	X	X	X	X	X	X		
AliquotAmount	X	X	X	X	X	X	X		Report the sample amount in grams for soil/sediment or mL for aqueous/water to at least three significant figures. Not required for wipes or filters.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
AliquotAmountUnits	X	X	X	X	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water. Not required for wipes or filters.
Analyst	X	X	X	X	X	X	X		Report the Analyst's initials.
CleanedUpDate									Not required.
CleanUpBatch									Not required.
CleanUpType									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X		Report the sample preparation ID as given in Exhibit B.
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X		Report "USEPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X		Report month and year the SOW was issued.
Comment									Not required.
FinalAmount	X	X	X	X	X	X	X		Report the volume of digestate produced by the preparation method in mL.
FinalAmountUnits	X	X	X	X	X	X	X		Report "mL".
InitialAmount									Not required.
InitialAmountUnits									Not required.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
LotNumber									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X		Report "ISM01.2".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X		Report "USEPA_CLP".
MethodVersion	X	X	X	X	X	X	X		Report month and year the SOW was issued.
PreparationBatch	X	X	X	X	X	X	X		Links all samples that were prepared together. Report a unique identifier for each batch.
PreparationPlusCleanupType	X	X	X	X	X	X	X		Report "Preparation".
PreparationType	X	X	X	X	X	X	X		Report "Automated" or "Manual".
PreparedDate	X	X	X	X	X	X	X		Report the date and time the sample was prepared.
ProcedureID									Not required.
ProcedureName									Not required.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability: Sample	MS	Dup	LCS	PB	PDS	SD	NCS	Instructions
Solvent									Not required.
Analyte	X	X	X	X	X	X	X		
AnalyteGroupID									Not required.
AnalyteName	X	X	X	X	X	X	X		Report analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target compounds, "Spike" for all target analytes designated as spike analytes, "Internal_Standard" for internal standards, or "Monitor" for non-target interferences and masses requiring monitoring.
BiasErrorRatio									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
Comment									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the Method Detection Limit (MDL).
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
DifferenceErrorRatio									Not required.
Efficiency									Not required.
ExpectedResult									Not required.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
ExpectedResultUnits									Not required.
Inclusion									Not required.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X	X	X	X		Report the vendor/manufacturer assigned lot number for this standard (Internal Standards and spiking compounds only).
PeakID	X	X	X	X	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak.
PercentRecovery									Not required.
PercentRecoveryLimitHigh									Not required.
PercentRecoveryLimitLow									Not required.
PercentRecoveryLimitType									Not required.
PercentRecoveryType									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Result	X	X	X	X	X	X	X		For target and spike analyte detects, and for monitored masses, report the final calculated result.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects.
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water, or "ug" for wipe and filter samples.
StandardSource	X	X	X	X	X	X	X		Report the vendor/manufacturer for this standard.

Table 2

Inorganics Data Element Instructions (Con't)

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB	PDS	SD	NCS	
Wavelength									Not required.
WavelengthUnits									Not required.
AnalyteGroup									Not required.

Table 3

Abbreviations Used in the Instructions

Abbreviation	Definition
%RSD	Percent Relative Standard Deviation
Cal.	(Initial) Calibration
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CRQL	Contract Required Quantitation Limit
DTD	Document Type Definition
Dup	Duplicate Sample
EDD	Electronic Data Deliverable
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICS	Interference Check Sample
ICSA	Interference Check Sample Solution A
ICSAB	Interference Check Sample Solution AB
ICV	Initial Calibration Verification
ID	Identifier
IPC	Instrument Performance Check (Tune)
Lab	Laboratory
LCS	Laboratory Control Sample
MDL	Method Detection Limit
MS	Matrix Spike
NCS	Non-Client (ZZZZZZ) Sample
PB	Preparation Blank
PDS	Post-Digestion/Distillation Spike
QATS	Quality Assurance Technical Support
QC	Quality Control
RPD	Relative Percent Difference
SD	Serial Dilution
SDG	Sample Delivery Group
SOW	Statement of Work

EXHIBIT D

INTRODUCTION TO ANALYTICAL METHODS

Exhibit D - Analytical Methods

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Part A - Analytical Methods for Inductively Coupled Plasma - Atomic Emission Spectroscopy

Part B - Analytical Methods for Inductively Coupled Plasma - Mass Spectrometry

Part C - Analytical Methods for Cold Vapor Mercury Analysis

Part D - Analytical Methods for Total Cyanide Analysis

1.0 INTRODUCTION

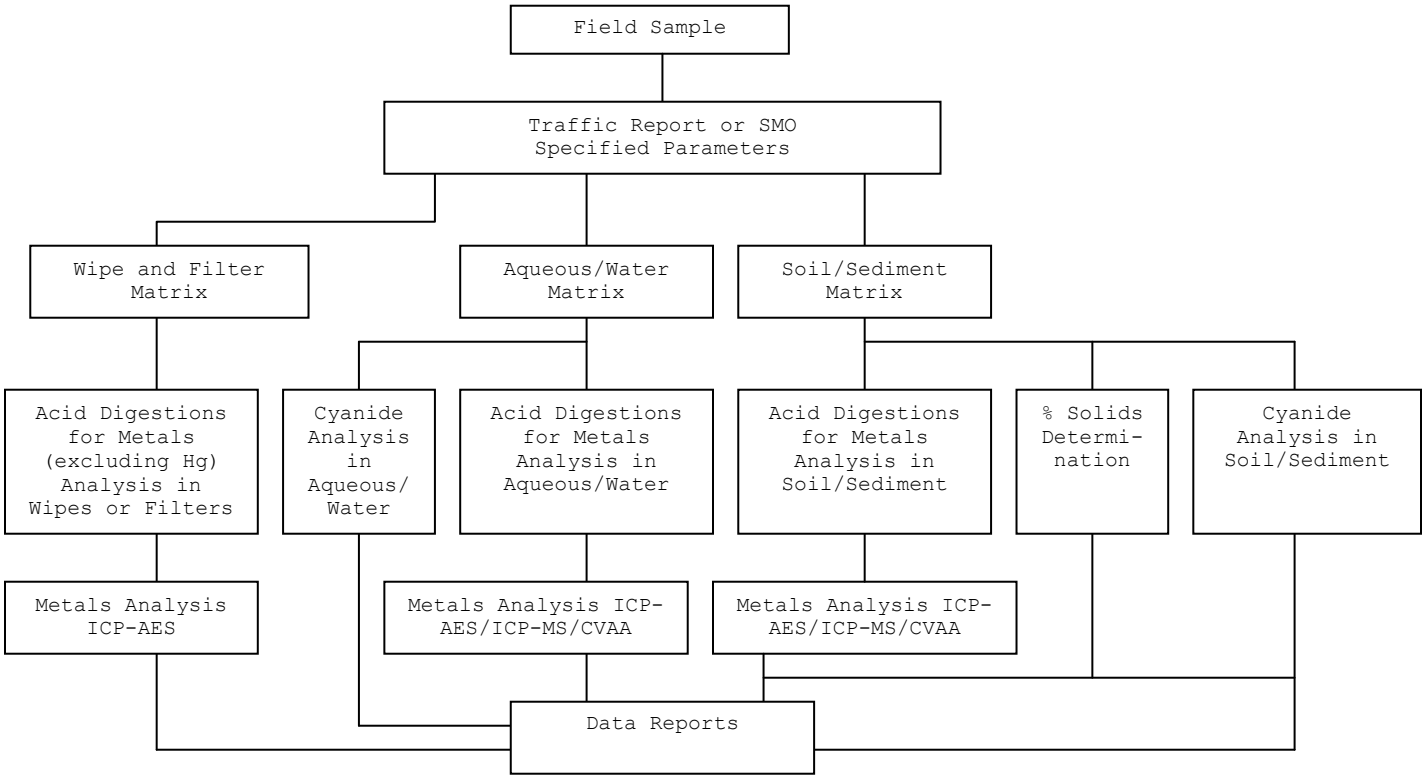
The inorganic analytical service provides a contractual framework for laboratories. This framework applies U.S. Environmental Protection Agency (USEPA) Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 23 metals (including mercury) and cyanide in aqueous/water, soil/sediment, and 22 metals (excluding mercury) in surface wipe and air filter samples.

The analytical methods that follow are designed to analyze aqueous/water, soil/sediment, surface wipe, and air filter samples from hazardous waste sites for the presence of inorganic analytes contained on the Inorganic Target Analyte List (TAL) (see Exhibit C). The inorganic methods include alternative analysis procedures for some analytes, multiple preparation procedures, and Quality Control (QC) requirements. A default preparation method is provided for each matrix, with alternative methods available for USEPA Regional use. Analytical techniques in the inorganic methodologies include Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma - Mass Spectrometry (ICP-MS), Cold Vapor Atomic Absorption Spectroscopy (CVAA), and Spectrophotometry.

1.1 Inorganic Methods Flow Chart

Figure 1 outlines the general analytical scheme the Contractor shall follow in performing standard trace metals and cyanide analyses under this contract.

1.2 Figure 1 – Inorganic Methods Flow Chart



1.3 Glassware Cleaning

Lab glassware to be used within the metals analysis must be acid cleaned according to USEPA's manual, Methods for Chemical Analysis of Water and Wastes (EPA Manual 600479020) or an equivalent procedure. Equivalent procedures are those which meet the Preparation Blank requirements in the Statement of Work (SOW). An electronic version of this manual can be found via USEPA's National Environmental Publications Internet Site (NEPIS) at http://nepis.epa.gov/EPA/html/Pubs/pubtitleORD.htm (search on EPA Manual 600479020).

1.4 Standard Stock Solutions

Stock solutions to be used for preparing instrument or method standards may be purchased or prepared as described in the individual methods of Exhibit D, Section 7 (Reagents and Standards).

1.5 Verification of Aqueous/Water Sample Preservation

1.5.1 At the time of sample receipt, the Contractor shall check the pH of the sample and note in a sample receipt log if the pH is less than or equal to 2 for metals. In addition, it should be noted if the pH is greater than or equal to 12 for a cyanide sample. If a metals sample has not been properly preserved, the Contractor may adjust the pH of a sample for metals, allow time for the sample to equilibrate prior to digestion, and note this in the Sample Delivery Group (SDG) Narrative. The Contractor shall not adjust the pH of a sample for cyanide. If the pH of a cyanide sample is <12, contact the Sample Management Office (SMO) for further instructions before proceeding with the preparation and analysis. The determination of pH for soil/sediment samples is not required.

1.5.2 Before preparation is initiated for an aqueous/water cyanide sample, the Contractor shall test for the presence of sulfides and oxidizing agents (e.g., residual chlorine). The test for sulfides shall be performed by placing a drop of the sample on a strip of lead acetate paper. If the test strip turns black, the Contractor shall treat the total volume of sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates when the sample contains sulfide. This operation shall be repeated until a drop of the treated sample solution does not darken the lead acetate test paper. The solution shall be filtered through a dry filter paper into a dry beaker, and the volume of sample to be used for analysis shall be measured from the filtrate. It is recommended that the Contractor avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. The test for oxidizing agents shall be performed by placing a drop of the sample on a strip of potassium iodide - starch test paper (KI - starch paper). If the test strip turns blue, the Contractor shall contact SMO for further instructions from the Region before proceeding with sample preparation and analysis. The Contractor shall document the presence of sulfides or oxidizing agents in the SDG Narrative.

1.6 Percent Solids Determination Procedure

1.6.1 Immediately following the weighing of the sample to be processed for analysis, add 5-10 g of sample to a tared disposable weigh boat. Weigh and record the weight to the nearest 0.01 g.

1.6.2 Place weigh boat plus sample, with the cover tipped to allow for moisture escape, in a drying oven maintained at 105°C (±5°C). Sample handling and drying should be conducted in a well-ventilated area.

1.6.3 Dry the sample overnight (12-24 hours) but no longer than 24 hours. If dried less than 12 hours, it must be documented that constant weight was attained.[1] Remove the sample from the oven and cool in a desiccator with the weigh boat cover in place before weighing. Weigh and record weight to nearest 0.01 g. Do not analyze the dried sample. Calculate percent solids by the formula below. The value thus obtained will be reported on the appropriate Forms IA-IN and IB-IN and, where applicable, Forms VA-IN and VI-IN. This value will be used for calculating analytical concentration on a dry weight basis.

EQ. 1 Percent Solids

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

1.6.4 If the sample contains less than 50% solids, the Contractor shall notify SMO immediately of the samples impacted. After notification to SMO, the Contractor shall proceed with sample analysis and document the issue in the SDG Narrative except as noted below.

1.6.5 For 14- and 21-day turnaround samples without Preliminary Results, the Contractor is required to perform the percent solids determination prior to sample preparation and analysis. Samples containing percent solids less than or equal to 30% are required to be prepared at higher sample weights to yield a dry weight equivalent to the weight range specified in the preparation method. Calculate the required sample weight by dividing the minimal method weight by the percent solids expressed as a decimal. This requirement does not apply to 7-day turnaround or Preliminary Results samples.

EQ. 2 Required Sample Weight

$$\text{Req. Wt} = \frac{\text{Minimal Method Wt}}{\%\text{Solids}/100}$$

1.7 Insufficient Sample Volume

If insufficient sample volume (less than the required amount) is received to perform the analysis, the Contractor shall contact SMO

[1] Drying time is defined as the elapsed time in the oven; thus raw data must record the time in and out of the oven to document the 12-hour drying time minimum. In the event it is necessary to demonstrate the attainment of constant weight, data must be recorded for a minimum of two repetitive weigh/dry/desiccate/weigh cycles with a minimum of 1-hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01 g between the start weight and final weight of the last cycle.

to apprise them of the problem. SMO will contract the USEPA Region for instructions. The Region will either approve that no sample analysis be performed or will require that a reduced volume be used for the sample analysis. No other changes in the analysis will be permitted. SMO will notify the Contractor of the Region's decision. The Contractor shall document the Region's decision in the SDG Narrative.

1.8 Sample Mixing

Unless instructed otherwise by the USEPA Regional CLP Project Officer (CLP PO), all samples shall be mixed thoroughly prior to aliquoting for digestion. There is no specific procedure provided herein for homogenization of soil/sediment samples; however, an effort should be made to obtain a representative aliquot. Coarse stones, twigs, or debris that are not representative of the soil/sediment should be excluded from the aliquot. For multi-phase samples, see Exhibit D, Section 10.1.

1.9 Undiluted Analysis

1.9.1 Unless the dilution-adjusted detection limits for all analytes are below the Contract Required Quantitation Limits (CRQLs), all samples for multi-analyte analysis shall be run undiluted. When an analyte concentration exceeds the calibrated range, appropriate dilution (but not below the CRQL) and re-analysis is required. The Contractor shall use the least dilution necessary to bring the analyte(s) instrument reading within the upper 75% of the calibrated range and report the highest valid value for each analyte as measured from the undiluted and diluted analyses. Unless the Contractor can submit proof that dilution was required to obtain valid results, or to avoid damage to ICP-MS instruments, both diluted and undiluted sample measurements must be contained in the raw data.

1.9.2 For single analyte analysis, a diluted sample analysis may be the only sample analysis performed if the analyte's instrument result is in the upper 75% of the calibrated range. An undiluted sample analysis does not have to be performed in this case. The sample and its associated matrix spike and duplicate shall initially be run at the same dilution.

1.9.3 All sample dilutions shall be made with reagent water appropriately acidified (except for cyanide) to maintain constant acid strength.

1.10 Dissolved Metals

For samples filtered in the field, the Contractor shall digest the samples designated as dissolved metals. If dissolved metals are requested by USEPA Regional Offices, the Contractor shall digest the field-filtered samples designated as dissolved metals. Only if requested as a Modified Analysis shall the Contractor filter samples prior to digestion or perform direct analysis.

1.11 Replicate Integrations/Exposures

If the Contractor analyzes samples using multiple integrations/exposures, the Contractor must use the data obtained from all integrations/exposures to calculate the final sample result even if more than the minimum number of integrations/exposures are taken.

1.12 Raw Data Requirements

The Contractor is reminded and cautioned that the collection and provision of raw data may or may not be referred to within the individual methods of Exhibit D or the Quality Assurance (QA) protocol of Exhibit E. The raw data deliverable requirements are specified in Exhibit B, Section 2.5.2.3. Raw data collected and provided in association with the performance of analyses under this contract shall conform to the appropriate provisions of Exhibit B.

1.13 Quality Control Samples

If the Sampler designated two (or more) samples as QC for the same matrix, and the QC samples are not specifically labeled with the analysis they are to be used for (dissolved metals and total metals), then the Contractor is to contact SMO to report the issue. SMO shall then contact the USEPA Region and notify the Contractor of the Regional decision. If the Sampler did not designate QC samples, then the Contractor is to select a sample for QC and to contact SMO to report the issue.

1.14 Safety

The toxicity or carcinogenicity of each reagent used in this SOW has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of chemicals specified in this method. A reference file of material handling data sheets should also be made available to all personnel involved in the chemical analysis.

1.15 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, USEPA recommends recycling as the next best option.

1.16 Waste Management

USEPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. USEPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

EXHIBIT D - PART A

ANALYTICAL METHODS
FOR
INDUCTIVELY COUPLED PLASMA -
ATOMIC EMISSION SPECTROSCOPY

Exhibit D – Analytical Methods for ICP-AES

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1.0 SCOPE AND APPLICATION

This method provides procedures for the use of Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) to determine the concentrations of total recoverable and dissolved elements in aqueous/water, soil/sediment, surface wipe, and air filter samples taken from hazardous waste sites. All metals (except mercury) contained in the Inorganic Target Analyte List (TAL) in Exhibit C are quantitated by this method.

2.0 SUMMARY OF METHOD

2.1 General Method Overview

This method describes the multi-element determination of trace metals by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES). Aqueous/water, soil/sediment, wipe, and air filter samples are treated with acids and heat to solubilize the metals present. These digestates are then analyzed for trace metals by an atomic emission optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to a plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed and the intensities of the lines are monitored by a photosensitive device. The signals from the photosensitive device are processed by a computer. A background correction technique is required to compensate for variable background contribution to the spectra of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

2.2 Summary of Digestion Procedures

2.2.1 Hotplate Acid Digestion of Aqueous/Water Samples (based on USEPA 200.7)

2.2.2 Hotplate Acid Digestion of Soil/Sediment Samples (based on USEPA 3050B)

2.2.3 Hotplate Acid Digestion of Wipe Samples (based on USEPA 3050B)

2.2.4 Hotplate Acid Digestion of Filter Samples (based on a modified version of NIOSH 7300)

2.2.5 Alternative digestion procedures are included in Section 18 of this Exhibit.

3.0 DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements in aqueous/water and soil/sediments. To prevent this, appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 mg/L and when total elements are determined after the appropriate digestion procedures are performed. Several types of interferences are given in Sections 4.1 through 4.3 below.

4.1 Spectral Interferences

Spectral interferences can be categorized as: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; (4) and/or background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data. This would require the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array.

4.2 Physical Interferences

Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies, especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may minimize these interferences. If these types of interferences are present, they must be reduced by dilution of the sample.

Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution has been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

Internal standardization may be effectively used to compensate for many physical interference effects.

4.3 Chemical Interferences

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, and by matrix matching. These types of interferences can be highly dependent on matrix type and the specific element.

5.0 SAFETY

See Section 1.14 in Exhibit D - Introduction to Analytical Methods.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 Glassware/Labware

6.1.1 250 mL beaker or other appropriate digestion vessel (glass or plastic)

6.1.2 Watch glasses (glass or plastic)

6.1.3 Funnels

6.1.4 Graduated cylinders

6.1.5 Various volumetric flasks and pipets (Class A)

6.1.6 Thermometer that covers a range of 0-200°C

6.1.7 Whatman No. 41 filter paper (or equivalent)

6.1.8 Hotplate, block digester, or other heating source capable of maintaining 95°C (±3°C).

6.1.9 Balances – Top loader balance, 300 gram (g) capacity, and a minimum sensitivity of ±1.0 milligram (mg)

6.2 Equipment and Supplies for Microwave Digestion

The equipment and supplies listed in this section are used if one of the alternate microwave sample preparation procedures was approved for use by the USEPA Region.

6.2.1 Whatman No. 41 filter paper (or equivalent)

6.2.2 Disposable polypropylene filter funnel

6.2.3 Polyethylene bottles, 125 mL, with caps

6.2.4 Microwave oven with programmable power settings up to at least 600 watts.

6.2.5 The system must use polytetrafluoroethylene perfluoroalkoxy (PTFE PFA) digestion vessels (120 mL capacity) capable of withstanding pressure of up to 110 (±10) psi [7.5 (±0.7) atm]. These vessels are capable of controlled pressure relief at pressures exceeding 110 psi.

6.2.6 A rotating turntable must be used to ensure homogeneous distribution of microwave radiation within the oven. The speed of the turntable must be a minimum of 3 revolutions per minute (rpm).

6.3 Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES)

Consisting of a computer-controlled atomic emission spectrometer with background correction, a radio-frequency generator, and a supply of Argon gas, welding grade or better.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-06). Use this water for all reagents, standards, and dilutions.

7.1.2 Hydrochloric acid - Concentrated (specific gravity 1.19).

7.1.3 Hydrochloric acid, (50% v/v) - Add 500 mL conc. hydrochloric acid (specific gravity 1.19) to 400 mL reagent water and dilute to 1 L.

7.1.4 Nitric acid - Concentrated (specific gravity 1.41).

7.1.5 Nitric acid, (50% v/v) - Add 500 mL conc. nitric acid (specific gravity 1.41) to 400 mL reagent water and dilute to 1 L.

7.1.6 Hydrogen peroxide (30%)

7.1.7 Nitric acid, (5% v/v) - Add 50 mL conc. nitric acid (specific gravity 1.41) to 500 mL reagent water and dilute to 1 L.

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards, except as noted, to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit E, Section 8.0. The Contractor must be able to verify that the standards are certified. Manufacturer’s certificates of analysis must be retained by the Contractor and presented upon request.

7.2.2 Stock Standard Solutions

7.2.2.1 Stock standard solutions may be purchased from a reputable source or prepared from reagent grade chemicals or metals (at least 99.99% pure). All salts must be dried for 1 hour at 105°C unless otherwise specified.

(CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.)

7.2.3 Secondary Dilution Standards

7.2.3.1 Mixed Secondary Dilution Standards

Prepare mixed secondary dilution standard solutions by diluting the appropriate volumes of stock standards with 2% (v/v) nitric acid, or as recommended by the manufacturer, to obtain the final volume. Mixed secondary dilution standard solutions may be purchased. The purchased standards shall meet the requirements in Section 7.2.1.

7.2.4 Working Standards

7.2.4.1 Interference Check Sample (ICS) Solution

The ICS consists of two solutions: Solution A (ICSA) and Solution AB (ICSAB). ICSA consists of the interferents and ICSAB consists of the analytes mixed with the interferents. The ICS standards (ICSA for the interferents only and ICSB for the analytes only) are normally supplied by USEPA.

7.2.4.2 Mixed Calibration Standard Solutions

7.2.4.2.1 Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add 2 mL of nitric acid and dilute to 100 mL with reagent water or these standards can be matrix matched to the digested samples. The analyte concentrations in the calibration standards should be sufficient to produce good measurement precision and to accurately define the slope of the response curve. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards that the analytes are compatible and stable. Transfer the mixed standard solutions to fluorinated ethylene propylene (FEP) fluorocarbon or unused polyethylene bottles for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change with aging.

7.2.4.2.2 Protect all standards from light. Samples, sample digestates, and standards must be stored separately.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in glass or polyethylene containers. Aqueous/water samples must be preserved with nitric acid to pH less than or equal to 2 immediately after collection. All aqueous/water and soil/sediment samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until digestion. Wipes and air filter samples shall remain in their original bags or cassettes until preparation and may be stored at room temperature within the laboratory.

8.1.1 Dissolved Metals

For the determination of dissolved metals, the sample must be filtered through a 0.45 micrometer (µm) pore diameter membrane filter at the time of collection or as soon as possible. Use a portion of the sample to rinse the filter flask, discard this portion, and collect the required volume of filtrate. Preserve the filtrate with nitric acid to pH less than or equal to 2 immediately after filtration.

8.2 Procedures for Sample Storage and Disposal

Following digestion, the remaining unused portion of aqueous/water and soil sediment samples may be stored at room temperature within the laboratory until 60 days after delivery of a complete, reconciled data package to USEPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.3 Procedure for Sample Digestate Storage

Sample digestates must be stored until 6 months after delivery of a complete, reconciled data package to USEPA.

8.4 Contract Required Holding Time

The maximum holding time for metals is 180 days from Validated Time of Sample Receipt (VTSR).

9.0 CALIBRATION AND STANDARDIZATION

9.1 Instrument Operating Parameters

Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. The Method Detection Limit (MDL), precision, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements used to determine interelement corrections must be within the instrument operating range. It is the responsibility of the analyst to verify the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2 Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES) Instrument Calibration Procedure

9.2.1 Instruments shall be calibrated each time the instrument is turned on, set up, or after Initial Calibration Verification (ICV) or Continuing Calibration Verification (CCV) failure. The instrument standardization date and time shall be included in the raw data.

9.2.2 The calibration standards shall be prepared as in Section 7.2.4.2.

9.2.3 Calibrate the ICP-AES instruments according to instrument manufacturer’s recommended procedures. At least six standards shall be used for ICP-AES calibration. One of the standards shall be a blank standard and one shall be at or below the Contract Required Quantitation Limit (CRQL). The rest of the standards shall be uniformly spread out in graduated amounts over the appropriate calibration range for each metal. A minimum of three replicate integrations are required for data acquisition. Use the average of all the integrations for instrument calibration and data reporting. The calibration curve shall be calculated using linear regression by plotting the concentration of the standard (in µg/L) on the X-axis versus the corrected instrument response on the Y-axis. The corrected instrument response are those corrections (e.g., such as correction for background, internal standards, Interelement Corrections (IECs), calibration blank) that may be applied to the raw uncorrected instrument response prior to determining the calibration curve. A standard linear regression, a weighted linear regression (e.g., 1/concentration or 1/concentration^2), or a linear regression with zero force calibration model can be used as appropriate. No other types of equations (e.g., quadratic) are to be used. The acceptance criteria for the calibration curve is a correlation coefficient greater than or equal to 0.995. Sample analysis shall not begin until this criteria and the criteria described in Sections 9.2.4 and 9.2.5 have been met.

9.2.4 The calibration equation must be checked to establish the representativeness of the data that were used to produce the calibration equation. This check involves the re-fitting of the non-blank calibration data back to the calibration equation or the comparison of the calculated concentration of each of the standards against the expected concentration of the associated standard. This difference is related to the actual residual. For this calculation, the Percent Difference shall be used where the above difference is

further divided by the expected amount or concentration of the respective standard. If these Percent Differences for each of the standards do not fall within ±30%, then the calibration equation is not acceptable and must be corrected. If a standard is analyzed for a particular analyte at a concentration that is below the CRQL and the above criteria is not met, that value can be excluded from the calibration equation as long as the lowest non-zero standard for each of the remaining standards for each analyte is still analyzed at or below the CRQL and all standards included in the calibration equation are continuous and consecutive.

9.2.5 The y-intercept from the linear regression initial calibration equation shall also be evaluated. If the y-intercept is not below the CRQL for each of the analyte initial calibration curves, the calibration is not acceptable and must be corrected. Samples are not to be analyzed until the y-intercept for each analyte's initial calibration curve meets the acceptance criteria.

9.2.6 Any changes or corrections to the analytical system shall be followed by recalibration.

9.3 Initial Calibration Verification (ICV)

9.3.1 Immediately after each instrument has been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of the ICV solution(s) at each wavelength used to report final results. The percent Relative Standard Deviation (%RSD) shall also be calculated from all replicate integrations and reported for each wavelength used to report final results.

9.3.2 The ICV solution(s) is (are) obtained from USEPA. If the solution(s) is (are) not available, then the ICV solution can be prepared by the laboratory using a certified solution for each analyte from an independent source. Analyses shall be conducted at a concentration other than that used for instrument calibration, but near the middle of the calibration range. An independent source is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

9.3.3 The ICV solution(s) shall be run at each wavelength used for reporting final results.

9.4 Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each analytical run sequence, a CCV shall be analyzed and reported for every wavelength used for reporting final results for each analyte, at a frequency not to exceed every 2 hours during an analytical run sequence. The standard shall be analyzed and reported for every wavelength used for reporting final results for each analyte at the beginning of the analytical run sequence and after the last analytical sample. The %RSD shall be calculated from all integrations and reported for each wavelength used to report final results. See the example analytical run sequence in Section 12.11. This analytical run sequence can continue indefinitely as long as samples are being continuously analyzed without the instrument being turned off and successive CCVs and their associated %RSDs meet the stated criteria. This CCV standard(s) shall be prepared from the same source and at or near the same mid-level concentration as used during the initial calibration.

The same CCV standard shall be used throughout the analytical run sequences for a Sample Delivery Group (SDG) of samples received.

9.4.1 Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding analytical samples up to the previous CCV as applicable and the analytical samples following this CCV up to the next CCV as applicable).

9.5 Initial and Continuing Calibration Blank (ICB/CCB)

An ICB or CCB shall be analyzed at each wavelength used for reporting final results for each analyte immediately after every ICV and CCV.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 If insufficient sample amount (less than 90% of the required amount) is received to perform the analyses, the Contractor shall contact the Sample Management Office (SMO) to inform them of the problem. SMO will contact the USEPA Region for instructions. The USEPA Region will either require that no sample analyses be performed or will require that a reduced volume be used for the sample analysis. No other changes in the analyses will be permitted. The Contractor shall document the USEPA Region's decision in the Sample Delivery Group (SDG) Narrative.

10.1.2 If multiphase samples (e.g., two-phase liquid sample, oily sludge/sandy soil/sediment sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the USEPA Region. If all phases of the sample are amenable to analysis, the USEPA Region may require the Contractor to do any of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.
- Separate the phases of the sample and analyze one or more of the phases, separately. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.1 If all of the phases are not amenable to analysis (i.e., outside scope), the USEPA Region may require the Contractor to do any of the following:

- Separate the phases and analyze the phase(s) that is (are) amenable to analysis. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.2 No other changes in the analyses will be permitted. The Contractor shall document the USEPA Region's decision in the SDG Narrative.

10.1.3 The primary sample preparation procedures to be used for this method are described in the following Sections (10.1.4, 10.1.5, 10.1.6, and 10.1.7). These methods are to be used without deviation. Alternative sample preparation procedures are listed in Section 18 of this method and may be requested by the USEPA Region. No other changes in the analyses will be permitted.

10.1.4 Aqueous/Water Sample Preparation

10.1.4.1 Preparation Method 200.7 - Total Recoverable Analytes [based on USEPA NERL Method 200.7, Revision 4.4 (1994)]

10.1.4.1.1 For the determination of total recoverable analytes in aqueous/water samples, transfer a 100 mL (±1 mL) aliquot from a well mixed, acid preserved sample to an appropriately sized (approximately 250 mL) beaker or other comparable digestion vessel. The sample shall not be diluted prior to digestion.

10.1.4.1.2 Add 2 mL 50% (v/v) nitric acid and 1 mL 50% (v/v) hydrochloric acid to the beaker containing the measured volume of sample. Place the beaker on a hotplate, or other comparable heating device, for solution evaporation. The hotplate should be located in a fume hood and previously adjusted to provide evaporation at a temperature of 95°C (±3°C), when covered. The beaker should be covered with an elevated watch glass or other necessary steps should be taken to prevent sample contamination from the fume hood environment.

10.1.4.1.3 Reduce the volume of the sample aliquot to about 20 mL by gently heating at 95°C (±3°C). **DO NOT BOIL.** This step takes about 2 hours for a 100 mL aliquot with the rate of evaporation rapidly increasing as the sample volume approaches 20 mL. (A spare beaker containing 20 mL of water can be used as a gauge.)

10.1.4.1.4 Cover the beaker with a watch glass to reduce additional evaporation and gently reflux the sample for 30 minutes. (Slight boiling may occur, but vigorous boiling must be avoided to prevent loss of the $HCl-H_2O$ azeotrope.)

10.1.4.1.5 Allow the beaker to cool. Quantitatively transfer the sample solution to a 100 mL volumetric flask, make to volume with reagent water, stopper, and mix.

10.1.4.1.6 Allow any undissolved material to settle overnight, or centrifuge or filter a portion of the prepared sample until clear to avoid plugging the nebulizer with solid particles. The sample is now ready for analysis. Because the effects of various matrices on the stability of the samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.

10.1.4.1.7 The concentrations as determined by this method shall be reported as "Total". A reduced sample volume of 50 mL can be used. If this reduced volume is used, then all other reagents and volumes shall be reduced appropriately. The digested sample can be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

10.1.5 Soil/Sediment Sample Preparation

10.1.5.1 Preparation Method 3050B (based on USEPA OSW Method 3050B, Revision 2, December 1996)

10.1.5.1.1 Mix the sample thoroughly to achieve homogeneity (see Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh to the nearest 0.01 g and transfer 1.00 – 1.50 g sample (wet weight) to an appropriately sized digestion vessel (e.g., a beaker or hot block digestion tube).

10.1.5.1.2 Add 10 mL of 50% (v/v) nitric acid, mix the slurry, and cover with a watch glass or vapor recovery device. Heat the sample to 95°C (± 3°C) and reflux for 10 to 15 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated nitric acid, replace the cover, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by the nitric acid, repeat this step (addition of 5 mL of concentrated nitric acid) over and over until no brown fumes are given off by the sample indicating the complete reaction with nitric acid. Using a ribbed watch glass or vapor recovery system, either allow the solution to evaporate to approximately 5 mL without boiling or heat at 95°C (± 3°C) without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times.

10.1.5.1.3 After the sample has cooled, add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide. Cover the vessel with a watch glass or vapor recovery device and return the covered vessel to the heat source for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until the effervescence subsides and cool the vessel. Continue to add 30% hydrogen peroxide in 1-mL amounts with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL of 30% hydrogen peroxide.) Cover the sample with a ribbed watch glass or vapor recovery device and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL or heat at 95°C (± 3°C) without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times.

10.1.5.1.4 After the sample has cooled, add 10 mL of concentrated hydrochloric acid to the sample digestate and cover with a watch glass or vapor recovery device. Place the sample on/in the heating source and reflux at 95°C (±3°C) for 15 minutes. Let the sample digestate cool.

10.1.5.1.5 Filter the sample digestate through Whatman No. 41 filter paper (or equivalent) and collect the filtrate in a 100-mL volumetric flask. Rinse the filter paper with a small amount of reagent water to complete the quantitative transfer of the analytes and collect the liquid in the same 100-mL volumetric flask. The solution being analyzed must be clear to avoid plugging the nebulizer with solid particles. Make to volume with reagent water, stopper, and mix. The sample is now ready for analysis.

10.1.5.1.6 The digested sample can be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

10.1.6 Wipe Sample Preparation

10.1.6.1 Preparation Method 3050B [based on USEPA OSW Method 3050B, Revision 2 (December 1996)]

10.1.6.1.1 Transfer the wipe to an appropriately sized digestion vessel (e.g., a beaker or hot block digestion tube). If material remains in the original sample container, use a small (5 mL) portion of reagent water to rinse the material into the digestion vessel.

10.1.6.1.2 Follow the procedure as described in Sections 10.1.5.1.2 through 10.1.5.1.6.

10.1.7 Filter Sample Preparation

10.1.7.1 Preparation Method 7300 (based on a modified version of NIOSH method 7300 from the Fourth Edition of the NIOSH Manual of Analytical Methods, Issue 3, dated 15 March 2003). This procedure is for small (e.g., 37 mm MCEF) filters. The method is not suitable for high-volume filters.

10.1.7.1.1 The method was modified by removing the use of all perchloric acid during the sample preparation due to safety issues. If the Contractor has the necessary equipment to safely work with and perform perchloric acid digestions, then the Contractor should contact SMO. SMO will then contact the USEPA Region and decide if this modified method or the original method should be used for the samples within this specific SDG.

10.1.7.1.2 Remove the filter from the cassette and transfer to a 50 mL beaker.

10.1.7.1.3 Add 3 mL of concentrated nitric acid and 1 mL of 30% of hydrogen peroxide and cover with a watch glass. Heat the sample to 95°C (±3°C) on a hotplate, block digester, or equivalent heat source and reflux until the volume has been reduced to approximately 0.5 mL.

10.1.7.1.4 Rinse the watch glass and the sides of the beaker with 3 mL of 5% (v/v) nitric acid and heat until the sample volume has been reduced to approximately 0.5 mL.

10.1.7.1.5 Add 1 mL of concentrated hydrochloric acid and heat for 10 minutes at 95°C (±3°C). Allow the sample to cool.

10.1.7.1.6 Adjust the final volume to 10.0 mL (±0.1 mL) with reagent water. The sample may be filtered to remove solid particles. The sample is now ready for analysis.

10.2 Sample Analysis

10.2.1 For every new or unusual matrix, it is recommended that a semi-quantitative analysis be conducted to screen for high element concentrations that may be beyond the calibration range of the instrument or high levels of interferences.

10.2.2 Set up the instrument with proper operating parameters established in Section 9.1. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 minutes of operation prior to calibration.

10.2.3 Initiate appropriate operating configuration of the computer.

10.2.4 Profile and calibrate the instrument according to instrument manufacturer’s recommended procedures. The initial calibration curve shall be established using mixed calibration standard solutions such as those described in Section 7.2.4.2.

10.2.5 For all sample analyses, a minimum of three replicate integrations are required for data acquisition. Use the average of all the integrations for data reporting.

10.2.6 The rinse blank should be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample. Samples should be aspirated for a sufficient period of time to obtain a stable response prior to collection of data.

10.2.7 Sample digestates having high levels of interferences or concentrations higher than the established calibrated range as determined by the expected concentration of the highest calibration standard shall be diluted into range and reanalyzed.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Aqueous/Water Sample Calculation

EQ. 1 Aqueous/Water Sample Concentration

$$\text{Concentration}(\mu g/L) = C \times \frac{V_f}{V} \times DF$$

WHERE,
- C = Instrument value in µg/L (The average of all replicate exposures)
- V_f = Final digestion volume (mL)
- V = Initial aliquot amount (mL)
- DF = Dilution Factor

11.2 Soil/Sediment Sample Calculation

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of mg/kg:

EQ. 2 Soil/Sediment Sample Concentration

$$\text{Concentration (dry wt.)}(mg/kg) = C \times \frac{V_f}{W \times S} \times DF / 1000$$

WHERE,
- C = Instrument value in µg/L (The average of all replicate exposures)
- V_f = Final digestion volume (mL)
- W = Initial aliquot amount (g)
- S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
- DF = Dilution Factor

11.3 Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, multiply the value of the MDL or CRQL (µg/L) by the sample dilution factor and the V_f/V term as noted in Equation 1.

Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

EQ. 3 Adjusted Soil/Sediment MDL or CRQL Concentration

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_M}{W \times S} \times \frac{V_f}{V_M} \times DF$$

WHERE,
- C = MDL or CRQL Concentration (mg/kg)
- W_M = Minimum method required aliquot amount (g) (1.00 g or 0.50 g)
- W = Initial aliquot amount (g)
- V_M = Method required final sample digestion volume (mL) (100 mL or 50 mL)
- V_f = Final digestion volume (mL)
- S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
- DF = Dilution Factor

11.4 Wipe/Filter Mass

EQ. 4 Wipe/Filter Mass

$$\text{Mass}(\mu g) = C \times V_f \times DF/1000$$

WHERE,
- C = Instrument value in µg/L (The average of all replicate exposures).
- V_f = Final digestion volume (mL)
- DF = Dilution Factor

12.0 QUALITY CONTROL (QC)

12.1 Initial Calibration Verification (ICV)

The ICV standard shall be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier. If measurements exceed the control limits of 90% Recovery (low) or 110% Recovery (high), or if the percent Relative Standard Deviation (%RSD) as calculated from all replicate exposures exceeds 5.0%, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. The results of the ICV analysis shall be reported.

12.2 Continuing Calibration Verification (CCV)

The CCV standard shall be prepared in the same acid matrix as the calibration standards by combining compatible analytes at a concentration equivalent to the mid-levels of their respective calibration curves. If the measurements exceed the control limits specified of 90% Recovery (low) or 110% Recovery (high), or if the %RSD as calculated from all replicate exposures exceeds 5.0%, the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration verification shall be performed for the analytes affected. The results of all CCV analyses shall be reported.

12.3 Blank Analyses

There are two different types of blanks required by this method. The calibration blank is used in establishing the analytical curve. The Initial and Continuing Calibration Blanks (ICB/CCB) are identical in composition to the calibration blank and are analyzed immediately after the ICV/CCV to monitor for potential carryover of analytes. The Preparation Blank is used to monitor for possible contamination throughout the entire sample preparation and analysis process.

12.3.1 Initial and Continuing Calibration Blank (ICB/CCB)

The ICB and CCB are identical in composition to the Calibration Blank as used to establish the initial calibration curve. If the absolute value of the calibration blank (ICB/CCB) result exceeds the Contract Required Quantitation Limit (CRQL) (see Exhibit C), the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration blank shall be performed for the analytes affected. The results of the ICB and CCB analyses shall be reported.

12.3.2 Preparation Blank

12.3.2.1 The Preparation Blank shall contain all the reagents and in the same volumes as used in processing the samples. The Preparation Blank shall be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

12.3.2.2 At least one Preparation Blank, consisting of reagent water, a clean wipe, or a clean air filter, processed through each sample preparation and analysis procedure (see Section 10), shall be prepared and analyzed with every Sample Delivery Group (SDG), or with each batch[1] of samples digested, whichever is more frequent. For wipes and filters, the sampler should provide clean wipes or filters. In the event that they are not provided by the sampler, the Contractor shall use reagent water and note this in the SDG Narrative.

12.3.2.3 The first batch of samples in an SDG is to be assigned to Preparation Blank one, the second batch to Preparation Blank two, etc. Each Complete SDG File (CSF) shall contain the results of all Preparation Blank analyses associated with the samples in that SDG.

12.3.2.4 The Preparation Blank(s) is (are) to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

12.3.2.4.1 If the absolute value of the concentration of the blank is less than or equal to the CRQL (see Exhibit C), no further action is required.

12.3.2.4.2 For aqueous/water and soil/sediment samples, if any analyte concentration in the blank is above the CRQL, the lowest concentration of that analyte in the associated samples (except those identified as field blanks) shall be greater than or equal to 10 times the blank concentration. Otherwise, all samples (except those identified as field blanks) associated with the blank, with the analyte concentration less

[1] A group of samples prepared at the same time.

than 10 times the blank concentration and above the CRQL, shall be redigested and reanalyzed with appropriate new Quality Control (QC) for that analyte. The sample concentration is not to be corrected for the blank value.

12.3.2.4.3 For aqueous/water and soil/sediment samples, if the concentration of the blank is below the negative CRQL, then all samples reported below 10 times the CRQL associated with the blank shall be redigested and reanalyzed with appropriate new QC.

12.3.2.4.4 The results for the Preparation Blank shall be reported.

12.4 Interference Check Sample (ICS)

12.4.1 The ICS solutions shall be obtained from USEPA. If not available, then the ICS can be prepared by the analyst.

12.4.2 To verify interelement and background correction factors, the Contractor shall analyze and report the results for the ICS for all elements on the Target Analyte List (TAL) and for all interferents (target and non-target) immediately after the initial calibration sequence, but not before the ICV/ICB. The analysis of the ICS shall be immediately followed by the analysis of a CCV/CCB pair. The ICS solutions shall be obtained from USEPA, if available, and analyzed according to the instructions supplied with the ICS. The Contractor shall always initially run the ICS undiluted. Dilution of the ICS (for the highest concentration elements) may be necessary to meet the calibrated range values of the instrument.

12.4.3 The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents; Solution AB consists of the analytes mixed with the interferents. Even though no analytes have been added to Solution A, low levels of some of the analytes may be present due to the purity of the materials used. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A.

12.4.4 The analytical results of ICS Solution A (ICSA) shall fall within the control limit of ±20% of the analyte's true value or ±1 times the CRQL of the analyte's true value, whichever is greater. If the true value for a given analyte is not listed in the certified values for the solution, then the true value shall be assumed to be zero and the ±1 times the CRQL control limits shall apply. For example, for Chromium (CRQL = 10 µg/L, ICSA true value = 43 µg/L) the correct control window to use would be the greater of ±20% of the true value (0.20 x 43 µg/L = ±8.6 µg/L) or ±1 times the CRQL (±10 µg/L). Therefore, the control window for the found value for Chromium in the ICSA is 43±10, or 33 to 53 µg/L. If the analytical results of the ICSA do not fall within the control limits, the analysis shall be terminated, the problem corrected, and the instrument recalibrated. New Interelement Corrections (IECs) may also need to be determined for the failed analyte(s). For analytes with CRQLs less than 1000 µg/L, the ICSA results shall be reported from an undiluted sample analysis.

12.4.5 Results for the ICS Solution AB (ICSAB) during the analytical runs shall fall within the control limit of ±20% of the true value or ±1 times the CRQL of the true value, whichever is greater, for the analytes included in the ICSAB. If the analytical results of the ICSAB do not fall within the control limits, the analysis shall be

terminated, the problem corrected, and the instrument recalibrated. New IEC's may also need to be determined for the failed analyte(s). For analytes with CRQLs less than 1000 µg/L, the ICSAB shall be reported from an undiluted sample analysis.

NOTE: The control limits and concentrations for the ICSAB are being monitored. These may be adjusted to provide greater control of interferences.

12.4.6 If true values for analytes contained in the ICS are not supplied with the solutions, the mean shall be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination shall be made during an analytical run where the results for the previously supplied ICS met all contract specifications. Additionally, the results of this initial mean determination shall be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted). Only if the ICS solutions are not available from USEPA, independent Check Samples shall be prepared with interferent and analyte concentrations at the levels specified in Table 1 - Interferent and Analyte Elemental Concentrations Used for ICP-AES Interference Check Sample (ICS). The mean value and standard deviation shall be established by initially analyzing the Check Samples at least five times repetitively for each parameter. Results shall fall within the control limit of ±20% of the established mean value or ±1 times the analyte's CRQL of the established mean value, whichever is greater. The mean and standard deviation shall be reported in the raw data.

12.4.7 The results of ICS analyses shall be reported.

12.5 Spike Sample Analysis

12.5.1 The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology. The spike is added before the digestion (i.e., prior to the addition of other reagents). At least one spike sample analysis (matrix spike) shall be performed on each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[2] Spike sample analysis is not required for wipe samples or air filter samples.

12.5.2 If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 12.6). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks and Performance Evaluation (PE) samples shall not be used for spiked sample analysis. USEPA may require that a specific sample be used for the spike sample analysis.

12.5.3 The analyte spike shall be added in the amount given in Table 2 - Spiking Levels for Spike Sample Analysis, for each element analyzed. This is the level of spike present in the final digestate.

NOTE: See Table 2 footnotes for concentration levels and applications.

[2] USEPA may require additional spike sample analyses, upon USEPA Regional CLP Project Officer (CLP PO) request.

12.5.4 If the spike recovery is not at or within the limits of 75-125%, the data for all samples received and associated with that spike sample shall be flagged with the letter "N". An exception to this rule is granted when the sample concentration exceeds the Spike Added (SA) concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

12.5.5 When the matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike shall be performed for those analytes that do not meet the specified criteria (exception: Ag). Note that if a post-digestion spike analysis is required for an analyte, the same EPA sample that was used for the matrix spike analysis shall be used for the post-digestion spike analysis. Spike the unspiked aliquot of the undiluted digestate at two times the indigenous level or two times the CRQL, whichever is greater.

12.5.6 In the instance where there is more than one spike sample per matrix per SDG, if one spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG. Individual component percent recoveries are calculated as follows:

EQ. 5 Matrix Spike and Post-Digestion Spike Percent Recovery

$$\%\text{Recovery} = \frac{SSR - SR}{SA} \times 100$$

WHERE, SSR = Spiked Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2
SR = Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2
SA = Spike Added Theoretical Result (µg/L or mg/kg). This is calculated by substituting the spiking amount used for the 'V_f' term and substituting the spiking standard concentration used for the 'C' term from EQ. 1 or EQ. 2.

12.5.7 When sample concentration is less than the Method Detection Limit (MDL), use SR = 0 only for purposes of calculating the percent recovery. The Spike Sample Results (SSRs), Sample Results (SRs), Spike Added Theoretical Results (SA), and percent recovery (positive or negative) shall be reported.

12.5.8 The units used for reporting Spiked Sample Results (SSRs) will be identical to those used for reporting Sample Results (SRs).

12.6 Duplicate Sample Analysis

12.6.1 One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[3] Duplicates cannot be averaged for reporting. Duplicate samples are not required for wipe samples or air filter samples.

[3] USEPA may require additional duplicate sample analyses, upon USEPA Regional CLP PO request.

12.6.2 Duplicate sample analyses are not required for percent solids. Samples identified as field blanks and PE samples shall not be used for duplicate sample analysis. USEPA may require that a specific sample be used for duplicate sample analysis. The Relative Percent Difference (RPD) for each analyte is calculated as follows:

EQ. 6 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

WHERE, RPD = Relative Percent Difference
S = Sample Result (original) (µg/L or mg/kg) from EQ. 1 or EQ. 2
D = Duplicate Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2

12.6.3 The results of the duplicate sample analyses shall be reported. A control limit of 20 for RPD shall be used for original and duplicate sample values greater than or equal to five times the CRQL (see Exhibit C). A control limit equal to the CRQL shall be entered in the "Control Limit" column if either the sample or duplicate value is less than five times the CRQL. If the sample and duplicate values are greater than or equal to five times the CRQL, or if the sample and duplicate values are less than the CRQL, the "Control Limit" field is left empty.

12.6.4 If one result is above five times the CRQL level and the other is below, use the CRQL criteria to determine if the duplicate analysis is in control. If both sample and duplicate values are less than the CRQL, the RPD is not calculated. For soil/sediment sample or soil/sediment duplicate results less than five times the CRQL, enter the value of the CRQL, corrected for sample weight and percent solids (i.e., original, not duplicate sample weight), in the "Control Limit" column. If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*". In the instance where there is more than one duplicate sample per SDG, if one duplicate result is not within contract criteria, flag all samples of the same matrix in the SDG. The percent difference data will be used by USEPA to evaluate the long-term precision of the methods for each analyte. Specific control limits for each analyte will be added at a later date based on these precision results.

12.7 Laboratory Control Sample (LCS) Analysis

12.7.1 Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte using the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures employed for the EPA samples received.

12.7.1.1 The LCS for aqueous/water and soil/sediment samples shall be prepared by spiking an aliquot of reagent water (50-100 mL for aqueous/water, 1 mL for soil/sediment) such that the final digestate shall contain each analyte at two times the CRQL for the associated matrix. One LCS shall be prepared for each prepared batch of aqueous/water or soil/sediment samples in an SDG.

12.7.1.2 The LCS for the wipe samples shall be prepared by spiking a clean wipe provided by the sampler such that the final digestate shall contain each analyte at two times the CRQL. One wipe LCS shall be prepared and analyzed for every group of wipe samples in an SDG, or for each batch of wipe samples digested, whichever is more frequent. If a clean wipe is not provided by the sampler, prepare the LCS using reagent water and note this in the SDG Narrative.

12.7.1.3 The LCS for the air filter samples shall be prepared by spiking a clean air filter provided by the sampler such that the final digestate shall contain each analyte at two times the CRQL. One filter LCS shall be prepared and analyzed for every group of filter samples in an SDG, or for each batch of filter samples digested, whichever is more frequent. If a clean filter is not provided by the sampler, prepare the LCS using reagent water and note this in the SDG Narrative.

12.7.2 All LCS and percent recovery results shall be reported. If the percent recovery for the LCS for aqueous/water or soil/sediment samples falls outside the control limits of 70-130% (exception: Ag and Sb, control limits 50-150%), the analyses shall be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed with appropriate new QC.

12.7.3 If the results for the LCS for wipes or filters fall outside the control limits of 70-130%, the Contractor shall note this in the SDG Narrative, since wipe and filter samples are fully consumed by initial analysis and cannot be reprepared and reanalyzed.

12.8 ICP-AES Serial Dilution Analysis

12.8.1 Prior to reporting concentration data for the analytes, the Contractor shall analyze and report the results of the ICP-AES serial dilution analysis. The ICP-AES serial dilution analysis shall be performed on a sample from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment, wipe, filter) or for each SDG, whichever is more frequent. Samples identified as field blanks and PE samples shall not be used for serial dilution analysis.

12.8.2 If the analyte concentration is sufficiently high (minimally a factor of 50 above the MDL in the original sample), the serial dilution (a five-fold dilution) shall then agree within 10% of the original determination after correction for dilution. If the dilution analysis for one or more analytes is not within a control limit of 10%, a chemical or physical interference effect must be suspected, and the data for all affected analytes in the samples received and associated with that serial dilution must be flagged with an "E".

12.8.3 The percent differences for each component are calculated as follows:

EQ. 7 Serial Dilution Percent Differences

$$\%\text{Difference} = \frac{|SR - SDSR|}{SR} \times 100$$

WHERE, SR = Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2
SDSR = Serial Dilution Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2

12.8.4 In the instance where there is more than one serial dilution per SDG, if one serial dilution result is not within contract criteria, flag all the samples of the same matrix in the SDG. Serial dilution results and "E" flags shall be reported.

12.9 MDL Determination

12.9.1 Before any field samples are analyzed under this contract, the MDLs shall be determined for each digestion procedure and instrument used prior to the start of contract analyses and annually thereafter. The MDLs shall meet the levels specified in Exhibit C. An MDL study shall also be performed after major instrument maintenance, or changes in instrumentation or instrumental conditions, to verify the current sensitivity of the analysis.

12.9.1.1 To determine the MDLs, the Contractor shall run MDL studies following the procedures given in 40 CFR, Part 136. The Contractor shall prepare the MDL samples by each digestion procedure used and shall analyze these samples on each instrument used.

12.9.1.2 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C.

12.9.1.3 The results of the MDL determination studies shall be forwarded to the USEPA Regional CLP PO, Sample Management Office (SMO), and Quality Assurance Technical Support (QATS).

12.9.1.4 The MDL results shall be reported on Form IX-IN.

12.10 Interelement Corrections

12.10.1 Before any field samples are analyzed under this contract, the interelement correction factors shall be determined prior to the start of contract analyses and at least annually thereafter. Correction factors for spectral interference due to Al, Ca, Fe, and Mg shall be determined for all ICP-AES instruments at all wavelengths used for each analyte reported by ICP-AES. Interelement correction factors shall also be reported for any other elements (including those on the TAL) that have been determined to interfere with the requested target analyte(s).

NOTE: Depending on sample matrix and interferences, it may be necessary to analyze interelement correction factors at a frequency greater than annually and/or at multiple concentrations comparable to the sample interferent levels.

If the instrument was adjusted in any way that may affect the ICP-AES interelement correction factors, the factors shall be redetermined and the results submitted for use. In addition, all data used for the determination of the interelement correction factors shall be available to the USEPA during an on-site laboratory evaluation. Results from the interelement correction factors determination shall be reported for all ICP-AES analytes.

12.11 Example Analytical Sequence for ICP-AES Including the Initial Calibration

S0
S
S
S
S
S
ICV
ICB
ICSA
ICSAB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

12.12 Summary of QC Operations

The QC operations performed for ICP-AES analysis are summarized in the table below.

QC Operation	**Frequency**
Instrument Calibration	Each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment.
Initial Calibration Verification	Following each instrument calibration for each wavelength or mass used.
Initial Calibration Blank	Following each instrument calibration, immediately after the ICV.
Continuing Calibration Verification	For each wavelength or mass used, at a frequency of every 2 hours of a run, at the beginning of each day, and at the beginning and end of each run.
Continuing Calibration Blank	Every 2 hours of a run, at the beginning of each day, and at the beginning and end of each run. Performed immediately after the last CCV.
Interference Check Sample	At the beginning of each run.
Serial Dilution for ICP	For each matrix type or for each SDG, whichever is more frequent.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.

QC Operation	Frequency
Laboratory Control Sample	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Post-Digestion/Distillation Spike	Each time Spike Sample Recovery is outside QC limits.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
Determination of Method Detection Limits	Prior to contract award, annually thereafter, and after major instrument adjustment.
Interelement Corrections	Prior to contract award, annually thereafter, and after major instrument adjustment.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 1.15 in Exhibit D - Introduction to Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 1.16 in Exhibit D - Introduction to Analytical Methods.

16.0 REFERENCES

16.1 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Draft Method 200.7, Revision 5 (2001).

16.2 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3050B, Revision 2. Third Edition, Update III. December 1996.

16.3 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3015A, Revision 1, Update IVa. January 1998.

16.4 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3051A, Revision 1, Update IVa. January 1998.

16.5 National Institute for Occupational Safety and Health (NIOSH). NIOSH Manual of Analytical Methods, Fourth Edition. Method 7300, Issue 3, March 2003.

16.6 US Government Printing Office. 40 Code of Federal Regulations, Part 136. Section 1. Appendix B.

16.7 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical methods (SW-846). Test Method 6010C, Revision 3, February 2007.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1: Interferent and Analyte Elemental Concentrations Used for ICP-AES Interference Check Sample (ICS)

Analytes	**(µg/L)**	**Interferents**	**(µg/L)**
Ag	200	Al	250000
As	100	Ca	250000
Ba	500	Fe	100000
Be	500	Mg	250000
Cd	1000		
Co	500		
Cr	500		
Cu	500		
Mn	500		
Ni	1000		
Pb	50		
Sb	600		
Se	50		
Tl	100		
V	500		
Zn	1000		

NOTE: ICS Solution A (ICSA) contains the interferents at the indicated concentrations. The ICSA may be analyzed at twice the concentration indicated when interferences are present at higher concentrations in the sample. ICS Solution AB (ICSAB) contains all of the analytes and interferents listed above at the indicated concentrations. The ICS solutions are normally provided by USEPA along with the established concentrations for each of the analytes and interferents.

TABLE 2: Spiking Levels for Spike Sample Analysis

Analyte	Water (µg/L)	Soil[1] (mg/kg)	Analyte	Water (µg/L)	Soil[1] (mg/kg)
Aluminum	2000	*	Magnesium	*	*
Antimony	100	20	Manganese	500	100
Arsenic	40	8	Nickel	500	100
Barium	2000	400	Potassium	*	*
Beryllium	50	10	Selenium	50	10
Cadmium	50	10	Silver	50	10
Calcium	*	*	Sodium	*	*
Chromium	200	40	Thallium	50	10
Cobalt	500	100	Vanadium	500	100
Copper	250	50	Zinc	500	100
Iron	1000	*			
Lead	20	4			

[*] No spike required. NOTE: Analytes without spike levels, and not designated with an asterisk, shall be spiked at appropriate levels.

[1] The levels shown indicate concentrations in the spike sample when the dry weight of 1 gram of sample is taken for analysis. Adjustment shall be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values. Appropriate adjustments shall be made for the alternative microwave digestion procedures where 0.5 grams of sample or 50 mL (45 mL of sample plus 5 mL of acid) of aqueous/water sample are required for analysis.

EQ. 8 Spiking Level Adjustment

$$\text{mg/kg} = \mu\text{g/L} \times \frac{\text{final volume (L)}}{\text{sample weight (g)}}$$

18.0 ALTERNATIVE SAMPLE PREPARATION PROCEDURES

18.1 Regionally-Selected Alternative Sample Preparation Procedures

Due to the variability of the matrices received and the interferences encountered, the sample preparation procedures described in Sections 10.1.4 through 10.1.7 may not always produce valid and/or reliable results. Sample preparation procedures are given in this section that describe alternative procedures that can be used if interferences or other issues are noted with the primary procedures. If the Contractor feels that one of these alternative sample preparation procedures would provide better results than the primary methods, then the Contractor shall contact the Sample Management Office (SMO). SMO will contact the USEPA Region for instructions. The Contractor shall document the USEPA Region's decision in the Sample Delivery Group (SDG) Narrative along with populating the Forms and the electronic data deliverable with the valid values that described the actual sample preparation procedures used by the Contractor.

18.2 Alternative Aqueous/Water Sample Preparation Procedures

18.2.1 Preparation Method 3015A – Nitric Acid Digestion (based on USEPA OSW Method 3015A, Revision 1, January 1998)

18.2.1.1 This method describes a sample preparation procedure for the determination of total analytes in various aqueous/water matrices using a microwave technique with a nitric acid only digestion.

18.2.1.2 Measure a 45.0 mL (±0.1 mL) aliquot of a well-shaken, homogenized sample using an appropriate volumetric delivery device, and quantitatively transfer the aliquot to an appropriate vessel equipped with a controlled pressure relief mechanism. The sample shall not be diluted prior to digestion.

18.2.1.3 Add 5.0 mL (±0.1 mL) of concentrated nitric acid to the digestion vessel.

18.2.1.4 Seal the digestion vessel according to the manufacturer's directions. Properly place the digestion vessels in the microwave system according to the manufacturer's recommended specifications and, when applicable, connect appropriate temperature and pressure monitoring equipment to the digestion vessels according to the manufacturer's specifications.

18.2.1.5 The Preparation Blank must have 45.0 mL of reagent water and the same amount (5.0 mL) of acid that was added to the samples.

18.2.1.6 The microwave program is then started. The temperature of each sample should rise to 170°C (±5°C) in approximately 10 minutes and remain at 170°C (±5°C) for 10 minutes, or for the remainder of the 20-minute digestion period. When using temperature feedback control, the number of samples that may be simultaneously digested may vary, from one sample up to the maximum number of digestion vessels that can be heated by the magnetron of the microwave unit. Whenever fewer than the recommended number of samples are to be digested, the remaining digestion vessels should be filled with 45.0 mL of water and 5.0 mL of acid so that the full complement of vessels is achieved. This provides an energy balance, since the microwave power absorbed is proportional to the total absorbing mass in the cavity.

18.2.1.7 At the end of the microwave program, allow the digestion vessels to cool for a minimum of 5 minutes before removing them from the microwave system. When the vessels have cooled to near room temperature, determine if the digestion vessels have maintained their seal throughout the digestion. For vessels that are sealed as discrete separate entities, the digestion vessel weight may be taken before and after digestion to evaluate seal integrity. If the weight loss of the sample exceeds 1% of the weight of the sample and reagents, then the sample is considered compromised and the sample must be redigested. For digestion vessels with burst disks, a careful visual inspection of the disk, in addition to weighing, may identify compromised digestion vessels.

18.2.1.8 Complete the preparation of the sample by carefully uncapping and venting each digestion vessel in a fume hood. Quantitatively transfer the sample to an acid-cleaned bottle. If the digested sample contains particulates which may clog nebulizers or interfere with the injection of the sample into the instrument, the sample may be centrifuged, allowed to settle, or filtered through Whatman No. 41 (or equivalent) filter paper.

18.2.1.9 The final solution typically requires nitric acid to maintain appropriate sample solution acidity and stability of the elements. Commonly, a 2% (v/v) nitric acid concentration is desirable.

18.2.1.10 Transfer or decant the digested sample into a 50 mL volumetric flask and dilute to volume. The samples are now ready for analysis. Concentrations so determined shall be reported as "Total".

18.2.1.11 The digested sample can be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

18.2.2 Preparation Method 3015A – Nitric Acid and Hydrochloric Acid Digestion (based on USEPA OSW Method 3015A, Revision 1, January 1998)

18.2.2.1 This method describes a sample preparation procedure for the determination of total analytes in various aqueous/water matrices using a microwave technique with a nitric acid and hydrochloric acid digestion. The addition of the concentrated hydrochloric acid to the nitric acid is appropriate for the stabilization of certain analytes (such as silver, barium, and antimony) and high concentrations of iron and aluminum in solution. The addition of hydrochloric acid may, however, limit the detection techniques or increase the difficulties of analysis for some detection systems.

18.2.2.2 Measure a 45.0 mL (±0.1 mL) aliquot of a well-shaken, homogenized sample using an appropriate volumetric measurement device, and quantitatively transfer the aliquot to an appropriate vessel equipped with a controlled pressure relief mechanism. The sample shall not be diluted prior to digestion.

18.2.2.3 Add 4.0 mL (±0.1 mL) of concentrated nitric acid and 1.0 mL (±0.1 mL) of concentrated hydrochloric acid to the digestion vessel.

18.2.2.4 The analyst should be aware of the potential for a vigorous reaction, especially with samples containing suspended solids composed of volatile or easily oxidized organic species. When digesting a matrix of this type, if a vigorous reaction occurs upon the addition of reagent(s), this sample represents a safety

hazard. Do not leach the sample as described in this method due to the high potential for unsafe and uncontrollable reactions.

18.2.2.5 Follow the procedure as described in Sections 18.2.1.4 through 18.2.1.11.

18.3 Alternative Soil/Sediment Sample Preparation Procedures

18.3.1 Preparation Method 3050B – Optional Procedure (based on USEPA OSW Method 3050B, Revision 2, December 1996)

18.3.1.1 This method describes an optional sample preparation procedure that may be used to improve the solubilities and recoveries of antimony, barium, lead, and silver when necessary. This procedure should not be required on a routine basis.

18.3.1.2 Mix the sample thoroughly to achieve homogeneity (See Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh to the nearest 0.01 g and transfer 1.0 – 1.5 g sample (wet weight) to an appropriately sized digestion vessel (e.g., a beaker or hot block digestion tube).

18.3.1.3 Add 2.5 mL of concentrated nitric acid and 10 mL of concentrated hydrochloric acid and cover with a watch glass or vapor recovery device. Heat the sample to 95°C (±3°C) and reflux for 15 minutes without boiling.

18.3.1.4 Filter the digestate through Whatman No. 41 filter paper (or equivalent) and collect the filtrate in a 100-mL volumetric flask. Wash the filter paper, while still in the funnel, with no more than 5 mL of hot (approximately 95°C) concentrated hydrochloric acid, and then with 20 mL of hot (approximately 95°C) reagent water. Collect the washings in the same 100-mL volumetric flask.

18.3.1.5 Remove the filter and residue from the funnel and place them back in the digestion vessel. Add 5 mL of concentrated hydrochloric acid, place the digestion vessel back on the heating source, and heat at 95°C (±3°C) until the filter paper dissolves. Remove the digestion vessel from the heating source and wash the cover and sides with reagent water. Filter the residue and collect the filtrate in the same 100 mL volumetric flask. Allow the filtrate to cool and then dilute to volume. NOTE: High concentrations of metal salts with temperature-sensitive solubilities can result in the formation of precipitates upon cooling of primary and/or secondary filtrates. If precipitation occurs in the flask upon cooling, <u>do not</u> dilute to volume.

18.3.1.6 If a precipitate forms on the bottom of the flask, add up to 10 mL of concentrated hydrochloric acid to dissolve the precipitate. After the precipitate is dissolved, dilute to volume with reagent water, stopper, and mix. The sample is now ready for analysis.

18.3.1.7 The digested sample can be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

18.3.2 Preparation Method 3051A – Nitric Acid Digestion (based on USEPA OSW Method 3051A, Revision 1, January 1998)

18.3.2.1 This method describes a sample preparation procedure for the determination of total recoverable analytes in various soil/sediment matrices using a microwave technique with a nitric acid only digestion.

18.3.2.2 Mix the sample thoroughly to achieve homogeneity (See Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh to the nearest 0.001 g and transfer a 0.50 g sample (wet weight) to an appropriate digestion vessel equipped with a controlled pressure relief mechanism.

18.3.2.3 Add 10 mL (±0.1 mL) of concentrated nitric acid to the digestion vessel in a fume hood.

18.3.2.4 The analyst should be aware of the potential for a vigorous reaction especially with samples containing volatile or easily oxidized organic species. When digesting a matrix of this type, initially use no more than 0.10 g (±0.001 g) of sample. If a vigorous reaction occurs upon the addition of the acid(s), allow the sample to predigest in the uncapped digestion vessel until the reaction ceases. Heat may be added in this step for safety considerations (e.g., the rapid release of carbon dioxide from carbonates, easily oxidized organic matter, etc.). Once the initial reaction has ceased, the sample may continue through the digestion procedure.

18.3.2.5 Seal the digestion vessel according to the manufacturer's directions. Properly place the digestion vessels in the microwave system according to the manufacturer's recommended specifications and, when applicable, connect appropriate temperature and pressure monitoring equipment to the digestion vessels according to the manufacturer's specifications.

18.3.2.6 The Preparation Blank must have 0.50 g (±0.001 g) of reagent water and the same amount of acid(s) that was added to the samples.

18.3.2.7 The microwave program is then started. The temperature of each sample should rise to 175°C (±5°C) in approximately 5.5 (±0.25) minutes and remain at 175°C (±5°C) for 4.5 minutes, or for the remainder of the 10-minute digestion period. When using temperature feedback control, the number of samples that may be simultaneously digested may vary, from one sample up to the maximum number of digestion vessels that can be heated by the magnetron of the microwave unit. Whenever fewer than the recommended number of samples are to be digested, the remaining digestion vessels should be filled with 0.50 g of water and the same amount of acid(s) so that the full complement of vessels is achieved. This provides an energy balance, since the microwave power absorbed is proportional to the total absorbing mass in the cavity.

18.3.2.8 At the end of the microwave program, allow the digestion vessels to cool for a minimum of 5 minutes before removing them from the microwave system. When the vessels have cooled to near room temperature, determine if the digestion vessels have maintained their seal throughout the digestion. For vessels that are sealed as discrete separate entities, the digestion vessel weight may be taken before and after digestion to evaluate seal integrity. If the weight loss of the sample exceeds 1% of the weight of the sample and reagents, then the sample is considered compromised and the sample must be redigested. For digestion vessels with burst disks, a careful visual inspection of the disk, in addition to weighing, may identify compromised digestion vessels.

18.3.2.9 Complete the preparation of the sample by carefully uncapping and venting each digestion vessel in a fume hood. Quantitatively transfer the sample to an acid-cleaned bottle. Filter the digested sample through Whatman No. 41 (or equivalent) filter paper to remove particulates which may clog nebulizers or otherwise interfere with the injection of the digested sample into the instrument.

18.3.2.10 The final solution typically requires nitric acid to maintain appropriate sample solution acidity and stability of the elements. Commonly, a 2% (v/v) nitric acid concentration is desirable.

18.3.2.11 Transfer or decant the digested sample into a 50 mL volumetric flask and dilute to volume. The samples are now ready for analysis.

18.3.2.12 The digested sample can be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

18.3.3 Preparation Method 3051A – Nitric Acid and Hydrochloric Acid Digestion (based on USEPA OSW Method 3051A, Revision 1, January 1998)

18.3.3.1 This method describes a sample preparation procedure for the determination of total recoverable analytes in various soil/sediment matrices using a microwave technique with a nitric acid and hydrochloric acid digestion. The addition of the concentrated hydrochloric acid to the nitric acid is appropriate for the stabilization of certain analytes (such as silver, barium, and antimony) and high concentrations of iron and aluminum in solution. Improvements are also noted with the recoveries of antimony, iron, and silver. The addition of hydrochloric acid may, however, limit the detection techniques or increase the difficulties of analysis for some detection systems.

18.3.3.2 Mix the sample thoroughly to achieve homogeneity (See Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh to the nearest 0.001 g and transfer a 0.50 g sample (wet weight) to an appropriate digestion vessel equipped with a controlled pressure relief mechanism.

18.3.3.3 Add 9 mL (±0.1 mL) of concentrated nitric acid and 3 mL (±0.1 mL) of concentrated hydrochloric acid to the digestion vessel in a fume hood.

18.3.3.4 Follow the procedure as described in Sections 18.3.2.4 through 18.3.2.12.

18.4 Microwave Calibration Procedure

18.4.1 The calibration procedure is a critical step prior to the use of any microwave unit. The microwave unit must be calibrated every 6 months. The data for each calibration must be available for review during on-site audits. In order that absolute power settings may be interchanged from one microwave unit to another, the actual delivered power must be determined.

18.4.2 Calibration of a laboratory microwave unit depends on the type of electronic system used by the manufacturer. If the unit has a precise and accurate linear relationship between the output power and the scale used in controlling the microwave unit, then the calibration can be a two-point calibration at maximum and 40% power. If the unit is not accurate or precise for some portion of the controlling scale, then a multiple-point calibration is necessary. If the unit power calibration needs a multiple-point calibration, then the point where linearity begins must be identified. For example: a calibration at 100, 99, 98, 97, 95, 90, 80, 70, 60, 50, and 40% power settings can be applied and the data plotted. The non-linear portion of the calibration curve can be excluded or restricted in use. Each percent is equivalent to approximately 5.5-6 watts and becomes the smallest unit of power that can be controlled. If 20-40 watts are contained from 99-100%, that portion of the microwave calibration is not controllable by 3-7 times that of the linear portion of the control scale and will prevent duplication of precise power conditions specified in that portion of the power scale.

18.4.3 The power available for heating is evaluated so that the absolute power setting (watts) may be compared from one microwave to another. This is accomplished by measuring the temperature rise in 1 kilogram (kg) of water exposed to microwave radiation for a fixed period of time. The water is placed in a polytetrafluoroethylene (PTFE) beaker (or a beaker that is made of some other material that does not absorb microwave energy) and stirred before measuring the temperature. Glass beakers absorb microwave energy and may not be used. The initial temperature of the water must be between 19°C and 25°C. The beaker is circulated continuously through the field for at least 2 minutes at full power. The beaker is removed from the microwave, the water is stirred vigorously, and the final temperature is recorded. The final reading is the maximum temperature reading after each energy exposure. These measurements must be accurate to ±0.1°C and made within 30 seconds of the end of heating. If more measurements are needed, do not use the same water until it has cooled down to room temperature. Otherwise, use a fresh water sample.

The absorbed power is determined by the following formula:

EQ. 9 Absorbed Power

$$P = \frac{(K)(C_p)(m)(DT)}{t}$$

WHERE,
- P = The apparent power absorbed by the sample in watts (joules per second)
- K = The conversion factor for thermochemical calories per second to watts (4.184)
- C_p = The heat capacity, thermal capacity, or specific heat (cal. g^{-1} $°C^{-1}$) of water (1.0)
- m = The mass of the sample in grams (g)
- DT = The final temperature minus the initial temperature (°C)
- t = The time in seconds (s)

Using 2 minutes and 1 kg of reagent water, the calibration equation simplifies to:

$$P = (DT)(34.87)$$

The microwave user can now relate power in watts to the percent power setting of the microwave.

18.5 Microwave Digestion Cleaning Procedure

18.5.1 Initial Cleaning of the Polytetrafluoroethylene Perfluoroalkoxy (PTFE PFA) Digestion Vessels

18.5.1.1 Prior to first use - new vessels must be annealed before they are used. A pretreatment/cleaning procedure must be followed. This procedure calls for heating the vessels for 96 hours at 200°C. The vessels must be disassembled during annealing and the sealing surfaces (the top of the vessel or its rim) must not be used to support the vessel during annealing.

18.5.1.2 Rinse in reagent water.

18.5.1.3 Immerse in 50% (v/v) hydrochloric acid for a minimum of 3 hours after the cleaning bath has reached a temperature just below boiling.

18.5.1.4 Rinse in reagent water.

18.5.1.5 Immerse in 50% (v/v) nitric acid for a minimum of 3 hours after the cleaning bath has reached a temperature just below boiling.

18.5.1.6 The vessels are then rinsed with copious amounts of reagent water prior to use for any analyses under this contract.

18.5.2 Cleaning Procedure between Sample Digestions

18.5.2.1 Wash entire vessel in hot water using laboratory-grade non-phosphate detergent.

18.5.2.2 Rinse with 50% (v/v) nitric acid.

18.5.2.3 Rinse 3 times with reagent water.

EXHIBIT D - PART B

ANALYTICAL METHODS
FOR
INDUCTIVELY COUPLED PLASMA -
MASS SPECTROMETRY

Exhibit D – Analytical Methods for ICP-MS

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1.0 SCOPE AND APPLICATION

This method provides procedures for the use of Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) to determine the concentration of total recoverable and dissolved elements in aqueous/water and soil/sediment samples taken from hazardous waste sites. This method is applicable to all metals in the Target Analyte List (TAL) for ICP-MS in Exhibit C - Inorganic Target Analyte List with Contract Required Quantitation Limits.

2.0 SUMMARY OF METHOD

2.1 General Method Overview

This method describes the multi-element determination of trace elements by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS). Sample material in solution is introduced by nebulization into a radio-frequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio. The separated ions are detected and the ion information processed by a data handling system. Interferences related to the technique must be recognized and corrected. Such corrections may include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from plasma gas, reagents, or sample matrix. Instrumental drift, as well as suppressions or enhancements of instrument response, must be corrected for with the use of internal standards.

2.2 Summary of Digestion Procedures

2.2.1 Hotplate Acid Digestion of Aqueous/Water Samples (based on USEPA 200.8)

2.2.2 Hotplate Acid Digestion of Soil/Sediment Samples (based on USEPA 3050B)

2.2.3 Alternative digestion procedures are included in Section 18 of this Exhibit.

3.0 DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

Several types of interferences may cause inaccuracies in the determination of trace elements by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS). To prevent this, appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. Possible interferences are given in Sections 4.1 through 4.5.

4.1 Isobaric Elemental Interferences

Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio, and which cannot be resolved by the mass spectrometer. All elements determined by this method have, at minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method, only selenium-82 has an isobaric elemental interference (krypton-82). If alternative analytical isotopes having higher natural abundances are selected, in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process shall be included with the data. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.

4.2 Abundance Sensitivity

Abundance sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. Abundance sensitivity is affected by ion energy and mass filter operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution should be adjusted to minimize them.

4.3 Isobaric Polyatomic Ion Interferences

Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified and are listed in Table 2 - Isobaric Molecular-Ion Interferences, with the target analytes affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence, since polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions.

4.4 Physical Interferences

Physical interferences are associated with the physical processes which govern the transport of the sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during the excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute to deposits of material on the extraction and/or skimmer cones. Deposits can reduce the effective diameter of the orifices and therefore ion transmission. Dissolved solid levels not exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects. Internal standards ideally should have similar analytical behavior to the elements being determined.

4.5 Memory Interferences

Memory interferences result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects, or carryover, can result from sample deposition on the sampler and skimmer cones, as well as from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples (see Section 7.3.3). The possibility of memory interferences should be recognized within an analytical run and suitable rinse times or monitoring should be used to reduce them. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if it was high. If a memory interference is suspected, the sample should be reanalyzed after a rinse period.

5.0 SAFETY

See Section 1.14 in Exhibit D - Introduction to Analytical Methods.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 Glassware/Labware

6.1.1 250 mL beaker or other appropriate vessel (glass or plastic)

6.1.2 Watch glasses (glass or plastic)

6.1.3 Funnels

6.1.4 Graduated cylinders

6.1.5 Various volumetric flasks and pipets (Class A)

6.1.6 Thermometer that covers range of 0-200°C

6.1.7 Whatman No. 41 filter paper (or equivalent)

6.1.8 Hotplate, block digester, or other heating source capable of maintaining 95°C (±3°C).

6.1.9 Balances – Top loader balance, 300 gram (g) capacity, and a minimum sensitivity of ±1.0 milligram (mg)

6.2 Equipment and Supplies for Microwave Digestion

The equipment and supplies listed in this section are used if one of the alternate microwave sample preparation procedures was approved for use by the USEPA Region.

6.2.1 Whatman No. 41 filter paper (or equivalent)

6.2.2 Disposable polypropylene filter funnel

6.2.3 Polyethylene bottles, 125 mL, with caps

6.2.4 Microwave oven with programmable power settings up to at least 600 watts.

6.2.5 The system must use polytetrafluoroethylene perfluoroalkoxy (PTFE PFA) digestion vessels (120 mL capacity) capable of withstanding pressure of up to 110 (±10) psi [7.5 (±0.7) atm]. These vessels are capable of controlled pressure relief at pressures exceeding 110 psi.

6.2.6 A rotating turntable must be used to ensure homogeneous distribution of microwave radiation within the oven. The speed of the turntable must be a minimum of 3 revolutions per minute (rpm).

6.3 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

6.3.1 The ICP-MS consists of:

- An instrument capable of scanning the mass range 5-250 atomic mass units (amu) with a minimum resolution capability of 1 amu peak width at 5% peak height and either a conventional or extended dynamic range detector.
- A radio-frequency generator compliant with Federal Communications Commission (FCC) regulations.
- A high purity (99.99%) argon gas supply.
- A variable speed peristaltic pump to deliver sample solution to the nebulizer.
- A mass-flow controller on the nebulizer gas supply is required.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagents may contain elemental impurities that might affect the integrity of analytical data. Owing to the high sensitivity of Inductively Coupled Plasma - Mass Spectrometry (ICP-MS), high-purity reagents should be used whenever possible. Suitable acids are available from a number of manufacturers or may be prepared by sub-boiling distillation. Nitric acid is preferred for ICP-MS in order to minimize polyatomic ion interferences. Several polyatomic ion interferences result when hydrochloric acid is used; however, it should be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and silver. When hydrochloric acid is used, corrections for the chloride polyatomic ion interferences must be applied to all data.

7.1.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-06). Use this water for all reagents, standards, and dilutions.

7.1.2 Nitric acid - Concentrated (specific gravity 1.41).

7.1.3 Nitric acid (50% v/v) - Add 500 mL conc. nitric acid (specific gravity 1.41) to 400 mL of reagent water and dilute to 1 L.

7.1.4 Nitric acid (10% v/v) - Add 100 mL conc. nitric acid (specific gravity 1.41) to 400 mL of reagent water and dilute to 1 L.

7.1.5 Nitric acid (2% v/v) – Add 20 mL conc. nitric acid (specific gravity 1.41) to 400 mL of reagent water and dilute to 1 L.

7.1.6 Nitric acid (1% v/v) – Add 10 ml conc. nitric acid (specific gravity 1.41) to 400 mL of reagent water and dilute to 1 L.

7.1.7 Hydrochloric acid - Concentrated (specific gravity 1.19).

7.1.8 Hydrochloric acid (50% v/v) - Add 500 mL conc. hydrochloric acid (specific gravity 1.19) to 400 mL of reagent water and dilute to 1 L.

7.1.9 Hydrochloric acid (HCl) (20% v/v) - Add 200 mL conc. hydrochloric acid (specific gravity 1.19) to 400 mL reagent water and dilute to 1 L.

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards, except as noted, to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit E, Section 8.0. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

7.2.2 Stock Standard Solutions

7.2.2.1 Stock standard solutions may be purchased from a reputable source or prepared from reagent grade chemicals or metals (at least 99.99% pure). All salts must be dried for 1 hour at 105°C unless otherwise specified. Stock solutions should be stored in Fluorinated Ethylene Propylene (FEP) fluorocarbon bottles. Note that some metals, particularly those which form surface oxides, require cleaning prior to being weighed. This may be achieved by pickling the surface of the metal in acid. An amount in excess of the desired weight should be pickled repeatedly, rinsed with reagent water, dried, and weighed until the desired weight is achieved.

7.2.3 Secondary Dilution Standards

Prepare mixed secondary dilution standard solutions by diluting the appropriate volumes of stock standards with 1% (v/v) nitric acid, or as recommended by the manufacturer, to obtain the final volume. Originating stock standards should be checked for the presence of impurities which might influence the accuracy of the standard. Freshly prepared standards should be transferred to acid-cleaned, not previously used, FEP fluorocarbon bottles for storage and monitored periodically for stability. Mixed secondary dilution standard solutions may be purchased. The purchased standards shall meet the requirements in Section 7.2.1.

7.2.4 Working Standards

7.2.4.1 Mixed Calibration Standard Solutions

Care must be taken in the preparation of mixed calibration standards to ensure that the analytes are compatible and stable. Fresh calibration standards should be prepared from mixed standard solutions every 2 weeks or less. Prepare the mixed standards to levels appropriate to the operating range of the instrument using 1% (v/v) nitric acid or to match the matrix of the digested samples. The analyte concentrations in the calibration standards should be sufficient to produce good measurement precision and to accurately define the slope of the response curve. Protect all standards from light. Samples, sample digestates, and standards must be stored separately.

7.2.4.2 Internal Standard Solution

The internal standard solution is to be added to all digested samples, blanks, and standards by the analyst prior to analysis, or it can be added automatically by the instrument during analysis of all digested samples, blanks, and standards. Prepare the mixed internal standard solution by following the manufacturer's guidelines.

7.2.4.3 Tuning Solution

This solution is used for instrument tuning and mass calibration prior to analysis. Prepare a mixed standard by diluting beryllium, magnesium, cobalt, indium, and lead stock standards to 100 µg/L with 1% (v/v) nitric acid. The concentration of this solution can be reduced based on recommendations from the instrument manufacturer.

7.2.4.4 Interference Check Sample (ICS) Solution

The ICS consists of two solutions: Solution A (ICSA) and Solution AB (ICSAB). ICSA consists of the interferents and ICSAB consists of the analytes mixed with the interferents. The ICS standards (ICSA for the interferents only and ICSB for the analytes only) are normally supplied by USEPA.

7.3 Blanks

Three types of blanks are required for this method. A Calibration Blank is used to establish the analytical calibration curve, the Preparation Blank (see Section 12.4.2) is used to assess possible contamination from the sample preparation procedure and to assess spectral background, and the Rinse Blank is used to flush the instrument between samples to reduce memory interferences.

7.3.1 Calibration Blank - Consists of 1% (v/v) nitric acid in reagent water or matrix matched to the digested samples. The Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) are identical in composition to the Calibration Blank and are used to monitor for analyte carryover.

7.3.2 Preparation Blank - Must contain all the reagents in the same volumes as used in preparing the samples. The Preparation Blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Soil/sediment blanks shall use 1.00 g (±0.01 g) of reagent water.

7.3.3 Rinse Blank – Must contain sufficient nitric acid to allow the instrument to return to baseline between the analysis of digested samples, blanks, and standards. The Rinse Blank would typically consist of 2% (v/v) nitric acid in reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in glass or polyethylene containers. Aqueous/water samples must be preserved with nitric acid to pH less than or equal to 2 immediately after collection. All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until digestion. If aqueous/water samples are received in glass containers, please note this in the Sample Delivery Group (SDG) Narrative.

8.1.1 Dissolved Metals

For the determination of dissolved metals, the sample must be filtered through a 0.45 micrometer (µm) pore diameter membrane filter at the time of collection or as soon as possible. Use a portion of the sample to rinse the filter flask, discard this portion, and collect the required volume of filtrate. Preserve the filtrate with nitric acid to pH less than or equal to 2 immediately after filtration.

8.2 Procedures for Sample Storage and Disposal

Following digestion, the remaining unused portion of aqueous/water and soil/sediment samples may be stored at room temperature within the laboratory until 60 days after delivery of a complete, reconciled data package to USEPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.3 Procedure for Sample Digestate Storage

Sample digestates must be stored until 6 months after delivery of a complete, reconciled data package to USEPA.

8.4 Contract Required Holding Time

The maximum holding time for metals is 180 days from Validated Time of Sample Receipt (VTSR).

9.0 CALIBRATION AND STANDARDIZATION

9.1 Instrument Operating Parameters

Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. The Method Detection Limit (MDL), precision, and interference effects must be investigated and established for each individual analyte on that particular instrument. All measurements must be within the operational range of the instrument where corrections are valid. It is the responsibility of the analyst to verify the instrument configuration and operating conditions used to satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2 Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) Instrument Calibration Procedure

9.2.1 Precalibration routine - The following precalibration routine must be completed prior to calibrating the instrument.

Set up the instrument with proper operating parameters established in Section 9.1. The instrument must be allowed to become stable prior to calibration. Conduct any necessary mass calibration and resolution routines to bring peak width within the manufacturer's specifications and adjust mass calibration to within 0.1 amu over the range of 6 to 210 amu.

Demonstrate instrument stability and precision by analyzing the tuning solution as a single analysis with at least five integrations. The percent relative standard deviation of the absolute signals for all the multiple integrations in the tuning solution, as calculated by the instrument, must be less than 5.0% for each analyte.

9.2.2 Internal Standardization

Internal standardization must be used in all analyses to correct the instrument drift and physical interferences. A list of acceptable internal standards is provided in Table 5 - Internal Standards. For full range mass scans, a minimum of five internal standards shall be used. The internal standards selected for a run must be consistent throughout the entire run. Internal standards shall be present in all samples, standards, and blanks (except the tuning solution) at identical levels. This may be achieved by directly adding an aliquot of the internal standard solution to each sample, standard, and blank, or by mixing with the sample solution prior to nebulization using a second channel of the peristaltic pump and mixing coil. The concentration of the internal standard should be sufficiently high for good precision and to minimize the possibility of correction errors if the internal standard is naturally present in the sample. Depending on the sensitivity of the instrument, a final concentration range of 20 µg/L to 200 µg/L of each internal standard in each digested sample, blank, and standard is recommended when the internal standards are added manually by the analyst. If dilutions are performed on the digested samples, then the internal standards must be added after the dilution. If the internal standards are added automatically by the instrument prior to analysis, then the manufacturer's guidelines for the appropriate concentration ranges should be followed.

9.2.3 Calibration

Instruments shall be calibrated each time the instrument is set up, or after Initial Calibration Verification (ICV) or Continuing Calibration Verification (CCV) failure. The instrument standardization date and time shall be included in the raw data. Calibration standards shall be prepared as in Section 7.2.4.1. Calibrate the instrument with at least six standards, one of which shall be a blank standard and one shall be at or below the Contract Required Quantitation Limit (CRQL). The rest of the standards shall be uniformly spread out in graduated amounts over the appropriate calibration range for each metal. A minimum of three replicate integrations are required for data acquisition. Use the average of all the integrations for instrument calibration and data reporting. The calibration curve shall be calculated using linear regression by plotting the concentration of the standard (in µg/L) on the X-axis

versus the corrected instrument response on the Y-axis. The corrected instrument response are those corrections (e.g., such as correction for background, internal standards, Interelement Corrections (IECs), calibration blank) that may be applied to the raw uncorrected instrument response prior to determining the calibration curve. A standard linear regression, a weighted linear regression (e.g., 1/concentration or 1/concentration^2), or a linear regression with zero force calibration model can be used as appropriate. No other types of equations (e.g., quadratic) are to be used. The acceptance criteria for the calibration curve is a correlation coefficient greater than or equal to 0.995. Sample analysis shall not begin until this criteria and the criteria described in Sections 9.2.4 and 9.2.5 have been met.

9.2.4 The calibration equation must be checked to establish the representativeness of the data that were used to produce the calibration equation. This check involves the re-fitting of the non-blank calibration data back to the calibration equation or the comparison of the calculated concentration of each of the standards against the expected concentration of the associated standard. This difference is related to the actual residual. For this calculation, the Percent Difference shall be used where the above difference is further divided by the expected amount or concentration of the respective standard. If these Percent Differences for each of the standards do not fall within ±30%, then the calibration equation is not acceptable and must be corrected. If a standard is analyzed for a particular analyte at a concentration that is below the CRQL and the above criteria is not met, that value can be excluded from the calibration curve as long as the lowest non-zero standard for each analyte is still analyzed at or below the CRQL and all standards included in the calibration curve are continuous and consecutive.

9.2.5 The y-intercept from the linear regression initial calibration equation shall also be evaluated. If the y-intercept is not below the CRQL for each of the analyte initial calibration curves, the calibration is not acceptable and must be corrected. Samples are not to be analyzed until the y-intercept for each analyte’s initial calibration curve meets the acceptance criteria.

9.2.6 Any changes or corrections to the analytical system shall be followed by recalibration.

9.3 Initial Calibration Verification (ICV)

9.3.1 Immediately after each instrument has been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of the ICV solution(s) for each mass used to report final results. The percent Relative Standard Deviation (%RSD) shall also be calculated from all replicate integrations and reported for each mass used to report final results.

9.3.2 The ICV solution(s) is (are) obtained from USEPA. If the solution(s) is (are) not available, then the ICV solution can be prepared by the laboratory using a certified solution for each analyte from an independent source. Analyses shall be conducted at a concentration other than that used for instrument calibration, but near the middle of the calibration range. An independent source is defined as a standard composed of the analytes from a different source other than those used in the standards for instrument calibration.

9.3.3 The ICV solution(s) shall be run at each mass used for reporting final results.

9.4 Continuing Calibration Verification (CCV)

9.4.1 To ensure calibration accuracy during each analytical run sequence, a CCV shall be analyzed and reported for each mass used for reporting final results for each analyte, at a frequency not to exceed every 2 hours during an analytical run sequence. The standard shall be analyzed and reported for each mass used for reporting final results for each analyte at the beginning of the analytical run sequence and after the last analytical sample. The %RSD shall be calculated from all integrations and reported for each mass used to report final results. See the example sample analysis sequence in Section 12.12. This analytical run sequence can continue indefinitely as long as samples are being continuously analyzed without the instrument being turned off and successive CCVs and their associated %RSDs meet the stated criteria.

This CCV standard(s) shall be prepared from the same source and at or near the mid-level concentration as used during the initial calibration.

The same CCV standard shall be used throughout the analytical run sequences for a Sample Delivery Group (SDG) of samples received.

9.4.2 Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding analytical samples up to the previous CCV as applicable and the analytical samples following this CCV up to the next CCV as applicable).

9.5 Initial and Continuing Calibration Blank (ICB/CCB)

An ICB or CCB shall be analyzed for each mass used for reporting final results for each analyte immediately after every ICV and CCV.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 If insufficient sample amount (less than 90% of the required amount) is received to perform the analyses, the Contractor shall contact the Sample Management Office (SMO) to inform them of the problem. SMO will contact the USEPA Region for instructions. The USEPA Region will either require that no sample analysis be performed or will require that a reduced volume be used for the sample analysis. No other changes in the analysis will be permitted. The Contractor shall document the USEPA Region’s decision in the Sample Delivery Group (SDG) Narrative.

10.1.2 If multiphase samples (e.g., two-phase liquid sample, oily sludge/sandy soil/sediment sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the USEPA Region. If all phases of the sample are amenable to analysis, the USEPA Region may require the Contractor to do any of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.
- Separate the phases of the sample and analyze one or more of the phases, separately. SMO will provide EPA sample numbers for the additional phases, if required.

- Do not analyze the sample.

10.1.2.1 If all of the phases are not amenable to analysis (i.e., outside scope), the USEPA Region may require the Contractor to do any of the following:

- Separate the phases and analyze the phase(s) that is (are) amenable to analysis. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.2 No other changes in the analyses will be permitted. The Contractor shall document the USEPA Region's decision in the SDG Narrative.

10.1.3 The primary sample preparation procedures to be used for this method are described in the following Sections (10.1.4 and 10.1.5). These methods are to be used without deviation. Alternative sample preparation procedures are listed in Section 18 of this method and may be requested by the USEPA Region. No other changes in the analyses will be permitted.

10.1.4 Aqueous/Water Sample Preparation

10.1.4.1 Preparation Method 200.8 – Total Recoverable Analytes [based on USEPA NERL Method 200.8 Rev 5.5 (October 1999)]

10.1.4.1.1 For the determination of total recoverable analytes in aqueous/water samples, transfer a 100 mL (±1 mL) aliquot from a well mixed, acid preserved sample to an appropriately sized (approximately 250 mL) beaker or other comparable digestion vessel. The sample shall not be diluted prior to digestion.

10.1.4.1.2 Add 2 mL 50% (v/v) nitric acid and 1 mL 50% (v/v) hydrochloric acid to the beaker containing the measured volume of sample. Place the beaker on a hotplate, or other comparable heating device, for solution evaporation. The hotplate should be located in a fume hood and previously adjusted to provide evaporation at a temperature of 95°C (±3 °C), when covered. The beaker should be covered with an elevated watch glass or other necessary steps should be taken to prevent sample contamination from the fume hood environment.

10.1.4.1.3 Reduce the volume of the sample aliquot to about 20 mL by gently heating at 95°C (±3 °C). **DO NOT BOIL.** This step takes about 2 hours for a 100 mL aliquot with the rate of evaporation rapidly increasing as the sample volume approaches 20 mL. (A spare beaker containing 20 mL of water can be used as a gauge.)

10.1.4.1.4 Cover the beaker with a watch glass to reduce additional evaporation and gently reflux the sample for 30 minutes. (Slight boiling may occur, but vigorous boiling must be avoided to prevent loss of the $HCl-H_2O$ azeotrope.)

10.1.4.1.5 Allow the beaker to cool. Quantitatively transfer the sample solution to a 100 mL volumetric flask, make to volume with reagent water, stopper, and mix.

10.1.4.1.6 Allow any undissolved material to settle overnight, or centrifuge or filter a portion of the prepared sample until clear to avoid plugging the nebulizer with solid particles.

10.1.4.1.7 Prior to analysis, if the chloride concentration interferes with the analysis, then the solution can be diluted to reduce the chloride concentration. The sample is now ready for analysis. Because the effects of various matrices on the stability of the samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.

10.1.4.1.8 The concentrations as determined by this method shall be reported as "Total". A reduced sample volume of 50 mL can be used. If this reduced volume is used, then all other reagents and volumes shall be reduced appropriately. The digested sample can be further diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

10.1.5 Soil/Sediment Sample Preparation

10.1.5.1 Preparation Method 3050B (based on USEPA OSW Method 3050B, Revision 2, December 1996)

10.1.5.1.1 Mix the sample thoroughly to achieve homogeneity (see Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh to the nearest 0.01 g and transfer 1.0 – 1.5 g sample (wet weight) to an appropriately sized digestion vessel (e.g., a beaker or hot block digestion tube).

10.1.5.1.2 Add 10 mL of 50% (v/v) nitric acid, mix the slurry, and cover with a watch glass or vapor recovery device. Heat the sample to 95°C (±3°C) and reflux for 10 to 15 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated nitric acid, replace the cover, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by the nitric acid, repeat this step (addition of 5 mL of concentrated nitric acid) over and over until <u>no</u> brown fumes are given off by the sample indicating the complete reaction with nitric acid. Using a ribbed watch glass or vapor recovery system, either allow the solution to evaporate to approximately 5 mL without boiling or heat at 95°C (±3°C) without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times.

10.1.5.1.3 After the sample has cooled, add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide. Cover the vessel with a watch glass or vapor recovery device and return the covered vessel to the heat source for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until the effervescence subsides and cool the vessel. Continue to add 30% hydrogen peroxide in 1-mL amounts with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL of 30% hydrogen peroxide.) Cover the sample with a ribbed watch glass or vapor recovery device and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL or heat at 95°C (±3°C) without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times.

10.1.5.1.4 After cooling, transfer the digestate to a 100 mL volumetric flask through Whatman No. 41 filter paper (or equivalent). Rinse the filter paper with a small amount of reagent water

to complete the quantitative transfer of the analytes and collect the liquid in the same 100-mL volumetric flask. The solution being analyzed must be clear to avoid plugging the nebulizer with solid particles. Dilute to volume with reagent water, stopper, and mix. The sample is now ready for analysis.

10.1.5.1.5 Prior to analysis, the digested sample can be diluted (e.g., 1:4 would be suggested) with 1% nitric acid if high levels of chloride are present which may cause problems with some instruments and interfere with the analysis. Dilution of the digestate may also be necessary if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

10.2 Sample Analysis

10.2.1 For every new or unusual matrix, it is highly recommended that a semi-quantitative analysis be carried out to screen for high element concentrations. This screening procedure can be performed using ICP-AES or some other technique. Information gained from this may be used to prevent potential damage to the detector during sample analysis and to identify elements which may be higher than the calibrated range. Matrix screening may be carried out by diluting the sample by a factor of 500 and analyzing in semi-quantitative mode. The sample should also be screened for background levels of all elements chosen for use as internal standards in order to prevent bias in the calculation of analytical data. Undiluted sample results are not required if elements are present in the undiluted sample digestate at levels which could damage the detector.

10.2.2 Initiate instrument operating configuration. Tune and calibrate the instrument for the analytes of interest. Establish instrument software run procedures for quantitative analysis. For all sample analyses, a minimum of three replicate integrations are required for data acquisition. Use the average of all the integrations for data reporting.

10.2.3 The rinse blank should be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample. Samples should be aspirated for a sufficient period of time to obtain a stable response prior to the collection of data.

10.2.4 Sample digestates having high levels of interferences or concentrations higher than the established calibrated range as determined by the expected concentration of the highest calibration standard shall be diluted into range and reanalyzed. The sample digestate should first be analyzed for the trace elements, protecting the detector from the high concentration elements, if necessary, by the selection of appropriate scanning windows. The sample digestate should then be diluted for the determination of the remaining elements.

10.2.5 All masses which might affect data quality must be monitored during the analytical run. At a minimum, those masses identified in Table 3 - Recommended Isotopes and Masses for Selected Elements, must be monitored in the same scan that is used for the collection of the data. This information should be used to correct the data for identified interferences.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Recommended Elemental Equations

Elemental expressions recommended for sample data calculations are listed in Table 4 - Recommended Elemental Expressions for Isobaric Interferences. Do not report element concentrations below the Method Detection Limit (MDL).

11.2 Data Value Corrections

Data values as produced by the instrument should be corrected for instrument drift or sample matrix induced interferences by the application of internal standardization. Corrections for characterized spectral interferences should be applied to the data. Chloride interference corrections should be made on all samples, regardless of the addition of hydrochloric acid, as the chloride ion is a common constituent of environmental samples.

11.3 Multiple Monitored Isotopes

If an element has more than one monitored isotope, examination of the concentration calculated for each isotope or the isotope ratios will provide useful information in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of sample concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes; therefore, differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

11.4 Aqueous/Water Sample Calculation

EQ. 1 Aqueous/Water Sample Concentration

$$\text{Concentration } (\mu g/L) = C \times \frac{V_f}{V} \times DF$$

WHERE, C = Instrument value in µg/L (The average of all replicate integrations)
V_f = Final digestion volume (mL)
V = Initial Aliquot Amount (mL)
DF = Dilution Factor

11.5 Adjusted MDL/Adjusted Contract Required Quantitation Limit (CRQL) Calculation for Aqueous/Water Samples

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, multiply the value of the MDL or CRQL(µg/L) by the sample dilution factor and the V_f/V term as noted in Equation 1.

11.6 Soil/Sediment Sample Calculation

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of mg/kg:

EQ. 2 Soil/Sediment Sample Concentration

$$\text{Concentration (dry wt.) (mg/kg)} = C \times \frac{V_f}{W \times S} \times DF / 1000$$

WHERE,
- C = Instrument value in µg/L (The average of all replicate integrations)
- V_f = Final digestion volume (mL)
- W = Initial aliquot amount (g)
- S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
- DF = Dilution Factor

11.7 Adjusted MDL/Adjusted CRQL Calculation for Soil/Sediment Samples

Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

EQ. 3 Adjusted Soil/Sediment MDL or CRQL Concentration

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_M}{W \times S} \times \frac{V_f}{V_M} \times DF$$

WHERE,
- C = MDL or CRQL (mg/kg)
- W_M = Minimum method required aliquot amount (g) (1.00g or 0.50g)
- W = Initial aliquot amount (g)
- V_M = Method required final sample digestion volume (mL) (100 mL)
- V_f = Final digestion volume (mL)
- S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
- DF = Dilution Factor

12.0 QUALITY CONTROL (QC)

12.1 Tune Standard

The Tune Standard shall be prepared in the same acid matrix as the calibration standards and analyzed as a single analysis with at least five integrations. If the mass calibration is not within 0.1 amu over the range of 6 to 210 amu, or the percent Relative Standard Deviation (%RSD) of all the integrations of the absolute signals of the analytes exceeds 5.0%, the analysis shall be terminated, the problem corrected, and the instrument re-tuned. In addition, the laboratory must also monitor the resolution by measuring and reporting the full peak width (in amu) and the percentage of the peak height this full peak width was measured at for each of the analyte masses in the tune solution. All sample results reported must be associated with an instrument tune that meets these requirements.

12.2 Initial Calibration Verification (ICV)

The ICV Standard shall be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier. If the measurements exceed the control limits of 90% Recovery (low) or 110% Recovery (high), or if the %RSD as calculated from all replicate integrations exceeds 5.0%, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. The results of the ICV analysis shall be reported.

12.3 Continuing Calibration Verification (CCV)

The CCV standard shall be prepared in the same acid matrix as the calibration standards by combining compatible analytes at a concentration equivalent to the mid-levels of their respective calibration curves. If the deviation of the CCV is greater than the specified control limits of 90% Recovery (low) or 110% Recovery (high), or if the %RSD as calculated from all replicate integrations exceeds 5.0%, the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration verification shall be performed for the analytes affected. The results of all CCV analyses shall be reported.

12.4 Blank Analyses

There are two different types of blanks required by this method. The calibration blank is used in establishing the analytical curve. The Initial and Continuing Calibration Blanks (ICB/CCB) are identical in composition to the calibration blank and are analyzed immediately after the ICV/CCV to monitor for potential carryover of analytes. The preparation blank is used to monitor for possible contamination throughout the entire sample preparation and analysis process.

12.4.1 Initial and Continuing Calibration Blank (ICB/CCB)

The ICB and CCB are identical in composition to the Calibration Blank as used to establish the initial calibration curve. If the absolute value of the calibration blank (ICB/CCB) result exceeds the Contract Required Quantitation Limit (CRQL) (see Exhibit C), the analysis shall be terminated, the problem corrected, the instrument

recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration blank shall be performed for the analytes affected. The results of the ICB and CCB analyses shall be reported.

12.4.2 Preparation Blank

12.4.2.1 The Preparation Blank shall contain all the reagents and in the same volumes as used in processing the samples. The Preparation Blank shall be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

12.4.2.2 At least one Preparation Blank, consisting of reagent water processed through each sample preparation and analysis procedure (see Section 10), shall be prepared and analyzed with every Sample Delivery Group (SDG), or with each batch[1] of samples digested, whichever is more frequent.

12.4.2.3 The first batch of samples in an SDG is to be assigned to Preparation Blank one, the second batch to Preparation Blank two, etc. Each Complete SDG File (CSF) shall contain the results of all Preparation Blank analyses associated with the samples in that SDG.

12.4.2.4 The Preparation Blank(s) is (are) to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

12.4.2.4.1 If the absolute value of the concentration of the blank is less than or equal to the CRQL (see Exhibit C), no further action is required.

12.4.2.4.2 If the analyte concentration in the blank is above the CRQL, the lowest concentration of that analyte in the associated samples (except those identified as field blanks) shall be greater than or equal to 10 times the blank concentration. Otherwise, all samples (except those identified as field blanks) associated with the blank, with the analyte concentration less than 10 times the blank concentration and above the CRQL, shall be redigested and reanalyzed with appropriate new Quality Control (QC) for that analyte. The sample concentration is not to be corrected for the blank value.

12.4.2.4.3 If the concentration of the blank is below the negative CRQL, then all samples reported below 10 times the CRQL associated with the blank shall be redigested and reanalyzed with appropriate new QC.

12.4.2.4.4 The results for the Preparation blank shall be reported.

12.5 Interference Check Sample (ICS)

12.5.1 The ICS is obtained from USEPA. If not available, then the ICS can be prepared by the analyst.

12.5.2 To verify corrections for elemental and polyatomic isobaric interferences, the Contractor shall analyze and report the results for the ICS for all elements on the Target Analyte List (TAL) and

[1] A group of samples prepared at the same time.

monitor for all interferents, immediately after the initial calibration sequence, but not before the ICV/ICB. The analysis of the ICS shall be immediately followed by the analysis of a CCV/CCB pair. The ICS solutions shall be obtained from USEPA, if available, and analyzed according to instructions supplied with the ICS. The Contractor shall always initially run the ICS undiluted. Dilution of the ICS (for the higher concentration elements) may be necessary to meet the calibrated range values of the instrument.

12.5.3 The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents; Solution AB consists of the analytes mixed with the interferents. Even though no analytes have been added to Solution A, low levels of some of the analytes may be present due to the purity of the materials used. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A.

12.5.4 The analytical results of ICS Solution A (ICSA) shall fall within the control limit ±20% of the analyte's true value or ±2 times the CRQL of the analyte's true value, whichever is greater. If the true value for a given analyte is not listed in the certified values for the solution, then the true value shall be assumed to be zero and the ±2 times the CRQL control limits shall apply. For example, for Copper (CRQL = 2 µg/L, ICSA true value = 8 µg/L) the correct control window to use would be the greater of ±20% of the true value (0.20 x 8 µg/L = ±1.6 µg/L) or ±2 times the CRQL (±4 µg/L). Therefore, the control window for the found value for Chromium in the ICSA is 8±4, or 4 to 12 µg/L. If the analytical results of the ICSA do not fall within the control limits, the analysis shall be terminated, the problem corrected, and the instrument recalibrated.

12.5.5 Results for the ICS Solution AB (ICSAB) shall fall within the control limit of ±20% of the true value or ±2 times the CRQL of the true value, whichever is greater. If the analytical results of the ICSAB do not fall within the control limits, the analysis shall be terminated, the problem corrected, and the instrument recalibrated.

NOTE: The control limits and concentrations for the ICSAB are being monitored. These may be adjusted to provide greater control of interferences.

12.5.6 If true values for analytes contained in the ICS are not supplied with the solutions, the mean shall be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination shall be made during an analytical run where the results for a previously supplied ICS met all contract specifications. Additionally, the results of this initial mean determination shall be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted). Only if the ICS solutions are not available from USEPA, independent Check Samples shall be prepared with interferent and analyte concentrations at the levels specified in Table 1 - Interferent and Analyte Elemental Concentrations Used for ICP-MS Interference Check Sample (ICS). The mean value and standard deviation shall be established by initially analyzing the Check Samples at least five times repetitively for each analyte listed. Results shall fall within the control limit of ±20% of the established mean value or ±2 times the analyte's CRQL of the established mean value, whichever is greater. The mean and standard deviation shall be reported in the raw data. Results from the ICS analyses shall be reported for all ICP-MS target analytes.

12.5.7 The results of the ICS analysis shall be reported.

12.6 Spike Sample Analysis

12.6.1 The spike sample analysis is designed to provide information about the effect of sample matrix on the digestion and/or measurement methodology. The spike is added before the digestion (i.e., prior to the addition of other reagents). At least one spike sample analysis (matrix spike) shall be performed on each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[2]

12.6.2 If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 12.7). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks and Performance Evaluation (PE) samples shall not be used for spiked sample analysis. USEPA may require that a specific sample be used for the spike sample analysis.

12.6.3 The analyte spike shall be added in the amount given in Table 6 - Spiking Levels for Spike Sample Analysis, for each element analyzed. This is the level of spike present in the final digestate.

12.6.4 If the spike recovery is not at or within the limits of 75-125%, the data for all samples received and associated with that spike sample shall be flagged with the letter "N". An exception to this rule is granted when the sample concentration exceeds the Spike Added (SA) concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

12.6.5 When the matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike shall be performed for those analytes that do not meet the specified criteria. Note that if a post-digestion spike analysis is required for an analyte, the same EPA sample that was used for the matrix spike analysis shall be used for the post-digestion spike analysis. Spike the unspiked aliquot of the undiluted digestate at two times the indigenous level or two times the CRQL, whichever is greater.

12.6.6 In the instance where there is more than one spike sample per matrix per SDG, if one spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG. Individual component percent recoveries are calculated as follows:

[2] USEPA may require additional spike sample analyses, upon USEPA Regional CLP Project Officer (CLP PO) request.

EQ. 4 Matrix Spike and Post-Digestion Spike Percent Recovery

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

WHERE, SSR = Spike Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2
SR = Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2
SA = Spike Added Theoretical Result (µg/L or mg/kg). This is calculated by substituting the spiking amount used for the 'V_f' term and substituting the spiking standard concentration used for the 'C' term from EQ. 1 or EQ. 2.

12.6.7 When sample concentration is less than the Method Detection Limit (MDL), use SR = 0 only for purposes of calculating the percent recovery. The Spike Sample Results (SSRs), Sample Results (SRs), Spike Added Theoretical Results (SA), and Percent Recovery (positive or negative) shall be reported.

12.6.8 The units used for reporting Spiked Sample Results (SSRs) will be identical to those used for reporting Sample Results (SRs).

12.7 Duplicate Sample Analysis

12.7.1 One duplicate sample shall be analyzed for each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[3] Duplicates cannot be averaged for reporting.

12.7.2 Duplicate sample analyses are not required for percent solids. Samples identified as field blanks and PE samples shall not be used for duplicate sample analysis. USEPA may require that a specific sample be used for duplicate sample analysis. The Relative Percent Difference (RPD) for each analyte is calculated as follows:

EQ. 5 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

WHERE, RPD = Relative Percent Difference
S = Sample Result (original) (µg/L or mg/kg) from EQ.1 or EQ. 2
D = Duplicate Sample Result (µg/L or mg/kg) from EQ.1 or EQ. 2

12.7.3 The results of the duplicate sample analyses shall be reported. A control limit of 20 for RPD shall be used for original and duplicate sample values greater than or equal to five times the CRQL (see Exhibit C). A control limit equal to the CRQL shall be entered in the "Control Limit" column if either the sample or duplicate value is less than five times the CRQL. If the sample and duplicate values are greater than or equal to five times the CRQL, or if the sample and duplicate values are less than the CRQL, the "Control Limit" field is left empty.

12.7.4 If one result is above five times the CRQL level and the other is below, use the CRQL criteria to determine if the duplicate analysis is in control. If both sample and duplicate values are less than

[3] USEPA may require additional duplicate sample analyses, upon USEPA Regional CLP PO request.

the CRQL, the RPD is not calculated. For soil/sediment sample or soil/sediment duplicate results less than five times the CRQL, enter the value of the CRQL, corrected for sample weight and percent solids (i.e., original, not duplicate sample weight), in the "Control Limit" column. If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*". In the instance where there is more than one duplicate sample per SDG, if one duplicate result is not within contract criteria, flag all samples of the same matrix in the SDG. The percent difference data will be used by USEPA to evaluate the long-term precision of the methods for each analyte. Specific control limits for each analyte may be added at a later date based on these precision results.

12.8 Laboratory Control Sample (LCS) Analysis

12.8.1 The LCS for aqueous/water and soil/sediment samples shall be prepared by spiking an aliquot of reagent water (50-100 mL for aqueous/water, 1 mL for soil/sediment) such that the final digestate shall contain each analyte at two times the CRQL for the associated matrix. One LCS shall be prepared for each prepared batch of aqueous/water or soil/sediment samples in an SDG.

12.8.2 All LCS and percent recovery results shall be reported. If the percent recovery for the LCS falls outside the control limits of 70-130%, the analyses shall be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed with appropriate new QC.

12.9 ICP-MS Serial Dilution Analysis

12.9.1 Prior to reporting concentration data for the analytes, the Contractor shall analyze and report the results of the ICP-MS serial dilution analysis. The ICP-MS serial dilution analysis shall be performed on a sample from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent. Samples identified as field blanks and PE samples shall not be used for serial dilution analysis.

12.9.2 If the analyte concentration is sufficiently high (minimally a factor of 50 above the MDL in the original sample), the serial dilution (a five-fold dilution) shall then agree within 10% of the original determination after correction for dilution. If the dilution analysis for one or more analytes is not within a control limit of 10%, and the internal standards in the original sample met the contract criteria, an interference effect must be suspected, and the data for all affected analytes in the samples received and associated with that serial dilution must be flagged with an "E".

12.9.3 The percent differences for each component are calculated as follows:

EQ. 6 Serial Dilution Percent Difference

$$\%Difference = \frac{|SR - SDSR|}{SR} \times 100$$

WHERE, SR = Sample Result (µg/L or mg/kg) from EQ.1 or EQ. 2
SDSR = Serial Dilution Sample Result (µg/L or mg/kg) from EQ.1 or EQ.2

12.9.4 In the instance where there is more than one serial dilution per SDG, if one serial dilution result is not within the contract criteria, flag all samples of the same matrix in the SDG. Serial dilution results and "E" flags shall be reported.

12.9.5 If the internal standard responses for the field sample chosen for serial dilution analysis are not within the limits and the appropriate corrective action (two-fold dilution and reanalysis) is taken, the following shall apply to the serial dilution analysis: if the internal standard responses of the field sample reanalysis are within the limits, the serial dilution results are to be reported from a five-fold dilution of the reanalyzed sample. If the internal standard responses of the field sample reanalysis are not within the limits, the serial dilution results are to be reported from a five-fold dilution of the original sample.

12.10 Internal Standards

The analyst shall monitor the responses from the internal standards throughout the sample set being analyzed. Ratios of the raw uncorrected internal standard responses between isotopes should also be routinely monitored. This information may be used to correct potential problems caused by mass dependent drift, errors incurred in adding the internal standards, or increases in the concentrations of individual internal standards caused by background contributions from the sample. The absolute response of any one internal standard must not deviate more than 60-125% from the original response in the calibration blank.

Calculate the Percent Relative Intensity as follows:

EQ. 7 Percent Relative Intensity

$$\%RI = \frac{I_n}{I_0} \times 100$$

WHERE, I_n = Raw Uncorrected Intensity of the internal standard in the sample
I_0 = Raw Uncorrected Intensity of the internal standard in the calibration blank (S0)

If deviations greater than these are observed in field samples, matrix spikes, or duplicate samples, the original sample shall be diluted by a factor of two, internal standards added (if not automatically added by the instrument), and the sample reanalyzed for the analyte(s) associated with the non-compliant internal standard(s). If the internal standard responses for the diluted sample analysis are within the limits, report the results of this analysis on the appropriate Summary Form. If the internal standard responses for the diluted sample analysis are not within the limits, note this in the SDG Narrative and report the results of the undiluted original sample analysis on the appropriate Summary Form.

Target analyte(s) concentration(s) must be within the calibrated range before assessing internal standard response for those internal standard(s) associated with the analyte(s).

12.11 MDL Determination

12.11.1 Before any field samples are analyzed under this contract, the MDLs shall be determined for each digestion procedure and instrument used prior to the start of contract analyses and annually thereafter. The MDLs shall meet the levels specified in Exhibit C. An MDL study shall be performed after major instrument maintenance, or changes in instrumentation or instrumental conditions, to verify the current sensitivity of the analysis.

12.11.1.1 To determine the MDLs, the Contractor shall run MDL studies following the procedures given in 40 CFR, Part 136. The Contractor shall prepare the MDL samples by each digestion procedure used and shall analyze these samples on each instrument used.

12.11.1.2 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C.

12.11.1.3 The results of the MDL determination studies shall be forwarded to the USEPA Regional CLP PO, Sample Management Office (SMO), and Quality Assurance Technical Support (QATS).

12.11.1.4 The MDL results shall be reported on Form IX-IN.

12.12 Example Analytical Sequence for ICP-MS Including the Initial Calibration

Tune
S0
S
S
S
S
S
ICV
ICB
ICSA
ICSAB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

12.13 Summary of QC Operations

The QC operations performed for ICP-MS analysis are summarized in the table below.

QC Operation	Frequency
Instrument Calibration	Each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment.
Initial Calibration Verification	Following each instrument calibration for each wavelength or mass used.
Initial Calibration Blank	Following each instrument calibration, immediately after the ICV.
Continuing Calibration Verification	For each wavelength or mass used, at a frequency of every 2 hours of a run, at the beginning of each day, and at the beginning and end of each run.
Continuing Calibration Blank	Every 2 hours of a run, at the beginning of each day, and at the beginning and end of each run. Performed immediately after the last CCV.
Interference Check Sample	At the beginning of the run after the ICB but before the CCV.
Serial Dilution for ICP	For each matrix type or for each SDG, whichever is more frequent.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Laboratory Control Sample	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Post-Digestion/Distillation Spike	Each time Spike Sample Recovery is outside QC limits.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
ICP-MS Tune	Prior to calibration.
Determination of Method Detection Limits	Prior to contract award, annually thereafter, and after major instrument adjustment.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 1.15 in Exhibit D - Introduction to Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 1.16 in Exhibit D - Introduction to Analytical Methods.

16.0 REFERENCES

16.1 US Environmental Protection Agency. Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry. Method 200.8. Revision 5.5. October 1999.

16.2 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 6020A. Third Edition, 1986, Update IV-A. 1998.

16.3 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3050B, Revision 2. Third Edition, Update III. December 1996.

16.4 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3015A, Revision 1, Update IVa. January 1998.

16.5 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3051A, Revision 1, Update IVa. January 1998.

16.6 US Government Printing Office. 40 Code of Federal Regulations, Part 136. Section 1. Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1: Interferent and Analyte Elemental Concentrations Used for ICP-MS Interference Check Sample (ICS)

Analytes	**(µg/L)**	**Interferents**	**(µg/L)**
Ag	20	Al	100000
As	20	Ca	100000
Ba	20	Fe	100000
Be	20	Mg	100000
Cd	20	K	100000
Co	20	Na	100000
Cr	40	P (as orthophosphate)	100000
Cu	25	S (as sulfate)	100000
Mn	30	C (as citrate)	200000
Ni	25	Cl	1000000
Pb	25	Mo	2000
Sb	20	Ti	2000
Se	20		
Tl	20		
V	20		
Zn	30		

NOTE: ICS Solution A (ICSA) contains the interferents at the indicated concentrations. The ICSA may be analyzed at twice the concentration indicated when interferences are present at higher concentrations in the sample. ICS Solution AB (ICSAB) contains all of the analytes and interferents listed above at the indicated concentrations. The ICS solutions are normally provided by USEPA along with the established concentrations of each of the analytes and interferents.

TABLE 2: Isobaric Molecular-Ion Interferences

Analyte	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	SeCl, AsCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl;	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	Arn, CaN			CaC	
^{59}Co	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	S_2, SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN			WC	

Analyte	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	ScO	CaOH	TiN	MgCl	SiS	TiC	Sn^{++}
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	S_2	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl,KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	NiOH	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	FeOH	NiN	Cl_2, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
$^{205}T1$							
$^{203}T1$		WOH					
^{51}V	C1O	SOH	C1N	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo^{++}
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	S_2	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	S_2	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	$C1_2$	ArS	NiC	

NOTE: The information provided in this table does not indicate that all of the described interferences need to be tested. However, this table can be consulted if unusual samples are encountered.

TABLE 3: Recommended Isotopes and Masses for Selected Elements

Element of Interest	**Analyte Masses - Choose One, or More - Calibrated**	**Masses to be Monitored**
Aluminum	27	
Antimony	121	
Arsenic	75	77, 82 (Isobaric Equation Required)
Barium	135, 137	
Beryllium	9	
Cadmium	111	106, 108 (Isobaric Equation Required)
Calcium	44	
Chromium	52	
Cobalt	59	
Copper	63, 65	
Iron	54, 56, 57	
Lead	206, 207, 208	
Magnesium	24, 25, 26	
Manganese	55	
Nickel	60	
Potassium	39	
Selenium	78, 82	
Silver	107, 109	
Sodium	23	
Thallium	203, 205	
Vanadium	51	52, 53 (Isobaric Equation Required)
Zinc	66	
Potential Interferent		
Titanium (TiO on 63Cu)		47 (No Isobaric Equation Required)
Krypton (Kr on 82Se)		83 (No Isobaric Equation Required)
Molybdenum		94, 95, 96, 97, 98
Tin (Sn on 115In)		118 (Isobaric Equation Required)

NOTE: Where possible, alternative isotopes are indicated. At least one of the listed masses shall be used as a quantitation ion. Those isotopes not listed shall not be used as a primary isotope for measurement, although they may be monitored for interference corrections if necessary.

TABLE 4: Recommended Elemental Expressions for Isobaric Interferences

Element	Isobaric Correction	Expression Proportional to Elemental Concentration
As	ArCl, Se	$(1.0000)(^{75}C) - (3.127)[(^{77}C) - (0.815)(^{82}C)]$
Cd	MoO, Pd	$(1.000)(^{111}C) - (1.073)[(^{108}C) - (0.712)(^{106}C)]$
V	ClO, Cr	$(1.0000)(^{51}C) - (3.127)[(^{53}C) - (0.113)(^{52}C)]$
In	Sn	$(1.0000)(^{115}C) - (0.0140)(^{118}C)$

C - Calibration blank subtracted counts at specified mass

The coefficients in correction equations were calculated using natural isotopic abundances, and assuming zero instrumental fractionation. For each particular instrument, these coefficients must be determined experimentally using the procedures or coefficients provided by the instrument manufacturer.

The correction equations shall not be applied if appropriate interference check sample measurement demonstrates absence of interference above the Contract Required Quantitation Limit (CRQL).

TABLE 5: Internal Standards (must use at least five)

Internal Standard	Mass	CAS Number
Lithium	6	7439-93-2
Scandium	45	7440-20-2
Yttrium	89	7440-65-5
Rhodium	103	7440-16-6
Terbium	159	7440-27-9
Holmium	165	7440-60-0
Lutetium	175	7439-94-3
Bismuth	209	7440-69-9

NOTE: Use of Li^6 requires enriched standard.

TABLE 6: Spiking Levels for Spike Sample Analysis

Analyte	Spike (µg/L)*	Spike (mg/kg)*
Sb	100	10
As	40	4
Ba	2000	200
Be	50	5
Cd	50	5
Cr	200	20
Co	500	50
Cu	250	25
Pb	20	2
Mn	500	50
Ni	500	50
Se	10	1
Ag	50	5
Tl	50	5
V	500	50
Zn	500	50

*Level in the final prepared sample

18.0 ALTERNATIVE SAMPLE PREPARATION PROCEDURES

18.1 Regionally-Selected Alternative Sample Preparation Procedure

Due to the variability of the matrices received and the interferences encountered, the sample preparation procedures described in Sections 10.1.4 and 10.1.5 may not always produce valid and/or reliable results. Sample preparation procedures are given in this section that describe alternative procedures that can be used if interferences or other issues are noted with the primary procedures. If the Contractor feels that one of these alternative sample preparation procedures would provide better results than the primary methods, then the Contractor shall contact the Sample Management Office (SMO). SMO will contact the USEPA Region for instructions. The Contractor shall document the USEPA Region's decision in the Sample Delivery Group (SDG) Narrative along with populating the Forms and the electronic data deliverable with the valid values that described the actual sample preparation procedures used by the Contractor.

18.2 Alternative Aqueous/Water Sample Preparation Procedures

18.2.1 Preparation Method 3015A – Nitric Acid Digestion (based on USEPA OSW Method 3015A, Revision 1, January 1998)

18.2.1.1 This method describes a sample preparation procedure for the determination of total analytes in various aqueous/water matrices using a microwave technique with a nitric acid only digestion.

18.2.1.2 Measure a 45.0 mL (±0.1 mL) aliquot of a well-shaken, homogenized sample using an appropriate volumetric delivery device, and quantitatively transfer the aliquot to an appropriate vessel equipped with a controlled pressure relief mechanism. The sample shall not be diluted prior to digestion.

18.2.1.3 Add 5.0 mL (±0.1 mL) of concentrated nitric acid to the digestion vessel.

18.2.1.4 Seal the digestion vessel according to the manufacturer's directions. Properly place the digestion vessels in the microwave system according to the manufacturer's recommended specifications and, when applicable, connect appropriate temperature and pressure monitoring equipment to the digestion vessels according to the manufacturer's specifications.

18.2.1.5 The Preparation Blank must have 45.0 mL of reagent water and the same amount (5.0 mL) of acid that was added to the samples.

18.2.1.6 The microwave program is then started. The temperature of each sample should rise to 170°C (±5°C) in approximately 10 minutes and remain at 170°C (±5°C) for 10 minutes, or for the remainder of the 20-minute digestion period. When using temperature feedback control, the number of samples that may be simultaneously digested may vary, from one sample up to the maximum number of digestion vessels that can be heated by the magnetron of the microwave unit. Whenever fewer than the recommended number of samples are to be digested, the remaining digestion vessels should be filled with 45.0 mL of water and 5.0 mL of acid so that the full complement of vessels is achieved. This provides an energy balance, since the microwave power absorbed is proportional to the total absorbing mass in the cavity.

18.2.1.7 At the end of the microwave program, allow the digestion vessels to cool for a minimum of 5 minutes before removing them from the microwave system. When the vessels have cooled to near room temperature, determine if the digestion vessels have maintained their seal throughout the digestion. For vessels that are sealed as discrete separate entities, the digestion vessel weight may be taken before and after digestion to evaluate seal integrity. If the weight loss of the sample exceeds 1% of the weight of the sample and reagents, then the sample is considered compromised and the sample must be redigested. For digestion vessels with burst disks, a careful visual inspection of the disk, in addition to weighing, may identify compromised digestion vessels.

18.2.1.8 Complete the preparation of the sample by carefully uncapping and venting each digestion vessel in a fume hood. Quantitatively transfer the sample to an acid-cleaned bottle. If the digested sample contains particulates which may clog nebulizers or interfere with the injection of the sample into the instrument, the sample may be centrifuged, allowed to settle, or filtered through Whatman No. 41 (or equivalent) filter paper.

18.2.1.9 The final solution typically requires nitric acid to maintain appropriate sample solution acidity and stability of the elements. Commonly, a 2% (v/v) nitric acid concentration is desirable.

18.2.1.10 Transfer or decant the digested sample into a 50 mL volumetric flask and dilute to volume. The samples are now ready for analysis. Concentrations so determined shall be reported as "Total".

18.2.1.11 The digested sample can be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

18.3 Alternative Soil/Sediment Sample Preparation Procedures

18.3.1 Preparation Method 3051A – Nitric Acid Digestion (based on USEPA OSW Method 3051A, Revision 1, January 1998)

18.3.1.1 This method describes a sample preparation procedure for the determination of total recoverable analytes in various soil/sediment matrices using a microwave technique with a nitric acid only digestion.

18.3.1.2 Mix the sample thoroughly to achieve homogeneity (See Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh to the nearest 0.001 g and transfer a 0.50 g sample (wet weight) to an appropriate digestion vessel equipped with a controlled pressure relief mechanism.

18.3.1.3 Add 10 mL (±0.1 mL) of concentrated nitric acid to the digestion vessel in a fume hood.

18.3.1.4 The analyst should be aware of the potential for a vigorous reaction especially with samples containing volatile or easily oxidized organic species. When digesting a matrix of this type, initially use no more than 0.10 g (±0.001 g) of sample. If a vigorous reaction occurs upon the addition of the acid(s), allow the sample to predigest in the uncapped digestion vessel until

the reaction ceases. Heat may be added in this step for safety considerations (e.g., the rapid release of carbon dioxide from carbonates, easily oxidized organic matter, etc.). Once the initial reaction has ceased, the sample may continue through the digestion procedure.

18.3.1.5 Seal the digestion vessel according to the manufacturer's directions. Properly place the digestion vessels in the microwave system according to the manufacturer's recommended specifications and, when applicable, connect appropriate temperature and pressure monitoring equipment to the digestion vessels according to the manufacturer's specifications.

18.3.1.6 The Preparation Blank must have 0.50 g (±0.001 g) of reagent water and the same amount of acid(s) that was added to the samples.

18.3.1.7 The microwave program is then started. The temperature of each sample should rise to 175°C (±5°C) in approximately 5.5 (±0.25) minutes and remain at 175°C (±5°C) for 4.5 minutes, or for the remainder of the 10-minute digestion period. When using temperature feedback control, the number of samples that may be simultaneously digested may vary, from one sample up to the maximum number of digestion vessels that can be heated by the magnetron of the microwave unit. Whenever fewer than the recommended number of samples are to be digested, the remaining digestion vessels should be filled with 0.50 g of water and the same amount of acid(s) so that the full complement of vessels is achieved. This provides an energy balance, since the microwave power absorbed is proportional to the total absorbing mass in the cavity.

18.3.1.8 At the end of the microwave program, allow the digestion vessels to cool for a minimum of 5 minutes before removing them from the microwave system. When the vessels have cooled to near room temperature, determine if the digestion vessels have maintained their seal throughout the digestion. For vessels that are sealed as discrete separate entities, the digestion vessel weight may be taken before and after digestion to evaluate seal integrity. If the weight loss of the sample exceeds 1% of the weight of the sample and reagents, then the sample is considered compromised and the sample must be redigested. For digestion vessels with burst disks, a careful visual inspection of the disk, in addition to weighing, may identify compromised digestion vessels.

18.3.1.9 Complete the preparation of the sample by carefully uncapping and venting each digestion vessel in a fume hood. Quantitatively transfer the sample to an acid-cleaned bottle. Filter the digested sample through Whatman No. 41 (or equivalent) filter paper to remove particulates which would clog the nebulizer or otherwise interfere with the injection of the digested sample into the instrument.

18.3.1.10 The final solution typically requires nitric acid to maintain appropriate sample solution acidity and stability of the elements. Commonly, a 2% (v/v) nitric acid concentration is desirable.

18.3.1.11 Transfer or decant the digested sample into a 50 mL volumetric flask and dilute to volume. The samples are now ready for analysis.

18.3.1.12 The digested sample can be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves.

18.4 Microwave Calibration Procedure

18.4.1 The calibration procedure is a critical step prior to the use of any microwave unit. The microwave unit must be calibrated every six months. The data for each calibration must be available for review during on-site audits. In order that absolute power settings may be interchanged from one microwave unit to another, the actual delivered power must be determined.

18.4.2 Calibration of a laboratory microwave unit depends on the type of electronic system used by the manufacturer. If the unit has a precise and accurate linear relationship between the output power and the scale used in controlling the microwave unit, then the calibration can be a two-point calibration at maximum and 40% power. If the unit is not accurate or precise for some portion of the controlling scale, then a multiple-point calibration is necessary. If the unit power calibration needs a multiple-point calibration, then the point where linearity begins must be identified. For example: a calibration at 100, 99, 98, 97, 95, 90, 80, 70, 60, 50, and 40% power settings can be applied and the data plotted. The non-linear portion of the calibration curve can be excluded or restricted in use. Each percent is equivalent to approximately 5.5-6 watts and becomes the smallest unit of power that can be controlled. If 20-40 watts are contained from 99-100%, that portion of the microwave calibration is not controllable by 3-7 times that of the linear portion of the control scale and will prevent duplication of precise power conditions specified in that portion of the power scale.

18.4.3 The power available for heating is evaluated so that the absolute power setting (watts) may be compared from one microwave to another. This is accomplished by measuring the temperature rise in 1 kilogram (kg) of reagent water exposed to microwave radiation for a fixed period of time. The reagent water is placed in a polytetrafluoroethylene (PTFE) beaker (or a beaker that is made of some other material that does not absorb microwave energy) and stirred before measuring the temperature. Glass beakers absorb microwave energy and may not be used. The initial temperature of the reagent water must be between 19°C and 25°C. The beaker is circulated continuously through the field for at least 2 minutes at full power. The beaker is removed from the microwave, the reagent water is stirred vigorously, and the final temperature is recorded. The final reading is the maximum temperature reading after each energy exposure. These measurements must be accurate to ±0.1°C and made within 30 seconds of the end of heating. If more measurements are needed, do not use the same reagent water until it has cooled down to room temperature. Otherwise, use a fresh reagent water sample.

The absorbed power is determined by the following formula:

EQ. 8 Absorbed Power

$$P = \frac{(K)(C_p)(m)(DT)}{t}$$

WHERE,
- P = The apparent power absorbed by the sample in watts (joules per second)
- K = The conversion factor for thermochemical calories per second to watts (4.184)
- C_p = The heat capacity, thermal capacity, or specific heat (cal. g^{-1} $°C^{-1}$) of reagent water (1.0)
- m = The mass of the sample in grams (g)
- DT = The final temperature minus the initial temperature (°C)
- t = The time in seconds (s)

Using 2 minutes and 1 kg of reagent water, the calibration equation simplifies to:

$$P = (DT)(34.87)$$

The microwave user can now relate power in watts to the percent power setting of the microwave.

18.5 Microwave Digestion Cleaning Procedure

18.5.1 Initial Cleaning of the Polytetrafluoroethylene Perfluoroalkoxy (PTFE PFA) Digestion Vessels

18.5.1.1 Prior to first use - new vessels must be annealed before they are used. A pretreatment/cleaning procedure must be followed. This procedure calls for heating the vessels for 96 hours at 200°C. The vessels must be disassembled during annealing and the sealing surfaces (the top of the vessel or its rim) must not be used to support the vessel during annealing.

18.5.1.2 Rinse in reagent water.

18.5.1.3 Immerse in 50% (v/v) hydrochloric acid for a minimum of 3 hours after the cleaning bath has reached a temperature just below boiling.

18.5.1.4 Rinse in reagent water.

18.5.1.5 Immerse in 50% (v/v) nitric acid for a minimum of 3 hours after the cleaning bath has reached a temperature just below boiling.

18.5.1.6 The vessels are then rinsed with copious amounts of reagent water prior to use for any analyses under this contract.

18.5.2 Cleaning Procedure between Sample Digestions

18.5.2.1 Wash entire vessel in hot water using laboratory-grade non-phosphate detergent.

18.5.2.2 Rinse with 50% (v/v) nitric acid.

18.5.2.3 Rinse 3 times with reagent water.

EXHIBIT D - PART C

ANALYTICAL METHODS
FOR
COLD VAPOR MERCURY ANALYSIS

Exhibit D – Analytical Methods for Cold Vapor Mercury Analysis

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1.0 SCOPE AND APPLICATION

The analytical method that follows is designed to analyze aqueous/water, soil/sediment, and sludge samples taken from hazardous waste sites using a cold vapor technique with Atomic Absorption (AA) for total mercury.

In addition to inorganic forms of mercury, organic mercury may also be present. These organo-mercury compounds will not respond to the cold vapor AA technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but studies have shown that a number of organo-mercury compounds, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant with these compounds. Therefore, a persulfate oxidation step following the addition of the permanganate has been included in most preparation procedures to ensure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in, or spiked to, a natural system.

2.0 SUMMARY OF METHOD

2.1 General Method Overview

This method is based on the absorption of radiation at 253.7 nanometers (nm) by mercury vapor. Inorganic and some organic forms of mercury are chemically reduced to the free atomic state by reacting the sample with a strong reducing agent like stannous chloride or stannous sulfate in a closed reaction vessel. The volatile free mercury is then driven from the reaction flask by bubbling air through the solution. Mercury atoms are carried in the air stream through tubing connected to an absorption cell, which is placed in the light path of the Atomic Absorption (AA) spectrophotometer. Sometimes the cell is heated slightly to avoid water condensation. As the mercury atoms pass into the sampling cell, measured absorbance rises indicating the increasing concentration of mercury atoms in the light path. Some systems allow the mercury vapor to pass from the absorption tube to waste, in which case the absorption peaks and then falls as the mercury is depleted. The highest absorbance observed during the measurement or the associated peak area are usually taken as the analytical signal.

2.2 Summary of Preparation and Analysis Procedures

2.2.1 Heated Acid Digestion and Analysis of Aqueous/Water Samples (based on USEPA 7470A) - Manual Preparation Method Followed by an Automated Analysis Method

2.2.2 Heated Acid Digestion and Analysis of Soil/Sediment Samples (based on USEPA 7471B) - Manual Preparation Method Followed by an Automated Analysis Method

2.2.3 Alternative digestion procedures are included in Section 18 of this Exhibit.

3.0 DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

4.1 Aqueous/Water

4.1.1 Some sea waters and wastewaters high in chlorides have shown a positive interference, and require additional permanganate [as much as 25 milliliters (mL)]. During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253.7 nanometers (nm). Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). Both inorganic and organic mercury spikes have been quantitatively recovered from the sea water using this technique.

4.1.2 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

4.1.3 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on recovery of mercury from spiked samples.

4.1.4 Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized by this procedure. When this occurs, the recovery of organic mercury will be low. The problem can be eliminated by reducing the amount of the original sample or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

4.2 Soil/Sediment

4.2.1 The same types of interferences that may occur in aqueous/water samples are also possible with soil/sediment samples (i.e., sulfides, high copper, high chlorides, etc.).

5.0 SAFETY

See Section 1.14 in Exhibit D - Introduction to Analytical Methods.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 Glassware/Labware

6.1.1 Graduated cylinders

6.1.2 Various volumetric flasks and pipets (Class A)

6.1.3 Suitable digestion vessels (300 mL BOD bottles, hot block digestion tubes, etc.), along with a suitable heating source/water bath for heating of the samples to about 95°C

6.1.4 Balances – Top loader balance, 300 gram (g) capacity, and a minimum sensitivity of ±1.0 milligram (mg)

6.2 General Instrumentation Information for the Automated Analysis of Previously Digested Aqueous/Water and Soil/Sediment Samples

6.2.1 Sampler for the uptake of the previously digested samples

6.2.2 Manifold/Pump system for the addition and mixing of reagents with the previously digested samples

6.2.3 Liquid-Vapor separator

6.2.4 Vapor dryer

6.2.5 Suitable absorption cell

6.2.6 Atomic Absorption (AA) Spectrophotometer – Any AA unit having an open sample presentation area in which to mount the absorption cell would be suitable. AA unit must also be equipped with a Mercury Hollow Cathode Lamp or other suitable light source. Instrument settings recommended by the particular manufacturer should be followed. The instrument must be capable of meeting the specified Contract Required Quantitation Limits (CRQLs) for mercury.

6.2.7 Suitable computer system for data processing

7.0 REAGENTS AND STANDARDS

7.1 Reagents for the Manual Digestion of Aqueous/Water and Soil/Sediment Samples

7.1.1 Aqueous/Water and Soil/Sediment by Manual Digestion Technique

7.1.1.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-06). Use this preparation for all reagents, standards, and dilutions of solutions.

7.1.1.2 Sulfuric acid, concentrated - Reagent grade.

7.1.1.2.1 Sulfuric acid, 0.5N - Dilute 14.0 mL of concentrated sulfuric acid to 1 L.

7.1.1.3 Nitric acid, concentrated - Reagent grade of low mercury content. If a high Preparation Blank is obtained, it may be necessary to distill the nitric acid.

7.1.1.4 Sodium chloride-hydroxylamine sulfate solution, 12% solution (w/v) - Add 12 g of sodium chloride and 12 g of hydroxylamine sulfate in reagent water and dilute to 100 mL.

NOTE: Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.

7.1.1.5 Potassium permanganate ($KMnO_4$) - 5% solution (w/v). Dissolve 5 g of potassium permanganate in 100 mL of reagent water.

7.1.1.6 Potassium persulfate - 5% solution (w/v). Dissolve 5 g of potassium persulfate in 100 mL of reagent water.

7.1.1.7 Aqua regia - Prepare immediately prior to use by carefully adding three volumes of concentrated hydrochloric acid to one volume of concentrated nitric acid.

7.1.2 Reagents for the Automated Analysis of Aqueous/Water and Soil/Sediment Digested Samples

7.1.2.1 Stannous sulfate - Add 25 g stannous sulfate to 250 mL of 0.5N sulfuric acid. This mixture is a suspension and should be stirred continuously during use.

NOTE: Stannous chloride may be used in place of stannous sulfate.

7.1.2.2 Additional reagents may be needed according to the manufacturer's instructions (e.g., for rinsing out sample and reagent lines).

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards, except as noted, to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit E, Section 8.0. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

7.2.1.1 Stock standard solutions may be purchased from a reputable source or prepared from reagent grade chemicals or metals. (CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.)

7.2.1.2 Stock mercury solution - Dissolve 0.1354 g of mercuric chloride in 75 mL of reagent water. Add 10 mL of concentrated nitric acid and adjust the volume to 100.0 mL [1.0 mL = 1.0 milligram (mg) Hg].

7.2.1.3 Working mercury solution - Make successive dilutions of the stock mercury solution (see Section 7.2.1.2) to obtain a working standard containing 0.1 µg/mL. This working standard and the dilutions of the stock mercury solution shall be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask as needed before the addition of the aliquot. From this solution, prepare standards.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in glass or polyethylene containers. Aqueous/water samples must be preserved with nitric acid to pH less than or equal to 2 immediately after collection. All samples must be iced or refrigerated at 4°C (±2°C) from the time of collection until digestion.

8.1.1 Dissolved Metals

For the determination of dissolved metals, the sample must be filtered through a 0.45 micrometer (um) pore diameter membrane filter at the time of collection or as soon as possible. Use a portion of the sample to rinse the filter flask, discard this portion, and collect the required volume of filtrate. Preserve the filtrate with nitric acid to pH less than or equal to 2 immediately after filtration.

8.2 Procedure for Sample Storage and Disposal

Following digestion, the remaining unused portion of aqueous/water and soil sediment samples may be stored at room temperature within the laboratory until 60 days after delivery of a complete, reconciled data package to USEPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.3 Contract Required Holding Time

The maximum holding time for mercury is 26 days from Validated Time of Sample Receipt (VTSR).

9.0 CALIBRATION AND STANDARDIZATION

9.1 Instrument Operating Parameters

Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of that particular instrument. The Method Detection Limit (MDL) and precision must be investigated and established for mercury on that particular instrument. All measurements must be within the operational range of the instrument. It is the responsibility of the analyst to verify the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2 Cold Vapor Atomic Absorption (AA) Instrument Calibration Procedure

9.2.1 Instruments shall be calibrated daily or once every 24 hours, each time the instrument is set up, or after Initial Calibration Verification (ICV) or Continuing Calibration Verification (CCV) failure. The instrument standardization date and time shall be included in the instrument output.

9.2.2 The date and time of preparation and analysis shall be given in the laboratory logs (preparation) and instrument output (analysis).

9.2.3 Calibration standards shall be prepared fresh with each calibration performed. Calibrate the instrument with at least six standards, one of which must be a blank standard and one shall be at or below the Contract Required Quantitation Limit (CRQL), in graduated amounts in the appropriate range (Typical range is 0.20 to 10.0 µg/L). Standards and samples must be prepared in the same way on the same day.

9.2.4 The calibration curve shall be calculated using linear regression by plotting the concentration of mercury in the standard (in µg/L) on the X-axis versus the instrument response on the Y-axis. The instrument response is typically the measured Absorbance (displayed as peak area or peak height) for each standard. A standard linear regression, a weighted linear regression (e.g., 1/concentration or 1/concentration^2), or a linear regression with zero force calibration model can be used as appropriate. No other types of equations (e.g., quadratic) are to be used. The acceptance criteria for the initial calibration curve is a correlation coefficient greater than or equal to 0.995. Sample analysis shall not begin until this criteria and the criteria described in Sections 9.2.5 and 9.2.6 have been met.

9.2.5 The calibration equation must be checked to establish the representativeness of the data that were used to produce the calibration equation. This check involves the re-fitting of the non-blank calibration data back to the calibration equation or the comparison of the calculated concentration of each of the standards against the expected concentration of the associated standard. This difference is related to the actual residual. For this calculation, the Percent Difference shall be used where the above difference is further divided by the expected amount or concentration of the respective standard. If these Percent Differences for each of the standards do not fall within ±30%, then the calibration equation is not acceptable and must be corrected. If a standard is analyzed at

a concentration that is below the CRQL and the above criteria is not met, that value can be excluded from the calibration curve as long as the lowest non-zero standard is still analyzed at or below the CRQL and all standards included in the calibration curve are continuous and consecutive.

9.2.6 The y-intercept from the linear regression initial calibration equation shall also be evaluated. If the y-intercept is not below the CRQL for the initial calibration curve, the calibration is not acceptable and must be corrected. Samples are not to be analyzed until the y-intercept for the initial calibration curve meets the acceptance criteria.

9.2.7 Any changes or corrections to the analytical system shall be followed by recalibration.

9.3 Initial Calibration Verification (ICV)

9.3.1 Immediately after the AA system has been calibrated, the accuracy of the initial calibration shall be verified and documented for mercury by the analysis of the ICV solution at the wavelength used for analysis.

9.3.2 The ICV solution is obtained from USEPA. If the ICV solution is not available, then the ICV solution can be prepared by the laboratory using a certified solution of the analyte from an independent source. Analyses shall be conducted at a concentration other than that used for instrument calibration, but near the middle of the calibration range. An independent source is defined as a standard composed of the analyte from a different source than that used in the standards for instrument calibration. The ICV shall be prepared and analyzed in the same manner as the other samples and standards.

9.3.3 The ICV solution shall be run at the wavelength used for reporting final results.

9.4 Continuing Calibration Verification (CCV)

9.4.1 To ensure calibration accuracy during each analytical run sequence, a CCV shall be analyzed and reported at a frequency not to exceed every 1 hour during an analytical run sequence. The standard shall be analyzed and reported at the beginning of the analytical run sequence and after the last analytical sample. See the example sample analytical run sequence in Section 12.7. This CCV standard shall be prepared from the same source and at the same mid-level concentration as used during the initial calibration.

The same CCV standard shall be used throughout the analytical run sequences for a Sample Delivery Group (SDG) of samples received.

9.4.2 Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding analytical samples up to the previous CCV as applicable and the analytical samples following this CCV up to the next CCV as applicable).

9.5 Initial and Continuing Calibration Blank (ICB/CCB)

An ICB or CCB shall be analyzed at the wavelength used for reporting final results for mercury immediately after every ICV and CCV. The ICB and CCB(s) shall be prepared and analyzed in the same manner as the other samples and standards.

NOTE: The results for the ICB and CCBs shall be reported.

10.0 PROCEDURE

10.1 Sample Preparation and Analysis Procedures

10.1.1 If insufficient sample amount (less than 90% of the required amount) is received to perform the analyses, the Contractor shall contact the Sample Management Office (SMO) to inform them of the problem. SMO will contact the USEPA Region for instructions. The USEPA Region will either require that no sample analyses be performed or will require that a reduced volume be used for the sample analysis. No other changes in the analyses will be permitted. The Contractor shall document the USEPA Region's decision in the Sample Delivery Group (SDG) Narrative.

10.1.2 If multiphase samples (e.g., two-phase liquid sample, oily sludge/sandy soil/sediment sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the USEPA Region. If all phases of the sample are amenable to analysis, the USEPA Region may require the Contractor to do any of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.
- Separate the phases of the sample, and analyze one or more of the phases separately. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.1 If all of the phases are not amenable to analysis (i.e., outside the scope), the USEPA Region may require the Contractor to do any of the following:

- Separate the phases and analyze the phase(s) that is (are) amenable to analysis. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.2 No other changes in the analyses will be permitted. The Contractor shall document the USEPA Region's decision in the SDG Narrative.

10.1.3 The primary sample preparation procedures to be used for this method are described in the following Sections (10.1.4 and 10.1.5). These methods are to be used without deviation. Alternative sample preparation procedures are listed in Section 18 of this method and may be requested by the USEPA Region. No other changes in the analyses will be permitted.

10.1.4 Aqueous/Water Preparation and Analysis of Standards and Samples (For the Manual Preparation Method Followed by an Automated Analysis Method - based on USEPA OSW Method 7470A, Revision 1, September 1994)

10.1.4.1 Manual Technique for the Preparation of Standards and Samples

10.1.4.1.1 For preparation of the standards, transfer aliquots of the working mercury solution (see Section 7.2.1.3) to a series of suitable digestion vessels. Add enough reagent water to each digestion vessel to make a total volume of 100 mL (±1.0 mL) and mix thoroughly. The working standard and the dilutions prepared

from this working standard should be prepared fresh daily. The acidity of all of the standards should be maintained at 0.15% nitric acid. This acid should be added to the digestion vessel as needed prior to the dilution of the working standard.

10.1.4.1.2 For preparation of the samples, shake the sample until well mixed and transfer an aliquot of 100 mL (±1.0 mL), containing not more than 1.0 µg of mercury, to a suitable digestion vessel.

10.1.4.1.3 Add 5 mL of concentrated sulfuric acid and 2.5 mL of concentrated nitric acid to each of the digestion vessels, mixing after each addition. Add 15 mL of 5% potassium permanganate solution to each digestion vessel. Some samples (e.g., sewage samples) may require additional potassium permanganate. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 minutes. Ensure that equal amounts of potassium permanganate solution are added to all standards, blanks, and samples. Add 8 mL of 5% potassium persulfate solution to each digestion vessel and heat for 2 hours in a water bath or block digester maintained at 95°C (±3°C). Cool and add 6 mL of 12% sodium chloride-hydroxylamine sulfate solution to reduce the excess potassium permanganate. All standards and samples must be at the same final volume. Reagent water can be used to make any necessary final volume adjustments.

10.1.4.1.4 At this point, the digested standards and samples can be allowed to stand at room temperature for up to 48 hours prior to analysis. However, it is recommended that the digested standards and samples be analyzed as soon as possible. Proceed to Section 10.1.4.2 for analysis.

10.1.4.1.5 A reduced volume of 50 mL can be used for all standards, blanks and samples for this digestion procedure. All standards and reagents used in the digestion process shall be scaled back by half of their original required amounts when this reduced volume is used. However, it is the responsibility of the laboratory to confirm that the resultant data meets the CRQL requirements for mercury.

10.1.4.2 Automated Technique for the Analysis of the Digested Standards and Samples

10.1.4.2.1 Set up the automated analyzer using the recommendations as provided by the manufacturer. Set up the manifold and fill the reagent reservoir with the 10% (w/v) stannous sulfate solution (prepared in 0.5 N sulfuric acid). All reagent and sample lines should be cleaned according to the manufacturer's recommendations.

Transfer appropriate aliquots of the digested standards and samples to the autosampler in the order as suggested by the manufacturer. Complete the analysis of all of the digested standards and samples and construct the calibration curve. The calibration curve shall be constructed based on the concentration of mercury (in µg/L) in the undigested standards, ignoring the volume of reagents added during the digestion process.

10.1.4.2.2 If a sample's response exceeds the calibrated range of the instrument, the laboratory shall dilute the sample and reanalyze. Dilute a portion of the previously digested sample, which has not been treated with stannous sulfate, using a solution which maintains the same acid and other reagent concentrations as are present in the calibration standards (e.g., one of the calibration blanks). The laboratory shall then promptly analyze the diluted sample.

10.1.4.2.3 After the analysis is complete, clean out the system and all of the reagent and sample lines according to the manufacturer's recommendations.

10.1.5 Soil/Sediment Preparation and Analysis of Standards and Samples (For the Manual Preparation Method Followed by an Automated Analysis Method - Based on USEPA OSW Method 7471B, Revision 2, January 1998)

10.1.5.1 Manual Technique for the Preparation of Standards and Samples

10.1.5.1.1 For preparation of the standards, transfer aliquots of the working mercury solution (see Section 7.2.1.3) to a series of suitable digestion vessels. Add enough reagent water to each digestion vessel to make a total volume of 10 mL (±0.1 mL) and mix thoroughly. This working standard and the dilutions prepared from this working standard shall be prepared fresh daily. The acidity of all of the standards should be maintained at 0.15% nitric acid. This acid should be added to the digestion vessel as needed prior to the dilution of the working standard.

10.1.5.1.2 For preparation of the samples, mix the sample thoroughly to achieve homogeneity (See Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh (to the nearest 0.01 g) an aliquot amount of 0.50-0.60 g and place in the bottom of a suitable digestion vessel. Add 5 mL of reagent water to each sample.

10.1.5.1.3 Add 5 mL of aqua regia to each of the digestion vessels and heat for 2 minutes at 95°C (±3°C) in a water bath or block digester. Allow the contents of each digestion vessel to cool and add 50 mL of reagent water and 15 mL of 5% potassium permanganate solution. Mix thoroughly and heat again for 30 minutes at 95°C (±3°C) in a water bath or block digester. Cool and add 6 mL of 12% sodium chloride-hydroxylamine solution to each digestion vessel to reduce the excess potassium permanganate. CAUTION: This addition should be performed under a hood, as chlorine could be evolved. Add 55 mL of reagent water to each sample or 50 mL of reagent water to each standard. All standards and samples must be at the same final volume. Reagent water can be used to make any final volume adjustments, if necessary.

10.1.5.1.4 At this point, the digested standards and samples can be allowed to stand at room temperature for up to 48 hours prior to analysis. However, it is recommended that the digested standards and samples be analyzed as soon as possible. Refer to Section 10.1.4.2 for analysis.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Aqueous/Water Sample Calculation

11.1.1 Prepare a standard curve by plotting the instrument response (e.g., peak area or peak height) of the processed standards against their true concentrations (in µg/L) using linear regression as discussed in Section 9.2.4. Determine the instrument response (e.g., peak area or peak height) of the processed field and Quality Control (QC) samples and determine the concentration of mercury (in µg/L) from the standard curve.

11.1.2 Calculate the mercury concentration in the sample by the following formula:

EQ. 1 Aqueous/Water Sample Concentration

$$\text{Hg Concentration } (\mu g/L) = C \times DF$$

WHERE, C = Instrument value in µg/L from the calibration curve
DF = Dilution Factor of the original sample

11.2 Soil/Sediment Sample Calculation

11.2.1 Prepare a standard curve by plotting the instrument response (e.g., peak area or peak height) of the processed standards against their true concentrations (in µg/L) using linear regression as discussed in Section 9.2.4. Determine the instrument response (e.g., peak area or peak height) of the processed field and Quality Control (QC) samples and determine the concentration of mercury (in µg/L) from the standard curve.

11.2.2 Calculate the mercury concentration in the sample by the formula:

EQ. 2 Soil/Sediment Sample Concentration

$$\text{Hg Concentration (mg/kg)} = C \times \frac{1}{W \times S} \times DF \times 0.1$$

WHERE, C = Instrument value in µg/L from the calibration curve
W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
DF = Dilution Factor

11.3 Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, multiply the value of the MDL or CRQL (µg/L) by the Dilution Factor.

Calculate the adjusted MDL or CRQL for soil/sediment samples as follows:

EQ. 3 Adjusted Soil/Sediment MDL or CRQL Concentration

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_m}{W \times S} \times DF$$

WHERE,

C = MDL or CRQL (mg/kg)
W_m = Method required minimum sample weight (g) (0.50 g)
W = Initial aliquot amount (g)
S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
DF = Dilution Factor

12.0 QUALITY CONTROL (QC)

12.1 Initial Calibration Verification (ICV)

The ICV standard shall be prepared in accordance with the instructions provided by the supplier, and carried through the entire preparation and analysis procedure. If measurements exceed the control limits of 85% Recovery (low) or 115% Recovery (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. The results of the ICV analysis shall be reported.

12.2 Continuing Calibration Verification (CCV)

The CCV Standard shall be prepared in the same acid matrix as the calibration standards and at a concentration equivalent to the mid-level of the calibration curve using the same standards that were used to prepare the initial calibration curve and carried through the entire preparation and analysis procedure. If the measurements exceed the control limits of 85% Recovery (low) or 115% Recovery (high), the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration verification shall be performed. The results of the CCV analyses shall be reported.

12.3 Blank Analyses

There are two different types of blanks required by this method. The calibration blank is used in establishing the analytical curve. The Initial and Continuing Calibration Blanks (ICB/CCB) are identical in composition to the calibration blank and are analyzed immediately after the ICV/CCV to monitor for potential carryover. The Preparation Blank is used to monitor for possible contamination throughout the entire sample preparation and analysis process.

12.3.1 Initial and Continuing Calibration Blank (ICB/CCB)

The ICB and CCB are prepared with acids and reagent water, and are identical in composition to the Calibration Blank as used to establish the initial calibration curve and carried through the entire preparation and analysis procedure. If the absolute value of the ICB/CCB exceeds the Contract Required Quantitation Limit (CRQL) (see Exhibit C), the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration blank shall be performed. The results of the ICB and CCB analyses shall be reported.

12.3.2 Preparation Blank

12.3.2.1 The Preparation Blank shall contain all the reagents and in the same volumes as used in processing the samples. The Preparation Blank shall be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

12.3.2.2 At least one Preparation Blank, consisting of reagent water processed through each sample preparation and analysis procedure (see Section 10), shall be prepared and analyzed with every Sample Delivery Group (SDG), or with each batch[1] of samples digested, whichever is more frequent.

12.3.2.3 The first batch of samples in an SDG is to be assigned to Preparation Blank one, the second batch to Preparation Blank two, etc. Each Complete SDG File (CSF) shall contain the results of all Preparation Blank analyses associated with the samples in that SDG.

12.3.2.4 The Preparation Blank(s) is (are) to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

12.3.2.4.1 If the absolute value of the concentration of the blank is less than or equal to the CRQL (see Exhibit C), no further action is required.

12.3.2.4.2 If the analyte concentration in the blank is above the CRQL, the lowest concentration of the analyte in the associated samples (except those identified as field blanks) shall be greater than or equal to 10 times the blank concentration. Otherwise, all samples (except those identified as field blanks) associated with that blank, with the analyte concentration less than 10 times the blank concentration and above the CRQL, shall be redigested and reanalyzed with appropriate new Quality Control (QC). The sample concentration is not to be corrected for the blank value.

12.3.2.4.3 If the concentration of the blank is below the negative CRQL, then all samples reported below 10 times the CRQL associated with the blank shall be redigested and reanalyzed with appropriate new QC.

12.3.2.4.4 The results for the Preparation Blank shall be reported.

12.4 Spike Sample Analysis

12.4.1 The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology. The spike is added before the digestion (i.e., prior to the addition of other reagents) and prior to any sample dilutions. At least one spike sample analysis (matrix spike) shall be performed on each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[2] The sample and its associated spike sample shall initially be run at the same dilution.

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[1] A group of samples prepared at the same time.

[2] USEPA may require additional spike sample analyses, upon USEPA Regional CLP Project Officer (CLP PO) request.

12.4.2 If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 12.5). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks and Performance Evaluation (PE) samples shall not be used for spiked sample analysis. USEPA may require that a specific sample be used for the spike sample analysis.

12.4.3 The analyte spike shall be added at 1 µg/L (aqueous/water) or 0.5 mg/kg (soil/sediment). Adjustment shall be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values.

12.4.4 If the spike recovery falls outside the control limits, the data of all samples received and associated with that spike sample shall be flagged with the letter "N". An exception to this rule is granted when the sample concentration exceeds the spike added (SA) concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

12.4.5 In the instance where there is more than one spike sample per matrix, per method, per SDG, if one spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG. Individual component percent recoveries (%R) are calculated as follows:

EQ. 4 Matrix Spike Percent Recovery

$$\%\text{Recovery} = \frac{SSR - SR}{SA} \times 100$$

WHERE, SSR = Spiked Sample Result (in µg/L or mg/kg) from EQ. 1 or EQ. 2
SR = Sample Result (in µg/L or mg/kg) from EQ. 1 or EQ. 2
SA = Spike Added Theoretical Result (in µg/L or mg/kg)

12.4.6 When sample concentration is less than the Method Detection Limit (MDL), use SR = 0 only for purposes of calculating the percent recovery. The Spike Sample Results (SSRs), Sample Results (SRs), Spike Added Theoretical Results (SA), and Percent Recovery (positive or negative) shall be reported.

12.4.7 The units used for reporting Spiked Sample Results (SSRs) will be identical to those used for reporting Sample Results (SRs).

12.5 Duplicate Sample Analysis

12.5.1 One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[3] Duplicates cannot be averaged for reporting.

[3] USEPA may require additional duplicate sample analyses, upon USEPA Regional CLP PO request.

12.5.2 Duplicate sample analyses are not required for percent solids. Samples identified as field blanks and PE samples shall not be used for duplicate sample analysis. USEPA may require that a specific sample be used for duplicate sample analysis. The Relative Percent Difference (RPD) is calculated as follows:

EQ. 5 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

WHERE, RPD = Relative Percent Difference
S = Sample Result (original) (in µg/L or mg/kg) from EQ. 1 or EQ. 2
D = Duplicate Sample Result (in µg/L or mg/kg) from EQ. 1 or EQ. 2

12.5.3 The results of the duplicate sample analyses shall be reported. A control limit of 20 for RPD shall be used for original and duplicate sample values greater than or equal to five times the CRQL (see Exhibit C). A control limit equal to the CRQL shall be entered in the "Control Limit" column if either the sample or duplicate value is less than five times the CRQL. If the sample and duplicate values are greater than or equal to five times the CRQL, or if the sample and duplicate values are less than the CRQL, the "Control Limit" field is left empty.

12.5.4 If one result is above five times the CRQL level and the other is below, use the CRQL criteria to determine if the duplicate analysis is in control. If both sample and duplicate values are less than the CRQL, the RPD is not calculated. For soil/sediment sample or soil/sediment duplicate results less than five times the CRQL, enter the value of the CRQL, corrected for sample weight and percent solids (i.e., original, not duplicate sample weight), in the "Control Limit" column. If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*". In the instance where there is more than one duplicate sample per SDG, if one duplicate result is not within contract criteria, flag all samples of the same matrix in the SDG. The percent difference data will be used by USEPA to evaluate the long-term precision of the method. Specific control limits for the analyte will be added at a later date based on these precision results.

12.6 MDL Determination

12.6.1 Before any field samples are analyzed under this contract, the MDLs shall be determined for each digestion procedure and instrument used, prior to the start of contract analyses and annually thereafter. The MDLs shall meet the levels specified in Exhibit C. An MDL study shall be performed after major instrument maintenance, or changes in instrumentation or instrumental conditions, to verify the current sensitivity of the analysis.

12.6.1.1 To determine the MDLs, the Contractor shall run MDL studies following the procedures given in 40 CFR, Part 136. The Contractor shall prepare the MDL samples by each digestion procedure used and shall analyze these samples on each instrument used.

12.6.1.2 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C.

12.6.1.3 The results of the MDL determination studies shall be forwarded to the USEPA Regional CLP PO, Sample Management Office (SMO), and Quality Assurance Technical Support (QATS).

12.6.1.4 The MDL results shall be reported on Form IX-IN.

12.7 Example Analytical Sequence for Mercury

S0
S0.20
S1.0
S2.0
S5.0
S10.0
ICV
ICB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

12.8 Summary of QC Operations

The QC operations performed for mercury analysis are summarized in the table below.

QC Operation	**Frequency**
Instrument Calibration	Daily or each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment
Initial Calibration Verification	Following each instrument calibration.
Initial Calibration Blank	Following each instrument calibration, immediately after the ICV.
Continuing Calibration Verification	At a frequency of every hour of a run, and at the beginning and end of each run.
Continuing Calibration Blank	Every hour of a run, and at the beginning and end of each run. Performed immediately after the last CCV.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
Determination of Method Detection Limits	Prior to contract award, annually thereafter, and after major instrument adjustment.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 1.15 in Exhibit D - Introduction to Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 1.16 in Exhibit D - Introduction to Analytical Methods.

16.0 REFERENCES

16.1 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 245.1. 1974.

16.2 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 245.2. 1974.

16.3 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 245.5. 1974.

16.4 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste (SW-846), Method 7470A, Revision 1, September 1994.

16.5 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste (SW-846), Method 7471B, Revision 2, January 1998.

16.6 US Government Printing Office. 40 Code of Federal Regulations, Part 136. Section 1. Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Not applicable.

18.0 ALTERNATIVE SAMPLE PREPARATION PROCEDURES

18.1 Regionally-Selected Alternative Sample Preparation Procedures

Due to the variability of the matrices received and the interferences encountered, the sample preparation and analysis procedures described in Sections 10.1.4 and 10.1.5 may not always produce valid and/or reliable results. Sample preparation procedures are given in this section that describe alternative procedures that can be used if interferences or other issues are noted with the primary procedures. If the Contractor feels that one of these alternative sample preparation procedures would provide better results than the primary methods, then the Contractor shall contact the Sample Management Office (SMO). SMO will contact the USEPA Region for instructions. The Contractor shall document the USEPA Region's decision in the Sample Delivery Group (SDG) Narrative along with populating the Forms and the electronic data deliverable with the valid values that described the actual sample preparation procedures used by the Contractor.

18.2 Soil/Sediment Preparation and Analysis of Standards and Samples (Manual Technique - Based on modified version of USEPA OSW Method 7471B, Revision 2, January 1998)

18.2.1 This method was modified by using sulfuric acid in combination with nitric acid rather than using aqua regia, which is a combination of hydrochloric acid and nitric acid, as the digestion acid. This method is consistent with how soil/sediment standards and samples were digested in the previous Statements of Work and may provide better consistency of results for certain project samples that were prepared by the older method.

18.2.2 Manual Technique for the Preparation of Standards and Samples

18.2.2.1 For preparation of the standards, transfer aliquots of the working mercury solution (see Section 7.2.1.3) to a series of suitable digestion vessels. Add enough reagent water to each digestion vessel to make a total volume of 10 mL (±0.1 mL) and mix thoroughly. This working standard and the dilutions prepared from this working standard shall be prepared fresh daily. The acidity of all the standards should be maintained at 0.15% nitric acid. This acid should be added to the digestion vessel as needed prior to the dilution of the working standard.

18.2.2.2 For preparation of the samples, mix the samples thoroughly to achieve homogeneity (see Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh (to the nearest 0.01 g) an aliquot of 0.50-0.60 g and place in the bottom of a suitable digestion vessel. Add 5 mL of reagent water to each sample.

18.2.2.3 Add 5 mL of concentrated sulfuric acid and 2.5 mL of concentrated nitric acid and heat for 2 minutes at 95°C (±3°C) in a water bath or block digester. Cool, then add 50 mL reagent water, 15 mL potassium permanganate solution, and 8 mL of potassium persulfate solution to each sample. Mix thoroughly, then heat for an additional 30 minutes at 95°C (±3°C) in a water bath or block digester. Cool and add 6 mL of sodium chloride- hydroxylamine sulfate to each digestion vessel to reduce the excess potassium permanganate. CAUTION: Do this addition under a hood, as chlorine could be evolved. Add 55 mL of reagent water to each

sample or 50 mL of reagent water to each standard. All standards and samples must be at the same final volume. Reagent water can be used to make any final volume adjustments, if necessary.

18.2.2.4 At this point, the digested standards and samples can be allowed to stand for up to 48 hours prior to analysis. However, it is recommended that the digested standards and samples be analyzed as soon as possible. Refer to Section 10.1.4.2 for analysis.

EXHIBIT D - PART D

ANALYTICAL METHODS
FOR
TOTAL CYANIDE ANALYSIS

Exhibit D – Analytical Methods For Total Cyanide Analysis

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1.0 SCOPE AND APPLICATION

The method is designed to determine the concentration of total cyanide in aqueous/water and soil/sediment samples taken from hazardous waste sites.

2.0 SUMMARY OF METHOD

2.1 Aqueous/Water and Soil/Sediment

2.1.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation, using either a midi- or micro-distillation process, and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined spectrophotometrically.

2.1.2 In the semiautomated spectrophotometric measurement, the cyanide is converted to cyanogen chloride (CNCl), by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read between 570 and 580 nanometers (nm). To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

3.0 DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0 INTERFERENCES

Several interferences are encountered with this method. Some of the known interferences are aldehydes, nitrate-nitrite, oxidizing agents such as chlorine, thiocyanate, thiosulfate and sulfide. Some interferences are eliminated or reduced by using the distillation procedure. Some specific interferences that are commonly encountered are further discussed in Sections 4.1 through 4.4.

4.1 Sulfides

Sulfides adversely affect the spectrophotometric procedure. The sample should be tested in the field for the presence of sulfides as described in Section 8.1.1.

4.2 Surfactants

The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent, or equivalent, will prevent the foam from collecting in the condenser.

4.3 Oxidizing Agents

Oxidizing agents such as chlorine decompose most of the cyanides. The sample should be tested in the field for the presence of oxidizing agents as described in Section 8.1.1.

4.4 Nitrates-Nitrites

High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid that will react with some organic compounds to form oximes. These oximes will decompose under test conditions to generate HCN. The interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid prior to the distillation process.

5.0 SAFETY

See Section 1.14 in Exhibit D - Introduction to Analytical Methods.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1 Midi-Distillation of Aqueous/Water and Soil/Sediment Samples

6.1.1 Midi-reflux distillation apparatus

6.1.2 Heating block - Capable of maintaining 125°C (±5°C)

6.1.3 Auto analyzer system with accessories:

6.1.3.1 Sampler

6.1.3.2 Pump

6.1.3.3 Cyanide cartridge

6.1.3.4 Spectrophotometer with 50 mm flow cells and 580 nm filter

6.1.3.5 Chart recorder or data system

6.1.4 Assorted volumetric glassware, pipets, and micropipets (Class A)

6.1.5 Top loader balance, 300 g capacity, and a minimum sensitivity of ±1.0 mg

6.2 Micro-Distillation of Aqueous/Water and Soil/Sediment Samples

6.2.1 Micro-distillation apparatus

6.2.1.1 Heating block - capable of maintaining 120°C (±5°C)

6.2.1.2 Micro-distillation tubes - Sample tubes and Collector tubes, either pre-filled or user-filled with trapping solution

6.2.1.3 Tube press

6.2.2 Flow Injection Analyzer with accessories

6.2.2.1 Spectrophotometer with 580 nm filters

6.2.3 Assorted volumetric glassware, pipets, and micropipets (Class A)

6.2.4 Top loader balance, 300 g capacity and sensitivity of ±1.0 mg

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-06). Use this preparation for all reagents, standards, and dilutions of solutions.

7.1.2 Lead acetate Test Paper.

7.1.3 Cadmium carbonate (Powdered).

7.1.4 Potassium Iodide – Starch Test Paper.

7.1.5 Ascorbic acid (Crystals).

7.1.6 Midi-Distillation and Preparation Reagents for Aqueous/Water and Soil/Sediment Samples

7.1.6.1 Sodium hydroxide absorbing solution and sample wash solution, 0.25N - Dissolve 10.0 g sodium hydroxide in reagent water and dilute to 1 L.

7.1.6.2 Sodium hydroxide solution, 1.25N - Dissolve 50.0 g sodium hydroxide in reagent water and dilute to 1 L.

7.1.6.3 Sulfuric acid, 50% (v/v) - Carefully add a portion of concentrated sulfuric acid to an equal portion of reagent water.

7.1.6.4 Magnesium chloride solution (2.5M) – Weigh 510 g of $MgCl_2 \bullet 6H_2O$ into a 1000 milliliter (mL) flask, dissolve, and dilute to 1 L with reagent water.

7.1.6.5 Sulfamic acid (Powdered).

7.1.7 Micro-Distillation and Preparation Reagents for Aqueous/Water and Soil/Sediment Samples

7.1.7.1 Sodium hydroxide absorbing solution and sample wash solution, 0.25N – Dissolve 10.0 g sodium hydroxide in reagent water and dilute to 1 L.

7.1.7.2 Sodium hydroxide solution, 1.25N - Dissolve 50.0 g sodium hydroxide in reagent water and dilute to 1 L.

7.1.7.3 Sulfuric Acid/Magnesium Chloride solution (7.11 M sulfuric acid/0.79 M magnesium chloride) - In a fume hood, weigh 32.2 g $MgCl_2 \bullet 6H_20$ into a tared 500 mL beaker and add 110.8 g reagent water. Add 139 g concentrated sulfuric acid in 40 g portions with stirring. Allow the solution to cool.

7.1.7.4 Sulfamic acid (Powdered).

7.1.8 Spectrophotometric Reagents for Midi- and Micro-Distillation of Aqueous/Water and Soil/Sediment Samples

7.1.8.1 Chloramine-T solution (0.014M) - Dissolve 0.40 g of chloramine-T in reagent water and dilute to 100 mL. Prepare fresh daily.

7.1.8.2 Acetate Buffer - Dissolve 410 g of $NaC_2H_3O_2 \bullet 3H_2O$ in 500 mL of reagent water. Add sufficient glacial acetic acid to adjust pH to 4.5 (approximately 500 mL). An equivalent phosphate buffer may be substituted for the acetate buffer.

7.1.8.3 Pyridine-barbituric acid solution - Transfer 15 g of barbituric acid into a 1 Liter volumetric flask. Add about 100 mL of reagent water and swirl the flask. Add 75 mL of pyridine and mix. Add 15 mL of concentrated hydrochloric acid and mix.

Dilute to about 900 mL with reagent water and mix until the barbituric acid is dissolved. Dilute to 1 L with reagent water. Store at 4°C (±2°C).

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards, except as noted, to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit E, Section 8.0. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

7.2.2 Stock Standard Solutions

7.2.2.1 Stock cyanide solution, 1000 mg/L CN - Dissolve 2.51 g of potassium cyanide and 2.0 g potassium hydroxide in reagent water and dilute 1 L. Standardize with 0.0192N silver nitrate. Standardization is not necessary if this standard is purchased as a certified solution.

7.2.2.2 Intermediate cyanide standard solution, 10 mg/L CN - Dilute 1.0 mL of stock cyanide solution plus 20 mL of 1.25N sodium hydroxide solution to 100 mL with reagent water. Prepare this solution at time of analysis.

7.2.3 Secondary Dilution Standards

7.2.3.1 Secondary Dilution Standards

Prepare secondary dilution standard solutions by diluting the appropriate volumes of the intermediate cyanide standard solution with 0.25N sodium hydroxide. The final concentration of sodium hydroxide in all standards should be 0.25N.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

8.1.1 Aqueous/Water Sample Preservation

Collection of total cyanide must be in polyethylene or glass containers. The sample must be tested for sulfides and oxidizing agents, and preserved by the sampler immediately upon sample collection. Place a drop of the sample on lead acetate test paper to detect the presence of sulfides. If sulfides are present (test strip turns black), the sample volume required for the cyanide determination should be increased by 25 milliliters (mL). The total volume of sample should then be treated with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. If no sulfides are present, test for the presence of oxidizing agents by placing a drop of the sample on a strip of potassium iodide - starch test paper (KI - starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on

the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume. Preserve the sample with sodium hydroxide to pH greater than or equal to 12 and maintain at 4°C (±2°C) until distillation.

8.1.2 Soil/Sediment Sample Preservation

Samples shall be kept at 4°C (±2°C) from the time of collection until distillation.

8.2 Procedure for Sample Storage and Disposal

8.2.1 Aqueous/water samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery of a complete, reconciled data package to the U.S. Environmental Protection Agency (USEPA). After distillation, soil/sediment samples may be stored at room temperature within the laboratory until 60 days after delivery of a complete, reconciled data package to USEPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

8.2.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

8.2.3 Samples, sample distillates, and standards must be stored separately.

8.3 Contract Required Holding Time

The maximum sample holding time for cyanide is 12 days from Validated Time of Sample Receipt (VTSR).

9.0 CALIBRATION AND STANDARDIZATION

9.1 Instrument Operating Parameters

Because of the difference between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. The Method Detection Limit (MDL) and precision must be investigated and established for cyanide on that particular instrument. All measurements must be within the operational range of the instrument. It is the responsibility of the analyst to verify the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2 General Procedure

The following general procedure applies to most semi-automated spectrophotometers. Set up the manifold and complete system per manufacturer's instructions. Allow the spectrophotometer and recorder to warm up for at least 30 minutes prior to use. Establish a steady reagent baseline, feeding reagent water through the sample line and appropriate reagents (see Section 7.1.8) through reagent lines. Adjust the baseline using the appropriate control on the spectrophotometer. Prepare a standard curve by plotting the instrument response (e.g., absorbance) of standard on the Y-axis vs. concentration of the standard (in µg/L) on the X-axis.

9.3 Spectrophotometric Instrument Calibration Procedure

9.3.1 Instruments shall be calibrated daily or once every 24 hours, each time the instrument is set up, or after Initial Calibration Verification (ICV) or Continuing Calibration Verification (CCV) failure. The instrument standardization date and time shall be included in the instrument output.

9.3.2 The date and time of preparation and analysis shall be given in the laboratory logs (preparation) or instrument output (analysis).

9.3.3 Calibration standards shall be prepared fresh with each calibration performed. Calibrate the instrument with at least six standards, one of which must be a blank standard and one shall be at or below the Contract Required Quantitation Limit (CRQL), in graduated amounts in the appropriate range. The calibration curve shall be calculated using linear regression by plotting the concentration of cyanide in the standard (in µg/L) on the X-axis versus the instrument response (e.g., Absorbance) on the Y-axis. A standard linear regression, a weighted linear regression (e.g., 1/concentration or 1/concentration^2), or a linear regression with zero force calibration model can be used as appropriate. No other types of equations (e.g., quadratic) are to be used. The acceptance criteria for the calibration curve is a correlation coefficient greater than or equal to 0.995. Sample analysis shall not begin until this criteria and the criteria in Sections 9.3.4 and 9.3.5 have been met.

9.3.4 The calibration equation must be checked to establish the representativeness of the data that were used to produce the calibration equation. This check involves the re-fitting of the non-blank calibration data back to the calibration equation or the comparison of the calculated concentration of each of the standards against the expected concentration of the associated standard. This difference is related to the actual residual. For this calculation the Percent Difference shall be used where the above difference is further divided by the expected amount or concentration of the respective standard. If these Percent Differences for each of the standards do not fall within ±30%, then the calibration equation is not acceptable and must be corrected. If a standard is analyzed at a concentration that is below the CRQL and the above criteria is not met, that value can be excluded from the calibration curve as long as the lowest non-zero standard is still analyzed at or below the CRQL and all standards included in the calibration curve are continuous and consecutive.

9.3.5 The y-intercept from the linear regression initial calibration equation shall also be evaluated. If the y-intercept is not below the CRQL for the initial calibration curve, the calibration is not acceptable and must be corrected. Samples are not to be analyzed until the y-intercept for the initial calibration curve meets the acceptance criteria.

9.3.6 Any changes or corrections to the analytical system shall be followed by recalibration.

9.4 Initial Calibration Verification (ICV)

9.4.1 Immediately after each cyanide system has been calibrated, the accuracy of the initial calibration shall be verified and documented for cyanide by the analysis of the ICV Solution at the wavelength used for analysis.

9.4.2 The ICV Solution is obtained from USEPA. If the solution is not available, the ICV solution can be prepared by the laboratory using a certified solution of the analyte from an independent source. Analyses shall be conducted at a concentration other than that used for instrument calibration, but near the middle of the calibration range. An independent source is defined as a standard composed of the analyte from a different source than those used in the standards for the instrument calibration.

9.4.3 The ICV shall be processed in the same manner as the standards used for the initial calibration for the method used.

9.5 Continuing Calibration Verification (CCV)

9.5.1 To ensure calibration accuracy during each analytical run sequence, a CCV shall be analyzed and reported at a frequency not to exceed every 1 hour during an analytical run sequence. The standard shall also be analyzed and reported at the beginning of the analytical run sequence and after the last analytical sample. See the example sample analytical run sequence in Section 12.7. This CCV standard shall be prepared from the same source and at the same mid-level concentration as the standards used during the initial calibration.

The same CCV standard shall be used throughout the analytical run sequences for a Sample Delivery Group (SDG) of samples received.

9.5.2 Each CCV analyzed shall be processed in the same manner as the standards used for the initial calibration for the method used.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 If insufficient sample amount (less than 90% of the required amount) is received to perform the analyses, the Contractor shall contact the Sample Management Office (SMO) to inform them of the problem. SMO will contact the USEPA Region for instructions. The USEPA Region will either require that no sample analyses be performed or will require that a reduced volume be used for the sample analysis. No other changes in the analyses will be permitted. The Contractor shall document the USEPA Region’s decision in the Sample Delivery Group (SDG) Narrative.

10.1.2 If multi-phase samples (e.g., two-phase liquid sample, oily sludge/sandy soil/sediment sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the USEPA Region. If all phases of the sample are amenable to analysis, the USEPA Region may require the Contractor to do any of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.
- Separate the phases of the sample, and analyze one or more of the phases separately. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.1 If all of the phases are not amenable to analysis (i.e., outside scope), the USEPA Region may require the Contractor to do any of the following:

- Separate the phases and analyze the phase(s) that is (are) amenable to analysis. SMO will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.2 No other changes in the analyses will be permitted. The Contractor shall document the USEPA Region's decision in the SDG Narrative.

10.1.3 Before preparation is initiated for an aqueous/water sample, the Contractor shall test for the presence of sulfides and oxidizing agents (e.g., residual chlorine). The test for sulfides shall be performed by placing a drop of the sample on a strip of lead acetate paper. If the test strip turns black, the Contractor shall treat the total volume of sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates when the sample contains sulfide.

This operation shall be repeated until a drop of the treated sample solution does not darken the lead acetate test paper. The solution shall be filtered through dry filter paper into a dry beaker, and the volume of sample to be used for analysis shall be measured from the filtrate. It is recommended that the Contractor avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. The test for oxidizing agents shall be performed by placing a drop of the sample on a strip of potassium iodide - starch test paper (KI - starch paper). If the test strip turns blue, the Contractor shall contact SMO for further instructions from the USEPA Region before proceeding with sample preparation and analysis. The Contractor shall document the presence of sulfides or oxidizing agents in the SDG Narrative. The Contractor shall document the results (positive or negative) of the tests for sulfides and oxidizing agents in the distillation log.

10.2 Aqueous/Water and Soil/Sediment Preparation of Standards and Samples

10.2.1 Standards Preparation

10.2.1.1 All standards for the midi-distillation and micro-distillation semi-automated spectrophotometric analysis shall be distilled in the same manner as the samples.

10.2.1.2 Standards for Midi-Distillation Preparation and Semi-Automated Spectrophotometric Analysis of Aqueous/Water and Soil/Sediment Samples

Calibration standards - Prepare a minimum of five standards and a calibration blank over the range of the analysis. These standards shall be prepared by pipetting suitable volumes of the secondary dilution standard solution (see Section 7.2.3.1) into volumetric flasks and diluting to volume with 0.25N sodium hydroxide. Add 50 mL of each standard to a midi-distillation tube and then prepare and distill these standards and the calibration blank in the same manner as the samples.

NOTE: The concentration of one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL).

10.2.1.3 Standards for Micro-Distillation Preparation and Semi-Automated Spectrophotometric Analysis of Aqueous/Water and Soil/Sediment Samples

Calibration Standards - Prepare a minimum of five standards and a calibration blank over the range of the analysis. These standards shall be prepared by pipetting suitable volumes of the secondary dilution standard solution (see Section 7.2.3.1) into volumetric flasks and diluting to volume with 0.25N sodium hydroxide. Add 6 mL of each standard to a sample tube and then prepare and distill these standards and the calibration blank in the same manner as samples.

NOTE: The concentration of one of the calibration standards shall be at or below the CRQL.

10.2.2 Aqueous/Water Samples Preparation

10.2.2.1 Preparation Method for Aqueous/Water Samples by Midi-Distillation

10.2.2.1.1 The procedure described here utilizes a midi-distillation apparatus and requires a sample aliquot of 50 mL for aqueous/water samples. The sample shall not be diluted prior to distillation.

10.2.2.1.2 Pipet 50 mL (±1 mL) of sample into the distillation flask along with 2 or 3 boiling chips.

10.2.2.1.3 Add 50 mL (±1 mL) of 0.25N sodium hydroxide to the gas absorbing tube.

10.2.2.1.4 Connect the boiling flask, condenser, and absorber in the train. The excess cyanide trap contains 0.5N sodium hydroxide.

10.2.2.1.5 Turn on the vacuum and adjust the gang (Whitney) valves to give a flow of between 2 to 3 bubbles per second from the impingers in each reaction vessel.

10.2.2.1.6 If the samples contain nitrate and/or nitrite, add 0.2 g of sulfamic acid through the air inlet tube. Mix for 3 minutes prior to adding the sulfuric acid.

10.2.2.1.7 After 5 minutes of vacuum flow, inject 5 mL of 50% (v/v) sulfuric acid through the top air inlet tube of the distillation head into the reaction vessel. Allow the airflow to mix the reaction vessel contents for 5 minutes.

NOTE: The acid volume must be sufficient to bring the sample/solution pH to below 2.0.

10.2.2.1.8 Add 2 mL of the 2.5M magnesium chloride solution through the top air inlet tube of the distillation head into the reaction vessel. Excessive foaming from samples containing surfactants may be quelled by the addition of either another 2 mL of the 2.5M magnesium chloride solution or a few drops of a commercially available anti-foam agent. The Contractor shall document the addition of magnesium chloride solution or anti-foam agent in the SDG Narrative.

10.2.2.1.9 Turn on the heating block and set for 125°C (±3°C). Heat the solution to boiling, taking care to prevent solution backup by periodic adjustment of the vacuum flow.

10.2.2.1.10 After 1 1/2 hours of refluxing, turn off the heat and continue the vacuum for an additional 15 minutes. The flasks should be cool at this time.

10.2.2.1.11 After cooling, close off the vacuum at the gang valve and remove the absorber. Seal the receiving solutions and store them at 4°C until analyzed.

10.2.2.2 Preparation Method for Aqueous/Water Samples by Micro-Distillation

10.2.2.2.1 Preheat the heater block to 120°C (±3°C). Add 6 mL (±0.1 mL) of sample to the sample tube. The sample shall not be diluted prior to distillation. If the Contractor is not using the prefilled collector tubes, add 2 mL (±0.1 mL) of the 0.25N absorbing solution to each collector tube. Add 0.75 mL of the (7.11M/0.79M) sulfuric acid/magnesium chloride solution to each sample tube and immediately cap with a collector tube and press to seal.

10.2.2.2.2 Place the assembled tubes into the heater block and heat for 30 minutes. After 30 minutes, remove each tube from the block and immediately pull off the sample tube.

10.2.2.2.3 Invert each collector tube and allow to cool. Mix the distillate and detach the upper portion. Dilute the distillate to 6 mL (±0.1 mL) with absorbing solution and mix. The distillate is now ready for analysis. Seal the distillate and store at 4°C until analyzed.

10.2.3 Soil/Sediment Samples Preparation

10.2.3.1 Preparation Method for Soil/Sediment Samples by Midi-Distillation

10.2.3.1.1 The procedure described here utilizes a midi-distillation apparatus and requires a sample aliquot of at least 1 g for soil/sediment.

10.2.3.1.2 Mix the sample thoroughly to achieve homogeneity (see Exhibit D - Introduction to Analytical Methods, Section 1.8). For each digestion procedure, weigh to the nearest 0.01 g and transfer 1.00 - 1.50 g of sample (wet weight) into the reaction vessel and add 50 mL of reagent water. Add 2 or 3 boiling chips.

10.2.3.1.3 Add 50 mL (±1 mL) of 0.25N sodium hydroxide to the gas absorbing impinger.

10.2.3.1.4 Connect the reaction vessel, condenser, and absorber in the train. The excess cyanide trap contains 0.5N sodium hydroxide.

10.2.3.1.5 Turn on the vacuum and adjust the gang (Whitney) valves to give a flow of between 2 to 3 bubbles per second from the impingers in each reaction vessel.

10.2.3.1.6 After 5 minutes of vacuum flow, inject 5 mL of 50% (v/v) sulfuric acid through the top air inlet tube of the distillation head into the reaction vessel. Allow to mix for 5 minutes.

NOTE: The acid volume must be sufficient to bring the sample/solution pH to below 2.0.

10.2.3.1.7 Add 2 mL of the 2.5M magnesium chloride solution through the top air inlet tube of the distillation head into the reaction vessel. Excessive foaming from samples containing surfactants may be quelled by the addition of either another 2 mL of the 2.5M magnesium chloride solution or a few drops of a

commercially available anti-foam agent. The Contractor shall document the addition of the 2.5M magnesium chloride solution or anti-foam agent in the SDG Narrative.

10.2.3.1.8 Turn on the heating block and set for 125°C (±3°C). Heat the solution to boiling, taking care to prevent solution backup by periodic adjustment of the vacuum flow.

10.2.3.1.9 After 1 1/2 hours of refluxing, turn off the heat and continue the vacuum for an additional 15 minutes. The flasks should be cool at this time.

10.2.3.1.10 After cooling, close off the vacuum at the gang valve and remove the absorber. Seal the receiving solutions and store them at 4°C until analyzed.

10.2.3.2 Preparation Method for Soil/Sediment Samples by Micro-Distillation

10.2.3.2.1 Preheat the heater block to 120°C (±3°C). Add 0.50-1.00 g (±0.01 g) of sample (wet weight) and 5 mL of reagent water to the sample tube. If the Contractor is not using the prefilled collector tubes, add 2 mL (±0.1 mL) of the 0.25N absorbing solution to the collector tube. Add 0.75 mL of the (7.11M/0.79M) sulfuric acid/magnesium chloride solution to each sample tube and immediately cap with collector tube and press to seal.

10.2.3.2.2 Place the assembled tubes into the heater block and heat for 30 minutes. After 30 minutes, remove each tube from the block and immediately pull off the sample tube.

10.2.3.2.3 Invert each collector tube and allow to cool. Mix the distillate and detach the upper portion. Dilute the distillate to 6 mL (±0.1 mL) with absorbing solution and mix. The distillate is now ready for analysis. Seal the distillate and store at 4°C until analyzed.

10.3 Sample Analysis

10.3.1 Semi-Automated Spectrophotometric Determination of Distillates

10.3.1.1 Set up the manifold. Pump the reagents through the system until a steady baseline is obtained.

10.3.1.2 Place the distilled calibration standards, blanks, and control standards in the sampler tray, followed by the distilled samples, duplicates, standards, spikes, and blanks. Allow all standards and samples to come to ambient room temperature prior to analysis.

10.3.1.3 When a steady reagent baseline is obtained and before starting the sampler, adjust the baseline using the appropriate knob on the spectrophotometer. Aspirate the distilled blank calibration standard and adjust the spectrophotometer until the desired signal is obtained. Establish the baseline and proceed to analyze the remainder of the distilled standards and distilled samples.

10.3.1.4 Sample distillates having concentrations higher than the established calibration range as determined by the expected concentration of the highest calibration standard shall be diluted into range with the absorbing solution and reanalyzed.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Calculations for Midi- and Micro-Distillation of Aqueous/Water and Soil/Sediment Samples

11.1.1 Calculations for Semi-automated Spectrophotometric Determination

11.1.1.1 Prepare a standard curve by plotting the instrument response of the standards against their true concentrations in µg/L using linear regression as discussed in Section 9.3.3. This standard curve is then used to determine the concentration of cyanide in the field and Quality Control (QC) samples.

11.1.1.2 Calculate the cyanide concentration in aqueous/water samples by the formula:

EQ. 1 Aqueous/Water Sample Concentration

$$\text{CN Concentration (ug/L)} = C \times \frac{V_f}{V} \times DF$$

WHERE,
- C = Instrument response in µg/L CN from the calibration curve
- V_f = Final prepared (absorbing solution) volume (mL)
- V = Initial aliquot amount (mL)
- DF = Dilution Factor

11.1.1.3 Calculate the cyanide concentration in soil/sediment samples by the formula:

EQ. 2 Soil/Sediment Sample Concentration

$$\text{CN Concentration (mg/kg)} = C \times \frac{V_f}{W \times S} \times (1/1000) \times DF$$

WHERE,
- C = Instrument response in µg/L CN from the calibration curve
- V_f = Final prepared (absorbing solution) volume (mL)
- W = Initial aliquot amount (g)
- S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
- DF = Dilution Factor

11.1.1.4 Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation

To calculate the adjusted aqueous/water MDL or CRQL for the midi and micro methods, multiply the MDL or CRQL (µg/L) by the sample Dilution Factor.

The adjusted soil/sediment MDL or CRQL for the midi- and micro-distillation methods shall be calculated as follows:

EQ. 3 Adjusted Soil/Sediment MDL or CRQL

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_M}{W \times S} \times DF$$

WHERE,
- C = MDL or CRQL (mg/kg)
- W_M = Minimum method required aliquot amount (1.00 g for midi or 0.50 g for micro)
- W = Initial aliquot amount (g)
- S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
- DF = Dilution Factor

12.0 QUALITY CONTROL (QC)

12.1 Initial Calibration Verification (ICV)

The ICV standard shall be prepared in the same matrix as the calibration standards and in accordance with the instructions provided by the supplier. The ICV standard shall be processed in the same manner as the standards used for the initial calibration for the method used. If measurements exceed the control limits of 85% Recovery (low) or 115% Recovery (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. The results of the ICV analysis shall be reported.

12.2 Continuing Calibration Verification (CCV)

The CCV standard shall be prepared in the same matrix as the calibration standards by the analyst at a concentration equivalent to the mid-point of the calibration curve using the same standards that were used to prepare the initial calibration curve. The CCV standard shall be processed in the same manner as the standards used for the initial calibration for the method used. If the measurement exceeds the control limits of 85% Recovery (low) or 115% Recovery (high), the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration verification shall be performed. The results of all CCV analyses shall be reported.

12.3 Blank Analyses

There are two different types of blanks required by this method. The calibration blank is used in establishing the analytical curve. The Initial and Continuing Calibration Blanks (ICB/CCB) are identical in composition to the calibration blank and are analyzed immediately after the ICV/CCV to monitor for potential carryover of the analyte. The Preparation Blank is used to monitor for possible contamination throughout the entire sample preparation and analysis process.

12.3.1 Initial and Continuing Calibration Blank (ICB/CCB)

The ICB and CCB are identical in composition to the Calibration Blank as used to establish the initial calibration curve. The ICB/CCB shall be processed in the same manner as the standards used for the initial calibration for the method used. If the absolute value of the calibration blank (ICB/CCB) result exceeds the Contract Required Quantitation Limit (CRQL) (see Exhibit C), the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration blank shall be performed. Results of the ICB and CCB analyses shall be reported.

12.3.2 Preparation Blank

12.3.2.1 The Preparation Blank shall contain all the reagents and in the same volumes as used in processing the samples. The Preparation Blank shall be carried through the complete preparation, distillation, and analysis method process.

12.3.2.2 At least one Preparation Blank, consisting of reagent water processed through each sample preparation and analysis procedure (see Section 10), shall be prepared and analyzed with every Sample Delivery Group (SDG), or with each batch[1] of samples distilled, whichever is more frequent.

12.3.2.3 The first batch of samples in an SDG is to be assigned to Preparation Blank one, the second batch of samples to Preparation Blank two, etc. Each Complete SDG File (CSF) shall contain the results of all the Preparation Blank analyses associated with the samples in that SDG.

12.3.2.4 The Preparation Blank(s) is (are) to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

12.3.2.4.1 If the absolute value of the concentration of the blank is less than or equal to the CRQL (see Exhibit C), no further action is required.

12.3.2.4.2 If the analyte concentration in the blank is above the CRQL, the lowest concentration of the analyte in the associated samples (except those identified as field blanks) shall be greater than or equal to 10 times the blank concentration. Otherwise, all samples (except those identified as field blanks) associated with the blank, with the analyte concentration less than 10 times the blank concentration and above the CRQL, shall be redistilled and reanalyzed with appropriate new QC. The sample concentration is not to be corrected for the blank value.

12.3.2.4.3 If the concentration of the blank is below the negative CRQL, then all samples associated with the blank and reported below 10 times CRQL shall be reprepared and reanalyzed with appropriate new QC.

12.3.2.4.4 The results for the Preparation Blank shall be reported.

12.4 Spike Sample Analysis

12.4.1 The spike sample analysis is designed to provide information about the effect of the sample matrix on the distillation and/or measurement methodology. The spike is added before the distillation step and prior to the addition of other reagents. At least one spike sample analysis (matrix spike) shall be performed on each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[2] The sample and its associated spike sample shall initially be run at the same dilution.

12.4.2 If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 12.5). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks and Performance Evaluation (PE) samples shall not be used for spiked sample

[1] A group of samples prepared at the same time.

[2] USEPA may require additional spike sample analyses, upon USEPA Regional CLP Project Officer (CLP PO) request.

analysis. USEPA may require that a specific sample be used for the spike sample analysis.

12.4.3 The analyte spike shall be added to achieve a concentration of 100 µg/L in the final sample solution prepared for analysis (i.e., post-distillation). For example, the midi-distillation procedure would require the addition of 5 µg of cyanide to the sample prior to distillation (based on the final distillate volume of 50 mL). For a typical 50 mL aqueous/water sample, this would be equivalent to a concentration of 100 µg/L in the original sample. For a typical 1.00 g soil/sediment sample, this would be equivalent to a concentration of 5 mg/kg in the original dry sample. Adjustments shall be made to maintain these spiking levels when the weight of the sample taken deviates by more than 10% of these values.

12.4.4 If the spike recovery is not at or within the limits of 75-125%, the data for all samples received and associated with that spike sample and determined by the same analytical method shall be flagged with the letter "N". An exception to this rule is granted when the sample concentration exceeds the Spike Added (SA) concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

12.4.5 When the matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-distillation spike shall be performed. Note that if a post-distillation spike analysis is required, the same USEPA sample that was used for the matrix spike analysis shall be used for the post-digestion spike analysis. Spike the unspiked aliquot of the undiluted distillate at two times the indigenous level or two times the CRQL, whichever is greater.

12.4.6 In the instance where there is more than one spike sample per matrix, per method, per SDG, if one spike sample recovery is not within contract criteria, flag all the samples of the same matrix and method in the SDG. Individual component percent recoveries are calculated as follows:

EQ. 4 Matrix Spike and Post-Digestion Spike Percent Recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

WHERE, SSR = Spiked Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2
SR = Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2
SA = Spike Added Theoretical Result (µg/L or mg/kg). This is calculated by substituting the spiking amount used for the 'V_f' term and substituting the spiking standard concentration used for the 'C' term from EQ. 1 or EQ.2.

12.4.7 When the sample concentration is less than the Method Detection Limit (MDL), use SR = 0 only for purposes of calculating the percent recovery. The Spike Sample Results (SSRs), Sample Results (SRs), Spike Added (SA), and percent recovery (positive or negative) shall be reported.

12.4.8 The units used for reporting Spike Sample Results (SSRs) will be identical to those used for reporting Sample Results (SRs).

12.5 Duplicate Sample Analysis

12.5.1 One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[3] Duplicates cannot be averaged for reporting.

12.5.2 Duplicate sample analyses are not required for percent solids. Samples identified as field blanks and PE samples shall not be used for duplicate sample analysis. USEPA may require that a specific sample be used for duplicate sample analysis. The Relative Percent Difference (RPD) is calculated as follows:

EQ. 5 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

WHERE, RPD = Relative Percent Difference
S = Sample Result (original) (µg/L or mg/kg) from EQ. 1 or EQ. 2
D = Duplicate Sample Result (µg/L or mg/kg) from EQ. 1 or EQ. 2

12.5.3 The results of the duplicate sample analyses shall be reported. A control limit of 20 for RPD shall be used for original and duplicate sample values greater than or equal to five times the CRQL (see Exhibit C). A control limit equal to the CRQL value shall be entered in the "Control Limit" column if either the sample or duplicate value is less than five times the CRQL. If the sample and duplicate values are greater than or equal to five times the CRQL, or if the sample and duplicate values are less than the CRQL, the "Control Limit" field is left empty.

12.5.4 If one result is above five times the CRQL level and the other is below, use the CRQL criteria to determine if the duplicate analysis is in control. If both sample and duplicate values are less than the CRQL, the RPD is not calculated. For soil/sediment sample or soil/sediment duplicate results less than five times the CRQL, enter the value of the CRQL, corrected for sample weight and percent solids, (i.e., original, not duplicate sample weight), in the "Control Limit" column. If the duplicate sample results are outside the control limits, flag all the data for samples received and associated with that duplicate sample with an "*". In the instance where there is more than one duplicate sample per SDG, if one duplicate result is not within contract criteria, flag all samples of the same matrix and method in the SDG. The percent difference data will be used by USEPA to evaluate the long-term precision of the method. Specific control limits for this analyte will be added at a later date based on the precision results.

[3] USEPA may require additional duplicate sample analyses, upon USEPA Regional CLP PO request.

12.6 MDL Determination

12.6.1 Before any field samples are analyzed under this contract, the MDLs shall be determined for each distillation procedure and instrument used, prior to the start of contract analyses, and annually thereafter. The MDLs shall meet the levels specified in Exhibit C. An MDL study shall be performed after major instrument maintenance, or changes in instrumentation or instrumental conditions, to verify the current sensitivity of the analysis.

12.6.1.1 To determine the MDLs, the Contractor shall run MDL studies following the procedures given in 40 CFR, Part 136. The Contractor shall prepare the MDL samples by each digestion procedure used and shall analyze these samples on each instrument used.

12.6.1.2 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C.

12.6.1.3 The results of the MDL determination studies shall be forwarded to the USEPA Regional CLP PO, Sample Management Office (SMO), and Quality Assurance Technical Support (QATS).

12.6.1.4 The MDL results shall be reported on Form IX-IN.

12.7 Example Analytical Sequence for Cyanide

S0
S10
S50
S100
S200
S400
ICV
ICB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

12.8 Summary of QC Operations

The QC operations performed for cyanide analysis are summarized in the table below.

QC Operation	Frequency
Instrument Calibration	Daily or each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment.
Initial Calibration Verification	Following each instrument calibration.
Initial Calibration Blank	Following each instrument calibration, immediately after the ICV.

QC Operation	Frequency
Continuing Calibration Blank	Every hour of a run, and at the beginning and end of each run. Performed immediately after the last CCV.
Continuing Calibration Verification	At a frequency of every hour of a run, and at the beginning and end of each run.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Post-Digestion/Distillation Spike	Each time Spike Sample Recovery is outside QC limits.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
Determination of Method Detection Limits	Prior to contract award, annually thereafter, and after major instrument adjustment.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 1.15 in Exhibit D - Introduction to Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 1.16 in Exhibit D - Introduction to Analytical Methods.

16.0 REFERENCES

16.1 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 335.4, Revision 1.0, August 1993.

16.2 American Water Works Association/American Public Health Association/Water Environment Federation. Standard Methods for the Examination of Water and Wastewater. Method 4500. 18th Edition.

16.3 US Government Printing Office. 40 Code of Federal Regulations, Part 136. Section 1. Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Not applicable.

USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

INORGANIC SUPERFUND METHODS

(Multi-Media, Multi-Concentration)

ISM01.2

January 2010

STATEMENT OF WORK

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EXHIBIT A

SUMMARY OF REQUIREMENTS

Exhibit A - Summary of Requirements

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1.0 PURPOSE

The purpose of the multi-media, multi-concentration inorganic analytical service is to provide analytical data for use by the U.S. Environmental Protection Agency (USEPA) in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). Other USEPA Program Offices that have similar analytical data needs can also use this service.

2.0 DESCRIPTION OF SERVICE

The inorganic analytical service provides a contractual framework for laboratories. This framework applies USEPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 23 metals (including mercury) and cyanide in aqueous/water and soil/sediment samples. The Statement of Work (SOW) also includes methods for total metals analysis in wipes and air filters. The analytical service contract provides specific contractual requirements by which USEPA will evaluate the data.

3.0 DATA USES

This analytical service contract provides data which USEPA uses for a variety of purposes, such as: determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including: site inspections, Hazard Ranking System (HRS) scoring, remedial investigation/feasibility studies, remedial design, treatability studies, and removal actions.

The data may also be used in litigation against Potentially Responsible Parties in the enforcement of Superfund legislation. As a result, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, since it is used to make major decisions regarding public health and environmental welfare. The Contractor may be required to appear and testify to the accuracy and/or validity of the data generated.

4.0 SUMMARY OF REQUIREMENTS

4.1 Introduction to the Inorganic Statement of Work

The Statement of Work (SOW) is comprised of eight Exhibits. Exhibit A provides an overview of the SOW and its general requirements. Exhibit B contains a description of the reporting and deliverables requirements, in addition to the data reporting forms and instructions. Exhibit C specifies the Inorganic Target Analyte List (TAL) for this SOW with the Contract Required Quantitation Limits (CRQLs) for the sample matrices. Exhibit D details the required analytical procedures to be used with this SOW and resulting contracts. Exhibit E provides descriptions of required Quality Assurance/Quality Control (QA/QC), Standard Operating Procedures (SOPs), QA/QC performance, and the reporting of data. Exhibit F contains chain-of-custody and sample documentation requirements. To ensure proper understanding of the terms utilized in this SOW, a glossary can be found in Exhibit G. When a term is used in the text without explanation, the glossary meaning shall be applicable. Specifications for electronically reporting data appear in Exhibit H.

4.2 Overview of Major Task Areas

For each sample, the Contractor shall perform the tasks described in each section. Specific requirements for each task are detailed in the Exhibits referenced in the following sections.

4.2.1 Task I: Sample Receiving, Storage, and Disposal

4.2.1.1 Chain-of-Custody

The Contractor shall receive and maintain samples under proper chain-of-custody. All associated document control and inventory procedures shall be developed and followed. Documentation described herein shall be required to show that all procedures are strictly followed. Inventory procedures shall include documenting sample location and transfer within the laboratory. This documentation shall be reported as the Complete Sample Delivery Group (SDG) File (CSF) (see Exhibit B). The Contractor shall establish and use appropriate procedures to safeguard confidential information received from USEPA.

4.2.1.2 Sample Scheduling/Shipments

Sample shipments to the Contractor’s facility will be scheduled and coordinated by the Contract Laboratory Program (CLP) Sample Management Office (SMO). USEPA may request analyses that include all or a subset of the Inorganic Target Analytes listed in Exhibit C. USEPA may also request modified analyses due to the nature of the samples received or project requirements. The Contractor shall communicate with SMO as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

4.2.1.2.1 Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing of the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier within the Contractor’s geographical area. The Contractor shall be available to receive and process sample

shipments at any time the delivery service is operating, including Saturdays, to ensure that short sample preparation and analysis time requirements can be met.

4.2.1.2.2 If there are problems with the samples (e.g., mixed media, containers broken or leaking), coolers (e.g., scheduled but not delivered), or sample documentation and paperwork (e.g., Traffic Reports/Chain of Custody Records not with shipment, sample and Traffic Report/Chain of Custody Record do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

4.2.1.2.3 To monitor the temperature of the sample shipping cooler more effectively, each USEPA Regional Office may include a sample shipping cooler temperature indicator bottle with each cooler shipped. The temperature indicator will be clearly labeled: COOLER TEMPERATURE INDICATOR BOTTLE. The Contractor shall record the presence or absence of the cooler temperature indicator bottle on Form DC-1, Item 8 - Cooler Temperature Indicator Bottle (see Exhibit B).

4.2.1.2.3.1 When the USEPA Regional Office supplies a cooler temperature indicator bottle in the sample shipping cooler, the Contractor shall use the USEPA supplied cooler temperature indicator bottle to determine the cooler temperature. The temperature of the cooler shall be measured at the time of sample receipt by the Contractor.

4.2.1.2.3.2 The temperature of the sample shipping cooler shall be measured and recorded immediately upon opening the cooler.

To determine the temperature of the cooler, the Contractor shall locate the cooler temperature indicator bottle in the sample shipping cooler, invert it several times, remove the cap, and insert a calibrated thermometer into the cooler temperature indicator bottle. The temperature can then be read after the reading has stabilized. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the calibrated thermometer (±1°C) shall have a measurable range of 0-50°C. Other devices which can measure temperature may be used if they can be calibrated to ±1°C and have a range of 0-50°C. If a temperature indicator bottle is not present in the cooler, an alternative means of determining cooler temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining cooler temperature. The Contractor shall contact SMO and inform them that a temperature indicator bottle was not present in the cooler. The Contractor shall document the alternative technique used to determine cooler temperature in the SDG Narrative.

4.2.1.2.3.3 If the temperature of the sample shipping cooler's temperature indicator exceeds 10°C, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the USEPA Region from which the samples were shipped for instruction on how to proceed. The Region will either require that no sample analysis(es) be performed or that the Contractor proceed with the analysis(es). SMO will in turn notify the Contractor of the Region's decision. The Contractor shall document the Region's decision and the EPA sample numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.

4.2.1.2.3.4 The Contractor shall record the temperature of the cooler on Form DC-1, under Item 9 - Cooler Temperature (see Exhibit B).

4.2.1.2.4 The Contractor is required to retain unused sample volume in the original containers for a period of 60 days after data submission. From time of receipt until analysis, the Contractor shall maintain <u>all</u> aqueous/water (preserved and unpreserved) and soil/sediment samples at 4°C (±2°C) (see Exhibit D). Wipe and filter samples may be stored at room temperature within the laboratory until preparation.

4.2.1.2.5 The Contractor shall be required to routinely return sample shipping containers (e.g., coolers) to the appropriate sampling office within 14 calendar days following shipment receipt (see contract Section G titled, "Government Furnished Samples").

4.2.1.2.6 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case.

4.2.1.2.6.1 A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:

- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- All samples scheduled with the same level of deliverables.
- In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG.

The laboratory shall take all precautions to meet the 20 sample per SDG criteria.

4.2.1.2.6.2 Samples may be assigned to SDGs by matrix (i.e., all soil/sediment in one SDG, all aqueous/water in another), at the discretion of the laboratory. However, PE samples received within a Case shall be assigned to an SDG containing field samples for that Case. Such assignment shall be made at the time the samples are received, and shall not be made retroactively.

4.2.1.2.6.3 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report/Chain of Custody Record bearing the sample number and descriptive information regarding the sample. EPA sample numbers are continuous, without spaces or hyphens. The Contractor shall complete and sign the Traffic Report/Chain of Custody Record, recording the date of sample receipt and sample condition on receipt for each sample container. The Contractor shall also follow the instructions given on the Traffic Report/Chain of Custody Record in choosing the Quality Control (QC) samples when such information is provided. If no QC sample is designated on the Traffic Report/Chain of Custody Record, the Contractor shall select a sample and notify SMO for USEPA Regional acceptance. SMO shall contact the Region for confirmation immediately after notification.

4.2.1.2.6.4 The Contractor shall submit signed copies of Traffic Reports/Chain of Custody Records for all samples in an SDG to SMO within **three working days** following receipt of the last sample in the SDG. Faxed copies of Traffic Reports/Chain of Custody Records do not meet this requirement. Traffic Reports/Chain of Custody Records shall be submitted in SDG sets (i.e., all Traffic Reports/Chain of Custody Records for an SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

4.2.1.2.6.5 EPA Case numbers, SDG numbers, and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally and in reports/correspondence.

4.2.1.3 Modified Analysis

The Contractor may be requested by USEPA to perform modified analyses. These modifications will be within the scope of this SOW and may include, but are not limited to: modified preparation, cleanup, or analysis procedures; analysis of additional analytes; additional sample matrices; and/or lower quantitation limits. These requests will be made in writing, prior to sample scheduling. All contract requirements specified in the SOW/Specifications will remain in effect unless specifically modified.

4.2.2 Task II: Sample Preparation and Analysis

4.2.2.1 Overview

The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high (greater than 15%) levels of organic and inorganic materials of a potentially hazardous nature and of

unknown structure and concentration, and should be handled throughout the analysis with appropriate caution. It is the Contractor's responsibility to take all necessary measures to ensure laboratory safety.

4.2.2.2 The Contractor shall prepare and analyze samples as described in Exhibit D. Sample preparation methods shall remain consistent for all samples analyzed within a Case. Prior to sample analysis, the Contractor shall review the Traffic Report/Chain of Custody Record for any special sample analysis instructions. Anomalies that occur during sample analysis shall be reported to SMO immediately.

The Contractor shall collectively review all analytical results associated with a sample. This includes undiluted, diluted, serial dilution, and interference results. The Contractor shall report any significant anomalies between these results in the SDG Narrative indicating possible matrix interferences.

4.2.2.3 Quality Assurance/Quality Control Procedures

4.2.2.3.1 The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D and E. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibits B and H.

4.2.2.3.2 The Contractor shall maintain a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection, as well as the quality assessment measures performed by management, to ensure acceptable data production.

4.2.2.3.3 Additional QC shall be conducted in the form of the analysis of laboratory PE samples submitted to the laboratory by USEPA. Unacceptable results of any such QC or laboratory PE samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to USEPA <u>or</u> rejection of the data for specific analyte(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values as determined by USEPA, as well as meeting the contract requirements for analysis (Exhibit D); QA/QC (Exhibit E); data reporting and other deliverables (Exhibits B and H); and sample custody, sample documentation, and SOP documentation (Exhibit F).

4.2.3 Task III: Sample Reporting

4.2.3.1 USEPA has provided to the Contractor formats for the reporting of data (Exhibits B and H). The Contractor shall be responsible for completing and submitting analysis data sheets and electronic data as requested in a format specified in this SOW and within the time specified in Exhibit B, Section 1.1.

4.2.3.2 Use of formats other than those designated by USEPA (see Exhibits B and H) will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to the Government shall be required.

4.2.3.3 Computer generated forms may be submitted in the hardcopy CSF(s) provided that the forms provide equivalent information as the **USEPA format**. This means that the order of data elements is the same as on each USEPA required form, including form numbers and titles, page numbers, and header information.

4.2.3.4 The data reported by the Contractor on the hardcopy data forms and the associated electronic data submitted by the Contractor shall contain identical information. If discrepancies are found during Government inspection, the Contractor shall be required to resubmit either the corrected hardcopy forms or the corrected electronic data, or both sets of corrected data, at no additional cost to USEPA.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

Exhibit B – Reporting and Deliverables Requirements

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1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 Report Deliverable Schedule

The following table reiterates the contract reporting and deliverables requirements, and specifies the distribution that is required for each deliverable. The turnaround times for Items B through E are 7, 14, or 21 days.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The U.S. Environmental Protection Agency (USEPA) Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM) will notify the Contractor in writing of such changes when they occur.

TABLE 1

Item		No. of Copies[A]	Delivery Schedule	Distribution	
				SMO	Region
A.	Sample Traffic Reports/Chain of Custody Records	1	3 working days after receipt of last sample in Sample Delivery Group (SDG).[1]	X	
B.[2]	Complete SDG File (CSF)	1	XX[C] days after Validated Time of Sample Receipt (VTSR) of last sample in SDG.[1]		X
C.[2,3]	Copy of CSF and Hardcopy Data in PDF Format[B]	1	XX[C] days after VTSR of last sample in SDG.	X	
D.[2,4]	Preliminary Results	1	Within 48 hours after receipt of each sample at laboratory, if requested.	X	X
E.[2]	Electronic Data Deliverable	1	XX[C] days after VTSR of last sample in SDG.	X	
F.[2]	Results of Intercomparison Study/PE Sample Analysis Study	1	XX[C] days after VTSR of last sample in SDG.	As Directed	
G.[5,6]	Annual Determination of Method Detection Limits (MDL) And Calculation of ICP-AES Interelement Correction Factors	1	Annually	As Directed	

TABLE 1 (Con't)

Item		No. of Copies[A]	Delivery Schedule	Distribution	
				SMO	Region
H.[6,7]	Standard Operating Procedures (SOPs)	1	Revise within 30 days after contract award and receipt of USEPA comments. Submit the latest version within 7 days of receipt of written request to recipients as directed. (See Exhibit E, Section 6) Submit the amended document within 14 days of amended SOP(s) as directed in Exhibit E, Section 6.4.	As Directed	
I.[6,7]	Quality Assurance Plan (QAP)	1	Revise within 30 days after contract award and receipt of USEPA comments. Submit the latest version within 7 days of receipt of written request to recipients as directed. (See Exhibit E, Section 5) Submit the amended document within 14 days of amended QAP as directed in Exhibit E, Section 5.3.	As Directed	
J.	Instrument Electronic Data	Lot	Retain for 3 years after data submission. Submit within 7 days after receipt of written request by the USEPA Regional CLP PO. (See Exhibit E, Section 13)	As Directed	
K.	Digestates	Lot	Retain for total metals 180 days after data submission. Submit within 7 days after receipt of written request by CLP PO or SMO at USEPA's direction.	As Directed	

Footnotes:

[A] The number of copies specified is the number of copies required to be delivered to each recipient.

[B] Contractor-concurrent delivery to USEPA's designated recipient [e.g., Quality Assurance Technical Support (QATS)] may be required upon request by the USEPA Regional Contract Laboratory Program Project Officer (CLP PO). Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the CLP PO. Supplemental data (i.e., logbooks) may be requested in writing from the USEPA Regional staff or QATS. All written communication sent by USEPA must include the laboratory's CLP PO in the distribution list. If the CLP PO has not been included in the distribution list, contact the OSRTI ASB Inorganic Program Manger.

[C] The number of days associated with these elements will be provided in the associated laboratory contract document and will also be provided at the time of sample scheduling by the Sample Management Office (SMO) Contractor.

[1] Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less (3 calendar day period for 7-day turnaround) with the same laboratory turnaround not exceeding 20 samples [excluding Performance Evaluation (PE) samples] and scheduled with the same level of deliverables. Data for all samples in the SDG are due concurrently to USEPA and SMO (or as designated). The date of delivery of the SDG, or any samples within the SDG, is the date that the last sample in the SDG is received. See Exhibit A for further description. Validated Time of Sample Receipt (VTSR) is the date of sample receipt at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report/Chain of Custody Record.

[2] **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE.** Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. This includes resubmission of both the hardcopy and electronic deliverable. The date of delivery of the SDG, or any sample within the SDG, is the date that all samples have been delivered. **If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables received after this time will be considered late.**

[3] Complete SDG File (CSF) will contain the original Sample Data for Level 2a or 2b deliverables, plus all of the original documents described in Exhibit B, Section 2.6.

[4] If requested at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of Form I sample analyses for field and Quality Control (QC) sample analyses via facsimile or email. The Contractor may submit Preliminary Results in electronic format [i.e., Portable Document Format (PDF), Microsoft Word, etc.) after obtaining permission from USEPA. The Contractor shall be notified of the Fax number or email address at the time of sample scheduling. The Contractor shall also be provided a backup Fax number or email address. Sample Traffic Reports/Chain of Custody Records (TRs/COCs) and SDG Cover Sheets shall be submitted with the Preliminary Results. The Contractor shall contact SMO after confirming transmission. The Contractor shall document all communication in a communication log.

Preliminary Results Delivery Schedule:

If a sample requiring Preliminary Results arrives at the laboratory before 5 p.m., the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received at the laboratory after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day. **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered the next business day. Deliverables reported after this time will be considered late.**

[5] Also required in each CSF.

[6] See Exhibit E for description. Time is cited in calendar days.

[7] The Contractor shall deliver both hardcopy and electronic (e.g., diskette/CD) copies of the Standard Operating Procedures (SOPs) and Quality Assurance Plan (QAP).

1.2 Distribution

The following addresses correspond to the "Distribution" column in Exhibit B, Section 1.1, Table 1.

SMO: USEPA Contract Laboratory Program (CLP)
Sample Management Office (SMO)[1]
15000 Conference Center Drive
Chantilly, VA 20151-3808

Region: USEPA REGIONS: SMO will provide the Contractor with the list of addressees for data delivery for the 10 USEPA Regions. SMO will provide the Contractor with updated USEPA Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

USEPA Regional CLP Project Officer (CLP PO):

SMO will provide the Contractor with the list of addresses for the USEPA Regional CLP POs. SMO will provide the Contractor with updated name/address lists as necessary throughout the period of the contract.

QATS: USEPA Contract Laboratory Program (CLP)
Quality Assurance Technical Support (QATS)[2]
2700 Chandler Avenue, Building C
Las Vegas, NV 89120
Attn: Data Audit Staff

[1] SMO is a Contractor-operated facility operating under the SMO contract awarded and administered by USEPA.

[2] The QATS laboratory is a Contractor-operated facility operating under the QATS contract awarded and administered by USEPA.

In addition, the mailing and delivery addresses for the USEPA ASB Inorganic Program Manager (ASB PM) are:

Mailing Address:	USEPA OSRTI Analytical Services Branch Ariel Rios Building (5203P) 1200 Pennsylvania Avenue, N.W. Washington, DC 20460 Attn: CLP Inorganic Program Manager
Fed-Ex/Overnight Delivery:	USEPA OSRTI Analytical Services Branch One Potomac Yard (South Building) 2777 South Crystal Drive 4th Floor, S4838 Arlington, VA 22202 Attn: CLP Inorganic Program Manager

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1 Introduction

The Contractor shall provide reports and other deliverables as specified in Exhibit B, Section 1.1. The required content and form of each deliverable is described in this Exhibit. All reports and documentation **shall be**:

- Legible;
- Clearly labeled and completed in accordance with instructions in this Exhibit;
- Arranged in the order specified in this Exhibit;
- Paginated sequentially according to instructions in this Exhibit; and
- Double-sided.
- Information reported on the forms listed in this Exhibit [excluding the Sample Log-In Sheet (DC-1) and the Complete SDG File (CSF)Inventory Sheet (DC-2)] must be computer-generated.

2.1.1 The Contractor shall use EPA Case numbers, SDG numbers, and EPA sample numbers to identify samples received under this contract, both verbally and in reports and correspondence. The contract number and the Statement of Work (SOW) number shall be specified in all correspondence. The Modification Reference Number (Mod. Ref. No.) shall also be included for all Modified Analyses.

2.1.2 The Contractor shall submit either Level 2a or 2b deliverables as specified at the time of scheduling. Level 2b deliverables include all data reporting forms and supporting raw data as specified in this Exhibit. Level 2a deliverables consist of a specified limited subset of the data reporting forms and supporting documents as specified in this Exhibit.

2.1.3 Section 4 of this Exhibit contains the required Data Reporting Forms in Agency-specified format. Section 3 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide USEPA with all required data. Data elements and instructions for electronically reporting data are contained in Exhibit H.

2.2 Resubmission of Data

If submitted documentation does not conform to the above criteria, the Contractor is required to resubmit such documentation with deficiency (ies) corrected within 6 business days, at no additional cost to USEPA.

2.2.1 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, through a USEPA Regional CLP Project Officer (CLP PO) action, or through a USEPA Regional data reviewer’s request, the data shall be clearly marked as "Additional Data" and shall be sent to both contractual data recipients [Sample Management Office (SMO) and USEPA Region] and to USEPA’s designated recipient [e.g., Quality Assurance Technical Support (QATS)] when a written request for a copy of the CSF has been made. A cover letter shall be included which describes what data is being delivered, to which USEPA Case(s) the data pertains, and **who requested the data**.

2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, through the USEPA Regional CLP Project Officer (CLP PO) action, or through a USEPA Regional data reviewer's request, the data shall be clearly marked as "Additional Data" and shall be sent to both contractual data recipients (SMO and USEPA Region) and to USEPA's designated recipient (e.g., QATS) when a written request for a copy of the CSF has been made. A cover letter shall be included which describes what data is being delivered, to which USEPA Case(s) the data pertains, and **who requested the data**.

2.2.3 Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data shall be sent to the two contractual data recipients (SMO and USEPA Region) and to USEPA's designated recipient (e.g., QATS) when a written request for a copy of the CSF has been made. In all instances, the Contractor shall include a cover sheet (Laboratory Response to Results of Contract Compliance Screening). Electronic deliverables shall be submitted or resubmitted to SMO only. Revised DC-1 and DC-2 forms shall be resubmitted to SMO and the USEPA Region.

2.3 Quality Assurance (QA) Management Plan and Standard Operating Procedures (SOPs)

The Contractor shall adhere to the requirements in Exhibits E and F.

2.4 Sample Traffic Reports/Chain of Custody Records

Each sample received by the Contractor will be labeled with an EPA sample number and will be accompanied by a Sample Traffic Report/Chain of Custody Record bearing the sample number and descriptive information regarding the sample. The current CLP Traffic Report is the "Inorganic Traffic Report & Chain of Custody Record". The CLP Traffic Report/Chain of Custody Record is one form divided into two sections: the Traffic Report section and the Chain of Custody Record section. The Contractor shall complete the CLP Traffic Report/Chain of Custody Record (marked "Lab Copy for Return to SMO"), recording the date of sample receipt, verifying the number of samples, and signing the CLP Traffic Report/Chain of Custody Record.

Upon receipt, the Contractor shall sign for the receipt of samples in the Chain of Custody Record section. The laboratory sample custodian or designated recipient opening and verifying the contents of the cooler shall then verify the receipt of all samples identified within the CLP Traffic Report section, and sign and date the signature box located in the CLP Traffic Report section. If a non-CLP Traffic Report/Chain of Custody Record is submitted with the samples (e.g., a Regional Traffic Report/Chain of Custody Record), then the Contractor shall (1) sign and date receipt of the samples to maintain the chain-of-custody and (2) the sample custodian or designated recipient shall sign and date the Traffic Report/Chain of Custody Record to verify sample information.

The Contractor shall also enter the Sample Delivery Group (SDG) number, Case number, and the laboratory contract number on the CLP Traffic Report/Chain of Custody Record, in the appropriate boxes. The EPA sample number of the first sample received in the SDG is the SDG number. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering

both alpha and numeric designations) in the first group of samples received under the SDG. Under no circumstances should any SDG number be replicated within a Case. If necessary, select an alternative sample number for the SDG number. The SDG number is also reported on all data reporting forms (see Exhibit B, Section 3 - Form Instructions). If the laboratory is requested to transfer samples to another facility, the Contractor shall date and enter the name of the facility to where the samples will be transferred on the CLP Traffic Report/Chain of Custody Record.

2.4.1 The Contractor shall submit Traffic Reports/Chain of Custody Records in SDG sets (i.e., Traffic Reports/Chain of Custody Records for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached. The SDG Cover Sheet shall contain the following items:

- Laboratory name;
- Contract number;
- Modified Analysis number;
- Sample analysis price (full sample price from the contract);
- Case number;
- List of methods/fractions analyzed (i.e., ICP-MS, ICP-AES, etc.); and
- List of EPA sample numbers of all samples in the SDG, identifying the **first** and **last** samples received, and their Laboratory Receipt Dates (LRDs).

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the sample with the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the sample with the highest sample number (considering both alpha and numeric designations).

2.4.2 EPA field sample numbers are continuous, without spaces or hyphens. The original Sample Traffic Report/Chain of Custody Record page marked "Lab Copy for Return to SMO", with laboratory receipt information and signed with original Contractor signature, shall be submitted for each sample in the SDG.

2.4.3 If samples are received at the laboratory with multi-sample Traffic Reports/Chain of Custody Records, all the samples on one multi-sample Traffic Report/Chain of Custody Record may not necessarily be in the same SDG. In this instance, the Contractor shall make the appropriate number of photocopies of the Traffic Report/Chain of Custody Record, and submit one copy with each SDG Cover Sheet.

2.5 Sample Data

The Sample Data shall include data for analysis of all samples in one SDG, including field and analytical samples, blanks, spikes, duplicates, and Laboratory Control Samples (LCSs). The Sample Data shall be complete before submission, and shall be consecutively paginated (starting with page number one and ending with the number of all pages in the package). The Sample Data shall include the following:

2.5.1 Cover Documentation

2.5.1.1 Cover Page for the inorganic analyses data shall include: laboratory name; laboratory code; contract number; Case number; SDG number; Modification Reference Number (Mod. Ref. No.) (if appropriate); Statement of Work (SOW) number; EPA sample numbers in alphanumeric order showing EPA sample numbers cross-referenced with laboratory Sample ID numbers; and the completion of the questions on the use of background and interelement corrections for the samples.

2.5.1.1.1 The Cover Page shall contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy Data Package and in the electronic data submitted has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature." This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

2.5.1.2 SDG Narrative. This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name, Case number, SDG number, SOW number, contract number, and detailed documentation of any Quality Control (QC), sample, shipment, and/or analytical problems encountered in processing the samples reported in the Complete SDG File (CSF). The Contractor shall list the target analytes for the SDG. The Contractor shall include any technical and administrative problems encountered, and the resolution or corrective actions taken. These problems may include, but are not limited to interference problems encountered during analysis, listing results from raw results less than the negative Contract Required Quantitation Limit (CRQL), and any problems with the analysis of wipes and filters. This also includes documenting the alternative technique used to determine cooler temperature if a temperature indicator bottle is not present in the cooler. The Contractor shall also provide, in the SDG Narrative, sufficient information, including equations with response factors or calibration curves with its fit expression (at least one equation or calibration curve per method), to allow the recalculation of sample results from raw instrument output. The Contractor shall also include a discussion of any SOW Modified Analyses. This includes attaching a copy of the USEPA approved modification form to the SDG Narrative. Additionally, the Contractor shall also identify and explain any differences which exist between the Form Is and supporting documentation provided in the data package and those previously provided as Preliminary Results.

2.5.1.3 Sample Log-In Sheet [Form DC-1]

2.5.1.4 Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

2.5.1.5 Sample Traffic Reports/Chain of Custody Records

2.5.2 Sample Data Forms and Raw Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. Data should be arranged in increasing alphanumeric EPA sample number order, followed by the QC analyses data, annual verification of method and instrument

parameters forms, raw data, and copies of the digestion and distillation logs (for Level 2b deliverables only).

2.5.2.1 Inorganic Analysis Data Sheet [Form IA-IN and Form IB-IN]. Tabulated analytical results of the requested analytes shall be included. The validation and release of these results is authorized by a specific signed statement on the Cover Page. In the event that the laboratory cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

2.5.2.1.1 Appropriate concentration units shall be specified and entered on Forms IA-IN and IB-IN. The quantitative values shall be reported in units of ug/L for aqueous/water samples, mg/kg for soil/sediment samples, and ug for wipe and air filter samples. (No other units are acceptable.) Results for soil/sediment samples shall be reported on a dry weight basis. Analytical results shall be reported to two significant figures if the result value is less than 10, and to three significant figures if the value is greater than or equal to 10. Results for percent solids shall be reported to this same format.

2.5.2.2 Quality Control (QC) Data

2.5.2.2.1 The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are only required for Level 2b deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

2.5.2.2.1.1 Initial and Continuing Calibration Verification [Form IIA-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.2 Blanks [Form III-IN]. Required for Level 2a and Level 2b deliverables. For Level 2a deliverables, only Preparation Blank data is required.

2.5.2.2.1.3 ICP-AES Interference Check Sample [Form IVA-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.4 ICP-MS Interference Check Sample [Form IVB-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.5 Matrix Spike Sample Recovery [Form VA-IN]

2.5.2.2.1.6 Post-Digestion Spike Sample Recovery [Form VB-IN]

2.5.2.2.1.7 Duplicates [Form VI-IN]

2.5.2.2.1.8 Laboratory Control Sample [Form VII-IN]

2.5.2.2.1.9 ICP-AES and ICP-MS Serial Dilutions [Form VIII-IN]

2.5.2.2.1.10 Method Detection Limit (MDL) (Annually) [Form IX-IN]

2.5.2.2.1.11 ICP-AES Interelement Correction Factors (Annually) [Form XA-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.12 ICP-AES Interelement Correction Factors (Annually) [Form XB-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.13 Internal Standard Association [Form XI-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.14 Preparation Log [Form XII-IN]

2.5.2.2.1.15 Analysis Run Log [Form XIII-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.16 ICP-MS Tune [Form XIV-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.17 ICP-MS Internal Standards Relative Intensity Summary [Form XV-IN]. Required for Level 2b deliverables only.

2.5.2.2.1.18 Initial Calibration [Form XVI-IN]. Required for Level 2b deliverables only.

2.5.2.3 Raw Data – Required for Level 2b deliverables only. Not required for Level 2a deliverables.

For each reported value, the Contractor shall include in the Sample Data all raw data used to obtain that value. This applies to all required Quality Assurance/Quality Control (QA/QC) measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the annual verification of method and instrument parameters submitted as a part of each CSF. When the analysis of the ICP-AES or ICP-MS target analytes listed in Exhibit C of this SOW (or any subset or additional analytes) is requested, the raw data for all samples shall include not only the results for the requested analyte(s), but also those for all the interferents (Exhibit D/ICP-AES, Table 1, or Exhibit D/ICP-MS, Table 1, as appropriate). The raw data shall also contain the results of any other analyte(s) which have been determined to interfere with the requested analytes(s).

2.5.2.3.1 Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. For example, if the instrument is applying an interelement correction for a reduced analyte list, the data used to calculate the correction must be present in the raw data. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.

2.5.2.3.2 The order of raw data in the Sample Data for inorganic analyses shall be: ICP-AES, ICP-MS, mercury, and cyanide. All raw data shall include concentration units for ICP, and absorbances or concentration units for mercury and cyanide.

2.5.2.3.3 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.5.2.3.4 Raw data shall be labeled with EPA sample numbers and appropriate codes, shown in Exhibit B, Table 2 - Codes for Labeling Data to unequivocally identify:

- Calibration standards, including source and preparation date. Standard preparation logbooks can be submitted if they contain this information;
- Initial and Continuing Calibration Blanks (ICBs/CCBs) and Preparation Blanks;
- Initial and Continuing Calibration Verification (ICV/CCV) standards, Interference Check Samples (ICSs), serial dilution samples, LCS, and post-digestion spike;
- Diluted and undiluted samples (by EPA sample number) and all weights, dilutions, and volumes used to obtain the reported values (if the volumes, weights, and dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters is sufficient);
- Duplicates;
- Spikes (indicating standard solutions used, final spike concentrations, and volumes involved). If spike information (e.g., source, concentration, and volume) is consistent for a given SDG, a general statement outlining these parameters is sufficient;
- Instrument used, any instrument adjustments, data corrections, or other apparent anomalies on the measurement record (including all data voided or data not used to obtain reported values), and a brief written explanation; and
- Time and date of each analysis. Instrument run logs can also be submitted if they contain time and date of analysis.

Table 2. Codes for Labeling Data[1,2]	
Sample	XXXXXX
Sample Not Part of the SDG	ZZZZZZ
Duplicate	XXXXXXD
Matrix Spike	XXXXXXS
Serial Dilution	XXXXXXL
Post-Digestion/Distillation Spike	XXXXXXA
Instrument Calibration Standards:	
ICP	S or S0 for blank standard
Atomic Absorption and Cyanide	S0, S10,...etc.
Initial Calibration Verification	ICV
Initial Calibration Blank	ICB
Continuing Calibration Verification	CCV
Continuing Calibration Blank	CCB
Interference Check Samples:	
Solution A	ICSA
Solution AB	ICSAB
Laboratory Control Samples:	LCS
Preparation Blank (Aqueous/Water)	PBW
Preparation Blank (Soil/Sediment)	PBS
Preparation Blank (Wipe/Filter)	PBF
ICP-MS Tune Check	TUNE

[1] The numeric suffix that follows the "S" suffix for the standards indicates the true value of the concentration of the standard in ug/L.

[2] ICP-AES and ICP-MS calibration standards usually consist of several analytes at different concentrations. Therefore, no numeric suffix can follow the ICP calibration standards unless all the analytes in the standard are prepared at the same concentrations. For instance, the blank for ICP shall be formatted "S0".

2.5.2.4 Digestion and Distillation Logs (only required for the Level 2b deliverable). The following logs shall be submitted as appropriate for each preparation procedure: digestion logs for ICP-AES, ICP-MS, and mercury preparations; and distillation logs for cyanide. These logs shall include: (1) date; (2) sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; (3) sufficient information to unequivocally identify which QC samples (i.e., LCS, Preparation Blank) correspond to each batch digested; (4) comments describing any significant sample changes or reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; (5) indication of pH less than or equal to 2 or greater than or equal to 12, as applicable; (6) any dilutions used in preparing the PE samples; and (7) identification of the sample preparer(s) [signature(s)].

2.5.2.5 Summary of Level 2a/2b Deliverables

The following table summarizes the contents of the Levels 2a and 2b deliverables.

Form	Level 2a	Level 2b
Cover Page	X	X
SDG Narrative	X	X
Form IA	X	X
Form IIA		X
Form III	X	X
Form IVA		X
Form IVB		X
Form VA	X	X
Form VB	X	X
Form VI	X	X
Form VII	X	X
Form VIII	X	X
Form IX	X	X
Form XA		X
Form XB		X
Form XI		X
Form XII	X	X
Form XIII		X
Form XIV		X
Form XV		X
Form XVI		X
Raw Data		X
Preparation Logs		X

2.6 Complete SDG File (CSF)

As specified in the Delivery Schedule, one CSF (including the original Sample Data) shall be delivered to the USEPA Region concurrently with the delivery of a copy to SMO. In addition, a copy in PDF format shall be delivered to SMO. Delivery to USEPA's designated recipient (e.g., QATS) is only required upon written request.

NOTE: The CSF for the Level 2a deliverable only includes summary forms as defined in Section 2.5.2.2.1 and the data listed in Section 2.6.2.

2.6.1 The CSF shall contain all original documents where possible. No photocopies of original documents shall be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to USEPA with another Case/SDG in accordance with the requirements described in Exhibit F. The CSF shall contain all original documents and be numbered according to the specifications in Exhibit B, Sections 3 and 4, and Form DC-2.

2.6.2 The CSF shall consist of the following original documents in addition to the documents in the Sample Data.

NOTE: All Case-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other Case-specific documents generated after the CSF is sent to USEPA, as well as copies that are altered in any fashion, are also deliverables to USEPA. Send the original to the USEPA Region and a copy to SMO. Send to USEPA's designated recipient (e.g., QATS) only upon written request.

2.6.2.1 Original Sample Data

2.6.2.2 A completed and signed Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

2.6.2.3 All original shipping documents, including, but not limited to, the following documents:

- USEPA Sample Traffic Reports/Chain of Custody Records;
- Airbills (if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information);
- Sample Tags (if present) sealed in plastic bags; and
- All original receiving documents, including, but not limited to, the following documents:
  - Form DC-1;
  - Other receiving forms or copies of receiving logbooks; and
  - SDG Cover Sheet.

2.6.2.4 For Level 2b deliverables, all original laboratory records of sample transfer, preparation, and analysis, including, but not limited to, the following documents:

- Original preparation and analysis forms, or copies of preparation and analysis logbook pages;
- Internal sample and sample digestate and distillate transfer Chain of Custody Records; and
- Performance Evaluation (PE) Instruction forms.

2.6.2.5 All other original SDG-specific documents in the possession of the laboratory, including, but not limited to, the following documents:

- Communication logs;
- Copies of personal logbook pages;
- All handwritten SDG-specific notes; and
- Any other SDG-specific documents not covered by the above.

2.6.3 If the Contractor does submit SDG-specific documents to USEPA after the submission of the CSF, the documents shall be numbered as an addendum to the CSF and a revised Form DC-2 shall be submitted; or the documents shall be numbered as a new CSF and a new Form DC-2 shall be submitted to the USEPA Region only.

2.6.4 The Contractor shall retain a legible electronic (PDF) or hardcopy of the CSF for 365 days after submission of the reconciled data package to the Government. After this time, the Contractor may dispose of the package.

2.7 Data in Electronic Format

The Contractor shall provide an electronic data deliverable for Levels 2a or 2b with analytical data for all samples in the SDG, as specified in Exhibit H, and delivered as specified in the Contract Schedule (Performance/Delivery Schedule).

2.8 Results of the Intercomparison and Performance Evaluation (PE) Sample Analyses

Tabulation of analytical results for intercomparison/PE sample analyses includes all requirements specified in Exhibit B, Sections 2.5 and 2.7.

2.9 Preliminary Results

The Form Is data results (including all appropriate qualifiers and flags) shall be submitted for all samples in one SDG of a Case. Sample analysis shall follow all requirements stipulated in Exhibit D. The Contractor shall clearly identify the Preliminary Results by labeling each Form I as "Preliminary Results" under the form title (i.e., under Inorganic Analysis Data Sheet). The Contractor shall also include a disclaimer in the "Comments" field on all Form Is stating that the "Data results contained on this Form I are for screening purposes only, and may not have been validated for CLP criteria." Sample Traffic Reports/Chain of Custody Records and SDG Cover Sheets shall be submitted with the Preliminary Results.

2.9.1 The Contractor shall submit the Cover Page following the specifications in Exhibit B, Sections 2.5.1 and 3.4.1. The Cover Page shall be clearly labeled to indicate that the data being reported are Preliminary Results. The Cover Page shall contain the following statement, verbatim: "**I certify that these Preliminary Results are in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.**" This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

2.10 Annual Calculation of Method Detection Limits (MDLs) and Interelement Correction Factors

The Contractor shall perform and report annual determination of the MDLs by the method specified in Exhibit D for each instrument used under this contract. The Contractor shall also perform and report annual ICP-AES interelement correction factors (including method of determination), wavelengths used, and integration times. Forms reporting results for the annual verification of method parameters for the current period shall be submitted using Form IX. Submission of the calculation of method and instrument parameters shall include the data used to determine the values reported as well as, for the Interelement Corrections (IECs), the standard preparation logs, sample preparation logs, and analysis logs with analytical sequences.

2.11 Instrument Electronic Data

The Contractor shall adhere to the requirements in Exhibit E.

2.12 Delivery of Hardcopy Data in PDF Format

In addition to all required deliverables identified in the laboratory's contract and the ISM01.2 SOW, the laboratory shall provide a complete copy of the hardcopy deliverable in PDF on a Compact Disc (CD).

2.12.1 The PDF file shall be organized in accordance with the directions provided in Exhibit B, "Reporting Requirements and Order of Data Deliverables", of the ISM01.2 SOW. The PDF shall be bookmarked as described below for ease of data retrieval and navigation.

2.12.2 Inorganic data shall be bookmarked using a hierarchical bookmark structure (i.e., an overview or "parent" bookmark, and a subordinate or "child" bookmark nested underneath the "parent" bookmark). The required hierarchical structure is shown in Table 3.

TABLE 3. Hierarchical Bookmark Structure

Group Bookmark	**Parent Bookmark**	**Child Bookmark**
Cover Page for inorganic analyses, SDG Narrative, Form DC-1,Form DC-2, and Sample TR/COCs		
Sample Data	QC Summary	Inorganic Analysis Data Sheets
		Initial and Continuing Calibration Verification
		Blanks
		ICP-AES Interference Check Sample
		ICP-MS Interference Check Sample
		Matrix Spike Sample Recovery
		Post-Digestion Spike Sample Recovery
		Duplicates
		Laboratory Control Sample
		ICP-AES and ICP-MS Serial Dilutions
		Method Detection Limits (Annually)
		ICP-AES Interelement Correction Factors (Annually)
		Internal Standard Association
		Preparation Log
		Analysis Run Log
		ICP-MS Tune
		ICP-MS Internal Standards Relative Intensity Summary
		Initial Calibration
	Raw Data	ICP-AES
		ICP-MS
		Mercury
		Cyanide
		Digestion and Distillation Logs
Receiving Documents, Transfer Records, Miscellaneous	Additional Documents	Receiving Logbooks
		Preparation and Analysis Logbooks
		Internal Sample, Digestate, and Distillate Transfer Chain-of-Custody Records
		PE Instruction Forms
		Communication Logs

2.13 Corrective Action Procedures

If the Contractor fails to adhere to the requirements detailed in this SOW, the Contractor will be in noncompliance with the contract and may be subjected to sanctions as described in the contract.

3.0 FORM INSTRUCTIONS

3.1 Introduction

This section contains specific instructions for the completion of all required Inorganic Data Reporting Forms.

3.2 General Information

Values shall be reported on the hardcopy forms according to the respective form instructions in this section.

3.2.1 The data reporting forms discussed in Exhibit B, Section 3.4, and presented in Exhibit B, Section 4.0, have been designed in conjunction with the electronic data format specified in Exhibit H. Information entered on these forms shall **not** exceed the size of the field given on the form, including such laboratory-generated items as "Lab Name" and "Lab Sample ID".

3.2.2 All characters which appear on the data reporting forms presented in the contract shall be reproduced by the Contractor when submitting data, and the format of the forms submitted shall provide exactly the same information as that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM). The names of various fields and analytes (i.e., "Lab Code", "Aluminum") shall appear as they do on the forms in the contract, including the options specified in the form.

3.3 Header Information

Six pieces of information are common to the header sections of each data reporting form. These are: Laboratory Name, Contract, Laboratory Code, Case number, Modification Reference Number (Mod. Ref. No.), and Sample Delivery Group (SDG) number. Except as noted for Mod. Ref. No., this information shall be entered on every form and shall match on all forms.

3.3.1 Laboratory Name. The "Lab Name" shall be the name chosen by the Contractor to identify the laboratory.

3.3.2 Contract. The "Contract" is the number of the USEPA contract under which the analyses were performed.

3.3.3 Laboratory Code. The "Lab Code" is an alphabetic abbreviation, <u>assigned by USEPA</u>, to identify the laboratory and aid in data processing. This laboratory code will be assigned by USEPA at the time a contract is awarded. The laboratory code <u>shall not</u> be modified by the Contractor, except at the direction of USEPA. If a change of name or ownership occurs at the laboratory, the laboratory code will remain the same until the Contractor is directed by USEPA to use another laboratory code.

3.3.4 Case Number. The "Case No." is the Sample Management Office (SMO)-assigned Case number associated with the sample, and reported on the Traffic Report/Chain of Custody Record.

3.3.5 Mod. Ref. Number. The "Mod. Ref. No." is the USEPA assigned number for analyses performed for an analytical method under the modified analysis clause in Exhibit A. If samples are to be analyzed under the modified analysis clause, the Contractor shall list both the Case No. and the modification reference number on all forms. If the analyses have no modified requirements, leave the "Mod. Ref. No." field blank.

3.3.6 SDG Number. The "SDG No." is the Sample Delivery Group (SDG) number. The SDG number is the EPA sample number of the first sample received in the SDG, except when this would cause duplication. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. If fractions of the same field samples are scheduled under different turnaround times, thus creating separate SDGs containing the same sample numbers, a different sample number shall be utilized in the assignment of the SDG number for each SDG. If a situation arises where there is an insufficient number of samples for assignment of SDG numbers, the contractor shall contact SMO for the assignment of an SDG number.

3.3.7 Sample Number. The "EPA Sample No." appears either in the header information of the form or as the left column of a table summarizing data from a number of samples. When an EPA sample number is entered in the box in the upper right-hand corner of a form, it shall be centered.

3.3.7.1 **All** samples, matrix spikes, post-digestion/distillation spikes, duplicates, and serial dilutions shall be identified with an EPA sample number. For samples, an EPA sample number is the unique identifying number given in the Traffic Report/Chain of Custody Record that accompanied that sample. In order to facilitate data assessment, the sample suffixes listed in Exhibit B, Table 2 - Codes for Labeling Data, must be used.

3.3.8 Matrix. For "Matrix", enter "Soil" for soil/sediment samples, "Water" for aqueous/water samples, "Wipe" for surface wipes, and "Filter" for air filter samples.

3.3.9 Rounding Rule. For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is greater than or equal to 5, the absolute value of the result is to be rounded up; otherwise the absolute value of the result is rounded down. For example, -0.4365 rounds to -0.437 and -2.3564 rounds to -2.356. Also see "Rounding Rules" in Exhibit G.

3.3.9.1 Before evaluating a number for being in control or out of control of a certain limit [other than the Contract Required Quantitation Limit (CRQL)], the number evaluated shall be rounded using the above rounding rules to the significance reported for that limit. For example, the control limit for an Initial Calibration Verification is plus or minus 10% of the true value. Then a calculated percent recovery of 110.46 shall be reported on Form IIA-IN as 110, which is within the control limits of 90-110. On the other hand, a calculated percent recovery of 110.50 shall be reported on Form IIA-IN as 111, which is not within the 90-110 percent control limits.

NOTE: All results shall be transcribed to Inorganic Forms IIA-IN through XV-IN from the instrument raw data to two significant figures if the value is less than 10, or three significant figures if the value is greater than or equal to 10 as described in Exhibits B and H. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. The instrument raw data files contain the raw data values. The hardcopy raw data may be a rounded or truncated representation of the instrument raw data.

3.4 Inorganic Forms

3.4.1 Cover Page - [COVER PAGE]

3.4.1.1 Purpose. This form is used to list all samples analyzed within an SDG and provide certain analytical information and general comments. It is also the document that is signed by the Laboratory Manager to authorize and release all data and deliverables associated with the SDG.

3.4.1.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.1.2.1 For samples analyzed using this Statement of Work (SOW), enter "ISM01.2" for the SOW Number.

3.4.1.2.2 Enter an EPA sample number including spikes and duplicates of every sample analyzed within the SDG. Spikes shall contain an "S" suffix and duplicates a "D" suffix. These sample numbers shall be listed on the form in ascending alphanumeric order. Thus, if MA1111 is the lowest (considering both alpha and numeric characters) EPA sample number within the SDG, it would be entered in the first EPA sample number field. Samples would be listed below it, in ascending sequence - MA1111, MA1111D, MAB124, MAB125, MAC111, etc.

3.4.1.2.3 A maximum of 20 field sample numbers can be entered on this form. Submit additional Cover Pages, as appropriate, if the total number of samples, duplicates, and spikes in the SDG is greater than 22.

3.4.1.2.4 A Laboratory Sample ID may be entered for each EPA sample number. If a Laboratory Sample ID is entered, it shall be entered identically (for each EPA sample number) on all associated data.

3.4.1.2.5 Enter "Yes" or "No" in answer to each of the two questions concerning Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) corrections. Each question shall be explicitly answered with a "Yes" or a "No". The third question shall be answered with a "Yes" or "No" if the answer to the second question is "Yes". It shall be left blank if the answer to the second question is "No".

3.4.1.2.6 Under "Comments", enter any statements relevant to the analyses or modifications performed under the SDG as a whole.

3.4.1.2.7 Each Cover Page shall be signed and dated, in original, by the Laboratory Manager or the Manager's designee to authorize the release and verify the contents of all data and deliverables associated with an SDG.

3.4.2 Inorganic Analysis Data Sheet [Forms IA-IN and IB-IN]

3.4.2.1 Purpose. These forms are used to tabulate and report sample analysis results for inorganic target analytes for an analytical method (see Exhibit C).

3.4.2.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.2.2.1 "Date Received" is the date (formatted MM/DD/YYYY) of sample receipt at the laboratory, as recorded on the Traffic Report/Chain of Custody Record [i.e., the Validated Time of Sample Receipt (VTSR)].

3.4.2.2.2 "% Solids" is the percent of solids on a weight-by-weight basis in the sample which is determined by drying the sample as specified in Exhibit D - Introduction to Analytical Methods, Section 1.6. Report to two significant figures if the value is less than 10, and to three significant figures if the value is greater than or equal to 10. If the percent solids is not required because the sample is fully aqueous, or is less than 1% solid, then leave blank.

3.4.2.2.3 Enter the appropriate concentration units (ug/L for aqueous/water, mg/kg for soil/sediment, and ug for wipe and air filter). Entering "mg/kg" means "mg/kg dry weight" on this form.

3.4.2.2.4 Under the column labeled "Concentration", enter for each analyte, the value of the result [if the concentration or mass is greater than or equal to the Method Detection Limit (MDL) corrected for any dilutions; or, enter the Contract Required Quantitation Limit (CRQL) for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL]. The concentration or mass result shall be reported to two significant figures if the result is less than 10, or three significant figures if the value is greater than or equal to 10.

3.4.2.2.5 Under the columns labeled "C", "Q", and "M", enter result qualifiers as identified below. If additional qualifiers are used, their explicit definitions shall be included on the Cover Page in the "Comments" section.

Forms IA-IN and IB-IN include fields for three types of result qualifiers. These qualifiers shall be completed as follows:

3.4.2.2.5.1 C (Concentration) Qualifier. Enter "J" if the reported value was obtained from a reading that was less than the CRQL but greater than or equal to the MDL. If the reading was less than the MDL, a "U" shall be entered.

The MDL obtained for a given preparation method, analysis method, and instrument shall be used for the qualification of the results for samples associated with that preparation method, analysis method, and instrument. Serial dilution

and post-digestion spike results shall be qualified using the MDL and CRQL values utilized for the corresponding field sample.

All three values (i.e., the instrument reading, CRQL, and MDL) shall be converted to the same units prior to determining the appropriate C (Concentration) Qualifier.

NOTE: For ICP-AES and ICP-MS, the Not-Prepared MDL (in ug/L) determined for an instrument and the aqueous/water CRQL shall be used to qualify the results of instrument QC standards that are not taken through a preparation procedure [e.g., Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)].

3.4.2.2.5.2 Q Qualifier. Specified entries and their meanings are as follows:

E: The reported value is estimated due to the presence of interference. An explanatory note shall be included under "Comments" on the Cover Page (if the problem applies to all samples), or on the specific Form IA-IN or Form IB-IN (if it is an isolated problem).

N: Spiked sample recovery not within control limits.

*: Duplicate analysis not within control limits.

D: The reported value is from a dilution.

3.4.2.2.5.3 M (Analysis Method) Qualifier. Specified entries and their meanings are as follows:

P: ICP/AES

MS: ICP/MS

CV: Cold Vapor Atomic Absorption (AA) for mercury

AS: Spectrophotometric for cyanide

3.4.2.2.6 A brief physical description of aqueous/water and soil/sediment samples, both before and after digestion, shall be reported in the fields for color (before and after), clarity (before and after), texture, and artifacts. For aqueous/water samples, report color and clarity. For soil/sediment samples, report color, texture, and artifacts. The following descriptive terms are recommended:

- Color - red, blue, yellow, green, orange, violet, white, colorless, brown, grey, and black;
- Clarity - clear, cloudy, and opaque; and
- Texture - fine (powdery), medium (sand), and coarse (large crystals or rocks).

If artifacts are present, enter "Yes" in the artifacts field and describe the artifacts in the "Comments" field. If artifacts are not present, enter "No". Note any significant changes that occur during sample preparation (e.g., emulsion formation) in the "Comments" field. Enter any sample-specific comments concerning the analyte results in the "Comments" field. Also document raw instrument results that are less than minus the CRQL (-CRQL) in the "Comments" field and in the SDG Narrative.

3.4.2.2.7 If more than two additional analytes were requested, submit Form IB-IN as appropriate.

3.4.3 Initial (ICV) and Continuing Calibration Verification (CCV) [Form IIA-IN]. This form is not required for Level 2a deliverables.

3.4.3.1 Purpose. This form is used to report analyte recoveries from calibration verification solutions.

3.4.3.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.3.2.1 Enter the ICV Source and the CCV Source. Enter sufficient information to identify the manufacturer and the solution (lot) used.

Use additional Form(s) IIA-IN if more calibration verification sources were used.

3.4.3.2.2 Under "Initial Calibration Verification True", enter the value in ug/L, of the concentration of each analyte in the ICV Solution.

3.4.3.2.3 Under "Initial Calibration Verification Found", enter the most recent value (in ug/L), of the concentration of each analyte measured in the ICV Solution.

3.4.3.2.4 Under "Initial Calibration Verification %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 1 ICV Percent Recovery

$$\%R = \frac{\text{Found(ICV)}}{\text{True(ICV)}} \times 100$$

WHERE, "True(ICV)" is the true concentration of the analyte in the ICV Solution and "Found(ICV)" is the found concentration of the analyte in the ICV Solution.

3.4.3.2.5 Under "Continuing Calibration Verification True", enter the value (in ug/L) of the concentration of each analyte in the CCV Solution.

3.4.3.2.6 Under "Continuing Calibration Verification Found", enter the value (in ug/L) of the concentration of each analyte measured in the CCV Solution.

NOTE: The form contains two "Continuing Calibration Verification Found" columns. The column to the left shall contain values for the first CCV, and the column to the right shall contain values for the second CCV.

3.4.3.2.7 If more than one Form IIA-IN is required to report multiple CCVs, then the column to the left on the second form shall contain values for the third CCV, the column to the right shall contain values for the fourth CCV, and so on.

3.4.3.2.8 Under "Continuing Calibration Verification %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 2 CCV Percent Recovery

$$\%R = \frac{\text{Found(CCV)}}{\text{True(CCV)}} \times 100$$

WHERE, "True (CCV)" is the true concentration of the analyte in the CCV Solution, and "Found (CCV)" is the found concentration of the analyte in the CCV Solution.

NOTE: The form contains two "Continuing Calibration Verification %R" columns. Entries to these columns shall follow the sequence detailed above for entries to the "Continuing Calibration Verification Found" columns.

3.4.3.2.9 Under "M", enter the method used, as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.3.2.10 If more than one wavelength/mass is used to analyze an analyte, submit additional Form(s) IIA-IN as appropriate.

3.4.3.2.11 The order of reporting ICVs and CCVs for each analyte shall follow the chronological order in which the standards were run. Start with the first Form IIA-IN and move from the left to the right, continuing to the following Form IIA-INs as appropriate. For instance, the first ICV for all analytes shall be reported on the first Form IIA-IN. In a run where three CCVs were analyzed, the first CCV shall be reported in the left CCV column on the first Form IIA-IN and the second CCV shall be reported in the right column of the same form. The third CCV shall be reported in the left CCV column of the second Form IIA-IN. On the second Form IIA-IN, the ICV column and the right CCV column shall be left empty in this example. In the previous example, if a second run for an analyte was needed, the ICV of that run shall be reported on a third Form IIA-IN and the CCVs follow in the same fashion as explained before. In the case where two wavelengths are used for an analyte, all ICV and CCV results of one wavelength from all runs shall be reported before proceeding to report the results of the second wavelength used.

3.4.4 Blanks [Form III-IN]. Required for Level 2a and Level 2b deliverables. For Level 2a deliverables, only Preparation Blank data is required.

3.4.4.1 Purpose. This form is used to report analyte concentrations found in the ICB, CCB, and the Preparation Blank. For Level 2a deliverables, only the Preparation Blank analyte concentrations are required.

3.4.4.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.4.2.1 Enter "Soil", "Water", "Wipe", or "Filter" as appropriate as the matrix of the Preparation Blank. No abbreviations or other matrix descriptors may be used.

3.4.4.2.2 According to the matrix specified for the Preparation Blank, enter the Preparation Blank concentration units as "ug/L" for aqueous, "mg/kg" for soil/sediment, or "ug" for wipes and filters.

3.4.4.2.3 Under "Initial Calibration Blank", enter the concentration (in ug/L) of each analyte in the most recent ICB, as described in Exhibit B, Section 3.4.4.2.8, below.

3.4.4.2.4 For each calibration blank associated with a given method and instrument, enter "J" under the "C" qualifier field on Form III-IN if the absolute value of the analyte concentration is less than the CRQL for aqueous/water but greater than or equal to the MDL (in ug/L) determined for the default aqueous/water preparation method on that particular instrument.

For prepared calibration blanks (e.g., mercury and cyanide), the CRQL for aqueous/water and the MDL (in ug/L or converted to ug/L) for the preparation method, analysis, and instrument shall be used.

Enter "U" if the absolute value of the analyte in the blank is less than the MDL (in ug/L or converted to ug/L) obtained from the default aqueous/water preparation method on that instrument (unprepared blanks) or determined for the preparation method (prepared blanks).

3.4.4.2.5 Under "Continuing Calibration Blank 1", enter the concentration (in ug/L) of each analyte detected in the first required CCB analyzed after the ICB, as described in Exhibit B, Section 3.4.4.2.8, below. Enter any appropriate qualifier, as explained for the "Initial Calibration Blank", to the "C" qualifier column immediately following the "Continuing Calibration Blank 1" column.

3.4.4.2.6 If up to three CCBs were analyzed, complete the columns labeled "2" and "3" in accordance with the instructions for the "Continuing Calibration Blank 1" column. If more than three CCBs were analyzed, then complete additional Form(s) III-IN as appropriate.

3.4.4.2.7 Under "Preparation Blank", enter the concentration in ug/L for an aqueous/water blank, mg/kg for a soil/sediment blank, or mass in ug for a wipe or filter blank, of each analyte in the Preparation Blank, as described in Exhibit B, Section 3.4.4.2.8, below. Evaluate the absolute value of the analyte concentration to determine the appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, and enter the qualifier in the "C" column immediately following the "Preparation Blank" column.

3.4.4.2.8 For all blanks, enter the concentration or mass (positive or negative) for each analyte, if the absolute value of the concentration or mass is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration or mass is less than the appropriate MDL.

3.4.4.2.9 Under "M", enter the method used, as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.4.2.10 If more than one wavelength/mass is used to analyze an analyte, submit additional Form(s) III-IN as appropriate.

3.4.4.2.11 The order of reporting ICBs and CCBs for each analyte shall follow the chronological order in which the blanks were run, starting with the first Form III-IN and moving from left to right and continuing to additional Forms III-IN. When multiple wavelengths are used for the analysis of one analyte, all the results of one wavelength shall be reported before proceeding to the next wavelength.

3.4.5 ICP-AES and ICP-MS Interference Check Sample (ICS) [Forms IVA-IN and IVB-IN]. This form is not required for Level 2a deliverables.

3.4.5.1 Purpose. These forms are used to report ICS results for each ICP-AES or ICP-MS instrument used in SDG analyses.

3.4.5.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions. The instructions for Forms IVA-IN and IVB-IN are identical except where specified.

3.4.5.2.1 For "ICP Instrument ID", enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two ICP instruments within a laboratory may have the same ICP Instrument ID.

3.4.5.2.2 Enter "ICSA Source" and "ICSB Source" as explained in Exhibit B, Section 3.4.3.2.1. For USEPA solutions, include in the source name a number identifying it (e.g., ICSA-1206).

3.4.5.2.3 Under "True Sol. A", enter the true concentration (in ug/L) of each analyte present in Solution A. Enter "0" for each analyte with no specified true value in Solution A.

3.4.5.2.4 Under "True Sol. AB", enter the true concentration (in ug/L) of each analyte present in Solution AB. Enter "0" for each analyte with no specified true value in Solution AB.

3.4.5.2.5 Under "Found Sol. A", enter the concentration (positive, negative, or zero, in ug/L). Enter the concentration of each analyte and interferent for ICP-AES, and of each analyte and interferent for ICP-MS in the initial analysis of Solution A as required in Exhibit D.

3.4.5.2.6 Under "Found Sol. A %R", enter the value (to the nearest whole number) of the percent recovery computed for True Solution A greater than zero according to the following equation:

EQ. 3 Initial Found Sol. A Percent Recovery

$$\%R = \frac{\text{Initial Found Solution A}}{\text{True Found Solution A}} \times 100$$

Leave the field blank if "True Solution A" equals zero.

3.4.5.2.7 Under "Found Sol. AB", enter the concentration (positive, negative, or zero, in ug/L. Enter the concentrations of each analyte and interferent for ICP-AES, and of each analyte and interferent for ICP-MS in the initial analysis of Solution AB as required in Exhibit D.

3.4.5.2.8 Under "Found Sol. AB %R", enter the value (to the nearest whole number) of the percent recovery computed for True Solution AB greater than zero according to the following equation:

EQ. 4 Initial Found Sol. AB Percent Recovery

$$\%R = \frac{\text{Initial Found Solution AB}}{\text{True Solution AB}} \times 100$$

Leave the field blank if "True Solution AB" equals zero.

3.4.5.2.9 If more ICS analyses were required, submit additional Form(s) IVA-IN and/or IVB-IN as appropriate.

3.4.5.2.10 The order of reporting ICSs for each analyte shall follow the chronological order in which the standards were run, starting with the first Form IVA-IN and/or IVB-IN and continuing to the following Forms IV-IN as appropriate. When multiple wavelengths/masses are used for one analyte, all the results of one wavelength/mass shall be reported before proceeding to the next wavelength/mass.

3.4.6 Matrix Spike Sample Recovery [Form VA-IN]

3.4.6.1 Purpose. This form is used to report results for the pre-digest spike.

3.4.6.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.6.2.1 Indicate the appropriate matrix and concentration units (ug/L for aqueous/water and mg/kg dry weight for soil/sediment) as explained in Exhibit B, Sections 2.5.2.1.1 and 3.3.8.

3.4.6.2.2 For "% Solids for Sample", enter the percent solids (see Exhibit B, Section 3.4.2.2.2) for the original sample of EPA sample number reported on the form. Note that this number must equal the one reported on Form IA-IN for that sample.

3.4.6.2.3 In the "EPA Sample No." box, enter an EPA sample number of the sample from which the spike results on this form were obtained.

3.4.6.2.4 Under "Control Limit %R", enter "75-125" if the sample result is less than or equal to four times the spike added value. If the sample result is greater than four times the Spike Added (SA) value, leave this field empty.

3.4.6.2.5 Under "Spiked Sample Result (SSR)", enter the measured value, in appropriate units, for each relevant analyte in the matrix spike sample. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Spiked Sample Result (SSR)" column.

3.4.6.2.6 Under "Sample Result (SR)", enter the measured value for each required analyte in the sample (reported in "EPA Sample No."

box) on which the matrix spike was performed. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Sample Result (SR)" column.

3.4.6.2.7 Under "Spike Added (SA)", enter the value for the concentration of each analyte added to the sample. The same concentration units shall be used for "SSR", "SR", and "SA". If the "SA" concentration is specified in the contract, then the value added and reported shall be the specific concentration in appropriate units, corrected for spiked sample weight and percent solids (soil/sediment) or spiked sample volume (aqueous/water).

3.4.6.2.8 Under "%R", enter the value (to the nearest whole number) of the percent recovery for all spiked analytes computed according to the following equation:

EQ. 5 Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Percent recovery shall be reported, whether it is negative, positive, or zero.

A value of zero shall be used in calculations for "SSR" or "SR" if the analyte value is less than the MDL.

3.4.6.2.9 Under "Q", enter "N" if the Spike Recovery (%R) is out of the control limits (75-125) and the Sample Result (SR) is less than or equal to four times the SA.

3.4.6.2.10 Under "M", enter the method used (as explained in Exhibit B, Section 3.4.2.2.5.3) or enter "NR" if the analyte is not required in the spike.

3.4.6.2.11 If different samples were used for spike sample analysis of different analytes, additional Form(s) VA-IN shall be submitted for each sample as appropriate.

3.4.7 Post-Digestion Spike Sample Recovery [Form VB-IN]

3.4.7.1 Purpose. This form is used to report results for the post-digest spike recovery which is based upon the addition of a known quantity of analyte to an aliquot of the <u>digested</u> sample.

3.4.7.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.7.2.1 In the "EPA Sample No." box, enter an EPA sample number of the sample from which the spike results on this form were obtained.

3.4.7.2.2 The "Control Limit %R" and "Q" fields shall be left blank until limits are established by USEPA. At that time, the Contractor will be informed how to complete these fields.

3.4.7.2.3 Under "Spiked Sample Result (SSR)", enter the measured value in appropriate units for each analyte in the post-digest spike sample. Enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL for the analyte if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Spiked Sample Result (SSR)" column.

3.4.7.2.4 Under "Sample Result (SR)", enter the measured value in appropriate units for the concentration of each analyte in the sample (reported in "EPA Sample No." box) on which the spike was performed. Enter the value (if the concentration is greater than or equal to the MDL); or enter the CRQL for the analyte if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Sample Result (SR)" column.

3.4.7.2.5 Under "Spike Added (SA)", enter the value in appropriate units for each analyte added to the sample. If the "SA" concentration is specified in the contract, the value added and reported shall be that specific concentration in appropriate units.

3.4.7.2.6 Under "%R", enter the value (to the nearest whole number) of the percent recovery for all spiked analytes computed according to EQ. 5 in Exhibit B, Section 3.4.6.2.8. Percent recovery shall be reported, whether it is negative, positive, or zero. A value of zero shall be substituted for "SSR" or "SR" if the analyte value is less than the MDL.

3.4.7.2.7 Under "M", enter the method used (as explained in Exhibit B, Section 3.4.2.2.5.3) or enter "NR" if the spike was not required.

3.4.7.2.8 If different samples were used for spike sample analysis of different analytes, additional Form(s) VB-IN shall be submitted for each sample as appropriate.

3.4.8 Duplicates [Form VI-IN]

3.4.8.1 Purpose. The duplicates form is used to report results of duplicate analyses. Duplicate analyses are required for percent solids values and all analyte results.

3.4.8.2 Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.8.2.1 Indicate the appropriate matrix and concentration units (ug/L for aqueous/water and mg/kg dry weight for soil/sediment) as explained in Exhibit B, Sections 2.5.2.1.1 and 3.3.8.

3.4.8.2.2 For "% Solids for Sample", enter the percent solids (as explained in Exhibit B, Section 3.4.2.2.2) for the original sample of the EPA sample number reported on the form. Note that this number must equal the one reported on Form IA-IN for that sample.

3.4.8.2.3 In the "EPA Sample No." box, enter the EPA sample number of the sample from which the duplicate sample results on this form were obtained.

3.4.8.2.4 Under "Control Limit", enter the CRQL (in appropriate units, ug/L for aqueous/water or mg/kg percent solids basis corrected for the original sample weight and percent solids) for the analyte if either the sample or duplicate value was less than 5 times the CRQL. If the sample and duplicate values were greater than or equal to 5 times the CRQL, or if the sample and duplicate values were less than the CRQL, leave the field empty.

3.4.8.2.5 Under "Sample (S)", enter the original measured value for the concentration of each analyte in the sample (reported in "EPA Sample No." box) on which a duplicate analysis was performed. Concentration units are those specified on the form. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Sample (S)" column.

3.4.8.2.6 Under "Duplicate (D)", enter the measured value for each analyte in the duplicate sample. Concentration units are those specified on the form. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Duplicate (D)" column.

3.4.8.2.7 For soil/sediment samples, the concentration of the original sample shall be computed using the weight and percent solids of the original sample. The concentration of the duplicate sample shall be computed using the weight of the duplicate sample, but the percent solids of the original sample.

3.4.8.2.8 Under "RPD", enter the absolute value (to the nearest whole number) of the Relative Percent Difference (RPD) for all analytes detected above the MDL in either the sample or the duplicate, computed according to the following equation:

EQ. 6 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

A value of zero shall be substituted for "S" or "D" if the analyte concentration is less than the MDL in either one. If the analyte concentration is less than the MDL in both "S" and "D", leave the "RPD" field empty.

3.4.8.2.9 Under "Q", enter "*" if the duplicate analysis for the analyte is out of control. If both sample and duplicate values are greater than or equal to 5 times the CRQL, then the RPD must be less than or equal to 20% to be in control. If either the sample or duplicate value is less than 5 times the CRQL, then the absolute difference between the sample and duplicate values shall be less than the CRQL to be in control.

3.4.8.2.10 If both values are below the CRQL, then no control limit is applicable.

3.4.8.2.11 Under "M", enter method used as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.9 Laboratory Control Sample [Form VII-IN]

3.4.9.1 Purpose. This form is used to report results for the soil/sediment, aqueous/water, wipe, and filter Laboratory Control Samples (LCSs).

3.4.9.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.9.2.1 Under "True", enter the value (in ug/L for aqueous/water, mg/kg for soil/sediment, or ug for wipes and filters) of the concentration of each analyte in the LCS.

3.4.9.2.2 Under "Found", enter the measured mass or concentration (in ug/L, mg/kg, or ug as appropriate) of each analyte found in the LCS solution.

3.4.9.2.3 Under "%R", enter the value of the percent recovery (to the nearest whole number) computed according to the following equation:

EQ. 7 LCS Percent Recovery

$$\%R = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

3.4.9.2.4 If the analyte concentration is less than the MDL, a value of zero shall be substituted for the LCS found.

3.4.9.2.5 Submit additional Form(s) VII-IN as appropriate if more than one LCS was required.

3.4.10 ICP-AES and ICP-MS Serial Dilutions [Form VIII-IN]

3.4.10.1 Purpose. This form is used to report results for ICP-AES and ICP-MS serial dilutions.

3.4.10.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.10.2.1 In the "EPA Sample No." box, enter EPA sample number of the sample for which serial dilution analysis results on this form were obtained.

3.4.10.2.2 Under "Initial Sample Result (I)", enter the measured value in appropriate units for each ICP analyte. Enter the value (if the concentration is greater than or equal to the MDL); or enter the CRQL if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Initial Sample Result (I)" column.

3.4.10.2.3 Under "Serial Dilution Result (S)", enter the measured value in appropriate units for each ICP analyte in the diluted sample. Enter the value (if the concentration is greater than or equal to the MDL); or enter the CRQL if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to

the "C" qualifier column immediately following the "Serial Dilution Result (S)" column.

3.4.10.2.4 Under "% Difference", enter the absolute value (to the nearest whole number) of the percent difference in the concentration of the required analytes, between the original sample and the diluted sample (adjusted for dilution) according to the following formula:

EQ. 8 Serial Dilution Percent Difference

$$\% \text{ Difference} = \frac{|I - S|}{I} \times 100$$

A value of zero shall be substituted for "S" if the analyte concentration is less than the MDL. If the analyte concentration in (I) is less than the MDL concentration, leave the "% Difference" field empty.

3.4.10.2.5 Under "Q", enter "E" if the percent difference is greater than 10% and the original sample concentration (reported on Form IA-IN) is greater than 50 times the MDL reported on Form IX-IN.

3.4.10.2.6 Under "M", enter the method of analysis for each analyte as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.11 Method Detection Limit (Annually) [Form IX-IN]

3.4.11.1 Purpose. This form documents the MDL for each preparation method and instrument that the Contractor used to obtain data for the SDG. Only the methods, instruments, and wavelengths used to generate data for the SDG shall be included. The Contractor shall also report the MDLs obtained from direct analysis, for each instrument used to obtain data for the SDG. The MDLs obtained from direct analysis shall be used in the qualification of data associated with samples and instrument QC standards that are not taken through a preparation procedure. Although the MDLs are determined annually, a copy of the annual MDLs shall be included with each Complete Sample Delivery Group File (CSF) on Forms IX-IN.

3.4.11.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.11.2.1 Enter the Analysis Method qualifier as specified in Exhibit B, Section 3.4.2.2.5.3, in the "Instrument Type:" field.

3.4.11.2.2 Enter the Instrument ID in the "Instrument ID:" field. These instrument IDs are used to uniquely identify each instrument that the laboratory used to perform the analysis.

3.4.11.2.3 Enter the date (formatted MM/DD/YYYY) on which the MDL calculation was updated in the "Date" field.

3.4.11.2.4 For "Preparation Method:", enter the method of preparation for which the MDLs listed on Form IX-IN were established. Use the appropriate sample preparation names as specified below:

200.7: ICP-AES aqueous/water samples
200.8: ICP-MS aqueous/water samples
3050B: ICP-AES, ICP-MS soil/sediment samples; ICP-AES wipe samples
7300: ICP-AES filter samples
7470A: Mercury aqueous/water samples
7471B: Mercury soil/sediment samples
Midi-distillation: Cyanide aqueous/water and soil/sediment samples
Micro-distillation: Cyanide aqueous/water and soil/sediment samples
3015A: Alternative for ICP-AES, ICP-MS aqueous/water samples
3051A: Alternative for ICP-AES, ICP-MS soil/sediment samples

3.4.11.2.5 Enter the concentration units (ug/L for aqueous/water, mg/kg for soil/sediment, or ug for wipes and filters) for the results reported on Form IX-IN in the "Concentration Units" field.

3.4.11.2.6 Under "Wavelength/Mass", enter the wavelength in nanometers (nm) or the mass in atomic mass units (amu) for each analyte for which an MDL has been established and is listed in the appropriate column. If more than one wavelength or mass is used for an analyte, use additional Form(s) IX-IN as appropriate to report the MDLs.

3.4.11.2.7 Under "MDL", enter the MDL [in ug/L for aqueous/water and non-prepared (direct analysis), mg/kg for soil/sediment, and ug for wipes and filters, to two significant figures for values less than 10, and three significant figures for values greater than or equal to 10] as determined by the Contractor for each analyte analyzed by the instrument for which the ID is listed on this form. When calculating MDL values, always round <u>up</u> to the appropriate significant figure (e.g., 14.81 rounds to 14.9 and 146.6 rounds to 147). This deviation from the rounding rule is necessary to prevent the reporting of detected values for results that fall in the noise region of the calibration curve.

NOTE: Zeros used to set the decimal point in a number less than one are not significant, but all trailing zeros are significant.

For example, a calculated MDL value of 0.074 ug/L will be reported as 0.074 and a calculated MDL value of 0.1 or 0.08 will be reported as 0.10 and 0.080, respectively.

3.4.11.2.8 Use additional Form(s) IX-IN if more preparation methods, instruments, and wavelengths or masses are used. Note that the date on this form shall not exceed the analysis dates in the CSF or precede them by more than one year.

3.4.11.2.9 Use the "Comments" section to indicate alternative wavelengths and the conditions under which they are used.

3.4.12 ICP-AES Interelement Correction Factors (Annually) [Form XA-IN]. This form is not required for Level 2a deliverables.

3.4.12.1 Purpose. This form documents for each ICP-AES instrument the interelement correction factors applied by the Contractor to obtain data for the SDG. Although the correction factors are

determined annually, a copy of the results of the annual interelement correction factors shall be included with each CSF on Form XA-IN and Form XB-IN as appropriate.

3.4.12.2 Instructions. Complete the header information according to instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.12.2.1 Enter the ICP-AES Instrument ID, which is a unique number designated by the Contractor to identify each ICP-AES instrument used to produce data in the CSF. If more than one ICP-AES instrument is used, submit additional Form(s) XA-IN as appropriate.

3.4.12.2.2 Report the date (formatted as MM/DD/YYYY) on which these correction factors were determined for use.

3.4.12.2.3 Under "Wavelength", list the wavelength in nm used for each ICP-AES analyte. If more than one wavelength is used, submit additional Form(s) XA-IN or Form(s) XB-IN as appropriate.

3.4.12.2.4 Under "Al", "Ca", "Fe", and "Mg", enter the correction factor (negative, positive, or zero) for each ICP-AES analyte. Correction factors for one other analyte shall be reported using the empty column and listing the analyte's chemical symbol in the blank two-space header field provided for that column.

3.4.12.2.5 If corrections are not applied for an analyte, a zero shall be entered for that analyte to indicate that the corrections were determined to be zero. Correction factors for more than one additional analyte shall be reported using Form XB-IN.

NOTE: Correction factors for Al, Ca, Fe, and Mg are all required and are to be listed first (as they appear on Form XA-IN).

3.4.13 ICP-AES Interelement Correction Factors (Annually) [Form XB-IN]. This form is not required for Level 2a deliverables.

3.4.13.1 Purpose. This form is used if correction factors for analytes other than Al, Ca, Fe, Mg, and one more analyte of the Contractor's choice were applied to the analytes analyzed by ICP-AES.

3.4.13.2 Instructions. Complete this form following the instructions for Form XA-IN (see Exhibit B, Section 3.4.12) by listing the chemical symbol for additional analytes in the heading of the empty columns in the two-space fields provided.

3.4.13.2.1 Columns of correction factors for additional analytes shall be entered left to right starting on Form XA-IN and proceeding to Form XB-IN, according to the alphabetical order of their chemical symbols.

3.4.14 Internal Standard Association [Form XI-IN]. This form is not required for Level 2a deliverables.

3.4.14.1 Purpose. This form is used to report the associated internal standards for each target analyte for each ICP-MS instrument used in SDG analyses.

3.4.14.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.14.2.1 For "ICP-MS Instrument ID", enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two ICP-MS instruments within a laboratory may have the same ICP-MS Instrument ID.

3.4.14.2.2 Report the date (formatted as MM/DD/YYYY) on which the ICP-MS tune was performed. This date shall not exceed the dates of analysis by ICP-MS in the CSF.

3.4.14.2.3 For "Assoc. Internal Standard 1", enter the chemical symbol of the internal standard associated with each target analyte in the run.

3.4.14.2.4 For "Assoc. Internal Standard 2" enter the chemical symbol of the second internal standard associated with each target analyte in the run, if a second internal standard is used for the analyte. Otherwise leave blank.

3.4.15 Preparation Log [Form XII-IN]

3.4.15.1 Purpose. This form is used to report the preparation run log.

3.4.15.1.1 All field samples and all Quality Control (QC) preparations (including duplicates, matrix spikes, LCSs, Performance Evaluation (PE) samples, Preparation Blanks (PBs), and re-preparations) associated with the SDG shall be reported on Form XII-IN. In addition, for mercury and cyanide analyses, all prepared calibration standards and QC standards (e.g., ICV, CCV, ICB, CCB) shall also be reported on Form XII-IN for Level 2b deliverables. For Level 2a deliverables, this form will contain data for the samples and associated QC.

3.4.15.1.2 Submit one Form XII-IN per batch, per method, if no more than 32 preparations, including QC preparations, were performed. If more than 32 preparations per batch, per method, were performed, then submit additional copies of Form XII-IN as appropriate. Submit a separate Form XII-IN for each batch.

3.4.15.1.3 The order in which the Preparation Logs are submitted is very important. Form XII-IN shall be organized by method, by batch. Later batches within a method shall follow earlier ones. Each batch shall start on a separate Form XII-IN.

3.4.15.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.15.2.1 For "Preparation Method", enter the method of preparation for which the preparations listed on Form XII-IN were made, as specified in Exhibit B, Section 3.4.11.2.4.

3.4.15.2.2 Under "EPA Sample No.", enter EPA sample number of each sample in the SDG, and of all other preparations such as duplicates, matrix spikes, LCSs, PBs, and re-preparations (all formatted according to Exhibit B, Table 2). Enter any digested/distilled calibration or QC standards. All EPA sample numbers shall be listed in ascending alphanumeric order, continuing to the next Form XII-IN if applicable.

3.4.15.2.3 Under "Preparation Date", enter the date (formatted MM/DD/YYYY) on which each sample was prepared for analysis by the method indicated in the header section of the form.

NOTE: The date never changes on a single Form XII-IN because the form shall be submitted per batch.

3.4.15.2.4 Under "Initial Weight/Volume", enter the wet weight (in g) of each soil/sediment sample or the initial volume (in mL) of each aqueous/water sample prepared for analysis by the method indicated in the header section of the form.

3.4.15.2.5 Under "Volume", enter the final volume (in mL) of the preparation for each sample prepared for analysis by the method indicated in the header section of the form. This field shall have a value for each sample listed.

3.4.16 Analysis Run Log [Form XIII-IN]. This form is not required for Level 2a deliverables.

3.4.16.1 Purpose. This form is used to report the sample analysis run log.

3.4.16.1.1 A run is defined as the totality of analyses performed by an instrument throughout the sequence initiated by, and including, the first SOW-required calibration standard or tune standard, and terminated by, and including, the CCV and CCB following the last SOW-required analytical sample.

3.4.16.1.2 All field samples and all QC analyses (including tunes, calibration standards, ICVs, CCVs, ICBs, CCBs, ICSs, LCSs, PBs, PE samples, duplicates, serial dilutions, matrix spikes, and post-digestion/distillation spikes) associated with the SDG shall be reported on Form XIII-IN. The run shall be continuous and inclusive of all analyses performed on the particular instrument during the run.

3.4.16.1.3 Submit one Form XIII-IN per run if no more than 32 analyses, including instrument calibration, were analyzed in the run. If more than 32 analyses were performed in the run, submit additional Form(s) XIII-IN as appropriate.

3.4.16.1.4 The order in which the Analysis Run Logs are submitted is very important. Form XIII-IN shall be organized by method, and by run. Later runs within a method shall follow earlier ones. Each analytical run shall start on a separate Form XIII-IN. Therefore, an instrument calibration or tune shall be the first entry on the form for each new run. In addition, the run is considered to have ended if it is interrupted for any reason, including termination for failing QC parameters.

3.4.16.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.16.2.1 For "Instrument ID", enter the Instrument ID which shall be an identifier designated by the Contractor to uniquely identify each instrument used to produce data which are required to be reported in the SDG deliverable. If more than one instrument is used, submit additional Form(s) XIII-IN as appropriate. The Instrument ID shall exactly match that reported on Forms IVA, IVB, IX, XA, XB, XI, XIV, XV, and XVI.

3.4.16.2.2 For "Analysis Method", enter the method code according to the specifications in Exhibit B, Section 3.4.2.2.5.3.

3.4.16.2.3 For "Start Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was started.

3.4.16.2.4 For "End Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was ended.

3.4.16.2.5 Under "EPA Sample No.", enter EPA sample number of each analysis, including all QC operations applicable to the SDG (formatted according to Exhibit B, Table 2). All EPA sample numbers shall be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XIII-IN for the instrument run, if applicable. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical run, shall be reported. Those analyses shall be identified with EPA sample number of "ZZZZZZ".

3.4.16.2.6 Under "D/F", enter the dilution factor by which the final digestate or distillate needed to be diluted for each analysis to be performed. The dilution factor does not include the dilution inherent in the preparation as specified by the preparation procedures in Exhibit D.

3.4.16.2.7 The dilution factor is required for all entries on Form XIII-IN.

NOTE: For a particular sample, a dilution factor of "1.0" shall be entered if the digestate or distillate was analyzed without adding any further volume of dilutant, or any other solutions to the "Volume", or an aliquot of the "Volume" listed on Form XII-IN for that sample.

3.4.16.2.8 For USEPA-supplied solutions such as ICVs and ICSs, a dilution factor shall be entered if the supplied solution had to be diluted to a dilution different from that specified by the instructions provided with the solution. The dilution factor reported in such a case shall be that which would make the reported true values on the appropriate form for the solution equal to those that were supplied with the solution by USEPA. For instance, ICV-2(0887) has a true value of 104.0 ug/L at a 20-fold dilution. If the solution is prepared at a 40-fold dilution, a dilution factor of "2.0" shall be entered on Form XIII-IN and the uncorrected instrument reading is compared to a true value of 52 ug/L. In this example, Form IIA-IN will have a true value of 104.0 regardless of the dilution used. The found value for the ICV shall be corrected for the dilution listed on Form XIII-IN using the following formula:

EQ. 9 ICV/CCV Correction for Dilution

$$\text{Found value on Form II} = \text{Instrument readout (ug/L)} \times D/F$$

3.4.16.2.9 Under "Time", enter the time (in military format - HHMM) that each analysis was performed.

3.4.16.2.10 Under "Analytes", enter "X" in the column of the designated analyte to indicate that the analyte value was used from the reported analysis to report data in the SDG. Leave the column empty for each analyte if the analysis was not used to report the particular analyte.

3.4.16.2.11 Entering "X" appropriately is very important. The "X" is used to link the samples with their related QC. It also links the dilution factor with the appropriate result reported on Forms I-VIII. For each analyte result reported on

any of the Forms I-VIII, there shall be one, and only one, properly identified entry on Form XIII-IN for which an "X" is entered in the column for that analyte.

3.4.16.2.12 If, on Form XIII-IN, an "X" is entered in the column for an analyte for a field sample associated with a dilution factor greater than 1.0, flag the data for that analyte with a "D" on the appropriate Form IA-IN or Form IB-IN.

3.4.17 ICP-MS Tune [Form XIV-IN]. This form is not required for Level 2a deliverables.

3.4.17.1 Purpose. This form is used to report the tuning results for each ICP-MS instrument used in SDG analyses.

3.4.17.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.17.2.1 For "ICP-MS Instrument ID", enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two ICP-MS instruments within a laboratory may have the same ICP-MS Instrument ID.

3.4.17.2.2 Report the date (formatted as MM/DD/YYYY) on which the ICP-MS tune was performed. This date shall not exceed the dates of analysis by ICP-MS in the CSF.

3.4.17.2.3 For "Avg. Measured Mass (amu)", enter the average mass calculated from the five or more tune integrations (in atomic mass units) measured for each isotope.

3.4.17.2.4 For "Avg. Peak Width (amu)" enter the average peak width calculated from the analysis (in atomic mass units) at the percent of peak height recommended by the instrument manufacturer for each isotope.

3.4.17.2.5 For "% Height", enter the percent of peak height at which the Average Peak Width was measured.

3.4.17.2.6 For "%RSD", enter the percent Relative Standard Deviation of the absolute signals (intensities) for each isotope calculated from the five or more tune integrations.

3.4.18 ICP-MS Internal Standards Relative Intensity Summary [Form XV-IN]. This form is not required for Level 2a deliverables.

3.4.18.1 Purpose. This form is used to report the relative internal standard intensity levels during an ICP-MS run. The relative intensity of each of the internal standards in all analyses performed by ICP-MS must be reported on the form. If more than one ICP-MS instrument or run is used, submit additional Form(s) XV-IN as appropriate. All runs for the lowest alphanumeric instrument must be reported in ascending order before proceeding to the runs for the next highest instrument.

3.4.18.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.18.2.1 For "ICP-MS Instrument ID", enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two ICP-MS instruments within a laboratory may have the same ICP-MS Instrument ID.

3.4.18.2.2 For "Start Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was started.

3.4.18.2.3 For "End Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was ended.

3.4.18.2.4 Under "EPA Sample No.", enter EPA sample number of each analysis, including all QC operations applicable to the SDG. All EPA sample numbers must be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XV for the instrument run, if applicable. The order must agree with the order reported on Form XIII-IN for that run. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical run, must be reported. Those analyses must be identified with an EPA Sample Number of "ZZZZZZ." Samples identified as "ZZZZZZ" need not have intensities reported for internal standards.

3.4.18.2.5 Under "Time", enter the time (in military format - HHMM) that each analysis was performed.

3.4.18.2.6 Under "Internal Standards %RI For:", enter the chemical symbol and elemental expression number of the internal standard in the "Element" header field provided to indicate the internal standard and elemental expression for which the Relative Intensity (RI) of the internal standards will be calculated in that column.

3.4.18.2.6.1 In the "Element" column, enter the internal standard relative intensity (to the nearest whole number) of the internal standard for each sample analysis listed on the form (excluding samples identified as "ZZZZZZ"). The internal standard relative intensity (%RI) is calculated using the following formula:

EQ. 10 Internal Standard Percent Relative Intensity

$$\%RI = \frac{I_n}{I_o} \times 100$$

WHERE, "I_o" is the intensity of the internal standard in the blank calibration standard and "I_n" is the intensity of the internal standard in the EPA sample number in the same units.

3.4.18.2.7 Under the "Q" column to the right of each "Element" column, enter an "R" if the %RI for a field sample, PE, duplicate, or spike is less than 60 or greater than 125; otherwise leave the field blank.

3.4.18.2.8 Columns of internal standard RI must be entered left to right, starting with the internal standards of the lower mass on the first Form XV-IN and proceeding to the following Form XV-IN as appropriate. All Forms XV-IN for the lowest numeric instrument must be reported in ascending order by the run number before proceeding to the next Form XV-IN.

3.4.18.3 All field samples and all QC samples (including calibration standards, ICVs, CCVs, ICBs, CCBs, ICSs, LCS, PBs, serial dilutions, duplicates, PE samples, and spikes) associated with the SDG must be reported on Form XV-IN. The run must be

continuous and inclusive of all analyses performed on the particular instrument during the run.

3.4.18.4 Submit one Form XV-IN per run if no more than 32 analyses, including instrument calibration, were analyzed in the run. If more than 32 analyses were performed in the run, submit additional Form(s) XV-IN as appropriate. Each new run must be started on the first line of Form XV-IN.

3.4.19 Initial Calibration [Form XVI-IN]. This form is not required for Level 2a deliverables.

3.4.19.1 Purpose: This form is used to report instrument response and concentration data for each standard in the initial calibration of an instrument. Submit one set of forms for each calibration performed for each instrument used to analyze samples in the reported SDG.

3.4.19.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.19.3 For Instrument ID, enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two instruments within a laboratory may have the same Instrument ID.

3.4.19.4 For "Start Date", enter the date (formatted MM/DD/YYYY) that the calibration was begun.

3.4.19.5 Under "True", enter the expected concentration or amount of each analyte in the calibration standard or level. It is not required to enter a value for each analyte in every standard, so long as a value is entered for each concentration used to calibrate the instrument for the analyte.

3.4.19.6 Under "Found", enter the actual measured found concentration or amount of each analyte in the calibration standard or level obtained when the calibration standard was re-fitted to the calibration equation.

3.4.19.7 Under "%D", enter the percent difference for each analyte at each concentration or amount used to calibrate the instrument other than the blank standard (S0). Calculate the percent difference (reported to the nearest whole number) using the following formula:

EQ. 11 Percent Difference

$$\%D = \frac{\text{True} - \text{Found}}{\text{True}}$$

3.4.19.8 Since a minimum of six levels of calibration are required (a blank plus five standards), submit a minimum of two Forms XVI-IN for each calibration performed. Submit a set of Forms XVI-IN for each calibration performed.

3.5 Sample Log-In Sheet [Form DC-1]

3.5.1 Purpose. This form is used to document the receipt and inspection of samples and containers. At least one original Form DC-1 is required for each sample shipping container (e.g., cooler). If the samples in a single sample shipping container must be assigned to

3.5.2 more than one SDG, the original Form DC-1 shall be placed with the deliverables for the SDG of the lowest alpha-numeric number and a copy of Form DC-1 shall be placed with the deliverables for the other SDG(s). The copies should be identified as "copy(ies)", and the location of the original should be noted on the copies.

3.5.3 Instructions

3.5.3.1 Sign and date the airbill. (If an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information).

3.5.3.2 Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in Item 1.

3.5.3.3 Record the custody seal numbers in Item 2.

3.5.3.4 Open the container, remove the enclosed sample documentation, and record the presence/absence of USEPA forms (i.e., Traffic Reports/Chain of Custody Records, packing lists) and airbills or airbill stickers in Items 3 and 4. Specify if there is an airbill present or an airbill sticker in Item 4. Record the airbill or sticker number in Item 5.

3.5.3.5 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample bottles (i.e., intact, broken, leaking) and presence or absence of sample tags in Items 6 and 7.

3.5.3.6 Record the presence or absence of a cooler temperature indicator bottle in Item 8.

3.5.3.7 Record the cooler temperature in Item 9.

3.5.3.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and mark the appropriate answer in Item 10.

3.5.3.9 The log-in date should be recorded at the top of Form DC-1; record the date and time of cooler receipt at the laboratory in Items 11 and 12.

3.5.3.10 If there are no problems observed during receipt, sign and date (include the time) Form DC-1 and Traffic Report/Chain of Custody Record, and write the sample numbers in the "EPA Sample No." column.

3.5.3.11 Record the pH for all aqueous/water samples received.

3.5.3.12 Record the appropriate sample tags and assigned laboratory numbers, if applicable.

3.5.3.13 Any comments should be made in the "Remarks" column.

3.5.3.14 Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the "Sample Transfer" block located in the bottom left corner of Form DC-1. Sign and date the sample transfer block.

3.5.3.15 For Items 1, 3, 4, 6, 7, 8, and 10, circle the appropriate response. Responses can be underlined if this form is completed by automated equipment. Unused columns and spaces shall be crossed out, initialed, and dated.

3.5.3.16 If there are problems observed during receipt (including samples that have not been preserved to the proper pH) or an answer marked with an asterisk (e.g., "absent*") was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

3.6 Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

3.6.1 Purpose. The CSF Inventory Sheet is used to record both the inventory of Complete SDG File (CSF) documents and the number of documents in the original Sample Data Package which is sent to the USEPA Region.

3.6.2 Instructions

3.6.2.1 Organize all EPA-CSF documents as described in Exhibit B, Sections 2 and 3. Assemble the documents in Exhibit B, Section 2 in the order specified on Form DC-2, and stamp each page with the consecutive number. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record in the "Comments" section on Form DC-2 all intentional gaps in the page numbering sequence (for example, "page numbers not used, XXXX-XXXX, XXXX-XXXX"). If there are no documents for a specific document type, enter an "NA" in the empty space.

3.6.2.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review Form DC-2 to determine if it is most appropriate to place them under Categories 32 through 35. Category 35 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

3.6.2.3 If it is necessary to insert new or inadvertently omitted documents, the Contractor shall follow these steps:

- Number all documents to be inserted with the next sequential numbers and file the inserts in their logical positions within the CSF (e.g., file document 1000 between documents 6 and 7).
- Identify where the inserts are filed in the CSF by recording the document numbers and their locations under the "Other Records" section of Form DC-2 (e.g., document 1000 is filed between 6 and 7).

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

USEPA - CLP

COVER PAGE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

SOW No.: _____

EPA Sample No.	Lab Sample ID

		ICP-AES	ICP-MS
Were ICP-AES and ICP-MS interelement corrections applied?	(Yes/No)	_____	_____
Were ICP-AES and ICP-MS background corrections applied?	(Yes/No)	_____	_____
If yes, were raw data generated before application of background corrections?	(Yes/No)	_____	_____

The laboratory did not receive any instructions with this SDG to modify the SOW standard laboratory sample preparation procedures (e.g., subsampling). To aid in the determination of data usability with respect to project decisions, any modifications performed are described below.

Comments:

_____

_____

_____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy Data Package and in the electronic data submitted has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

USEPA - CLP

1A-IN

INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Matrix: _____ Lab Sample ID: _____

% Solids: _____ Date Received: _____

Concentration Units (**ug/L**, **ug**, or **mg/kg** dry weight):

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum				
7440-36-0	Antimony				
7440-38-2	Arsenic				
7440-39-3	Barium				
7440-41-7	Beryllium				
7440-43-9	Cadmium				
7440-70-2	Calcium				
7440-47-3	Chromium				
7440-48-4	Cobalt				
7440-50-8	Copper				
7439-89-6	Iron				
7439-92-1	Lead				
7439-95-4	Magnesium				
7439-96-5	Manganese				
7439-97-6	Mercury				
7440-02-0	Nickel				
7440-09-7	Potassium				
7782-49-2	Selenium				
7440-22-4	Silver				
7440-23-5	Sodium				
7440-28-0	Thallium				
7440-62-2	Vanadium				
7440-66-6	Zinc				
57-12-5	Cyanide				

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

_____

_____

_____

EPA SAMPLE NO.

USEPA - CLP

1B-IN

INORGANIC ANALYSIS DATA SHEET

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Matrix: _____ Lab Sample ID: _____

% Solids: _____ Date Received: _____

Concentration Units (**ug/L**, **ug**, or **mg/kg** dry weight):

CAS No.	Analyte	Concentration	C	Q	M

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

_____

_____

_____

USEPA - CLP

2A-IN

INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Initial Calibration Verification Source: _____

Continuing Calibration Verification Source: _____

Concentration Units: ug/L

	Initial Calibration Verification			Continuing Calibration Verification					M
Analyte	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

(1) Control Limits: Mercury 85-115; Other Metals 90-110; Cyanide 85-115

USEPA - CLP

3-IN

BLANKS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Preparation Blank Matrix (soil/water/wipe/filter): _____

Preparation Blank Concentration Units (ug/L, ug, or mg/kg): _____

Analyte	Initial Calibration Blank (ug/L)		Continuing Calibration Blank (ug/L)						Preparation Blank		
		C	1	C	2	C	3	C		C	M
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide											

USEPA - CLP

4A-IN

ICP-AES INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

ICP-AES Instrument ID: _____ ICSA Source: _____

ICSB Source: _____

Concentration Units: ug/L

Analyte	True		Found			
	Sol. A	Sol. AB	Sol. A	%R	Sol. AB	%R
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

USEPA - CLP

4B-IN

ICP-MS INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ ICSA Source: _____

ICSB Source: _____

Concentration Units: ug/L

Analyte	True		Found			
	Sol. A	Sol. AB	Sol. A	%R	Sol. AB	%R
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Molybdenum						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Titanium						
Vanadium						
Zinc						

USEPA - CLP

5A-IN

MATRIX SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Matrix: _____

% Solids for Sample: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

_____

_____

_____

USEPA - CLP

5B-IN

POST-DIGESTION SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Matrix: _____

Concentration Units: (ug/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

_____

_____

_____

USEPA - CLP
6-IN
DUPLICATES

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Matrix: _____

% Solids for Sample: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

USEPA - CLP

7-IN

LABORATORY CONTROL SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Analyte	Aqueous/Water (ug/L), Soil/Sediment (mg/kg), Wipe/Filter (ug)		
	True	Found	%R
Aluminum			
Antimony			
Arsenic			
Barium			
Beryllium			
Cadmium			
Calcium			
Chromium			
Cobalt			
Copper			
Iron			
Lead			
Magnesium			
Manganese			
Mercury			
Nickel			
Potassium			
Selenium			
Silver			
Sodium			
Thallium			
Vanadium			
Zinc			
Cyanide			

USEPA - CLP

8-IN

ICP-AES AND ICP-MS SERIAL DILUTIONS

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Matrix: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Initial Sample Result (I)	C	Serial Dilution Result (S)	C	% Difference	Q	M
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							

USEPA - CLP

9-IN

METHOD DETECTION LIMIT (MDL) (ANNUALLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Instrument Type: _____ Instrument ID: _____ Date: _____

Preparation Method: _____

Concentration Units (ug/L, mg/kg, or ug): _____

Analyte	Wavelength/Mass	MDL
Aluminum		
Antimony		
Arsenic		
Barium		
Beryllium		
Cadmium		
Calcium		
Chromium		
Cobalt		
Copper		
Iron		
Lead		
Magnesium		
Manganese		
Mercury		
Nickel		
Potassium		
Selenium		
Silver		
Sodium		
Thallium		
Vanadium		
Zinc		
Cyanide		

Comments:

_____

_____

_____

USEPA - CLP

10A-IN

ICP-AES INTERELEMENT CORRECTION FACTORS (ANNUALLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

ICP-AES Instrument ID: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for: Al	Ca	Fe	Mg	____
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

_____

_____

_____

USEPA - CLP

10B-IN

ICP-AES INTERELEMENT CORRECTION FACTORS (ANNUALLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

ICP-AES Instrument ID: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for: _____	_____	_____	_____	_____
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

_____

_____

_____

USEPA - CLP

11-IN

INTERNAL STANDARD ASSOCIATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ Date: _____

Analyte	Assoc. Internal Standard 1	Assoc. Internal Standard 2
Aluminum		
Antimony		
Arsenic		
Barium		
Beryllium		
Cadmium		
Calcium		
Chromium		
Cobalt		
Copper		
Iron		
Lead		
Magnesium		
Manganese		
Nickel		
Potassium		
Selenium		
Silver		
Sodium		
Thallium		
Vanadium		
Zinc		

USEPA - CLP

12-IN

PREPARATION LOG

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Preparation Method: _____

EPA Sample No.	Preparation Date	Initial Weight/Volume (g) or (mL)	Final Volume (mL)

USEPA - CLP

13-IN

ANALYSIS RUN LOG

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Instrument ID: _____ Analysis Method: _____

Start Date: _____ End Date: _____

EPA Sample No.	D/F	Time	Analytes																									
			Al	Sb	As	Ba	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Hg	Ni	K	Se	Ag	Na	Tl	V	Zn	CN		

USEPA - CLP

14-IN

ICP-MS TUNE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ Date: _____

Element - Mass	Avg. Measured Mass (amu)	Average Peak Width (amu)	%Height	%RSD
Be - 9				
Mg - 24				
Mg - 25				
Mg - 26				
Co - 59				
In - 113				
In - 115				
Pb - 206				
Pb - 207				
Pb - 208				

Comments:

_____

_____

_____

USEPA - CLP

15-IN

ICP-MS INTERNAL STANDARDS RELATIVE INTENSITY SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ Start Date: _____ End Date: _____

EPA Sample No.	Time	Internal Standards %RI For:									
		Element	Q	Element	Q	Element	Q	Element	Q	Element	Q

USEPA - CLP

16-IN

INITIAL CALIBRATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ Mod. Ref. No.: _____ SDG No.: _____

Instrument ID: _____ Start Date: _____

Concentration Units: ug/L

Analyte	True	Found	%D	True	Found	%D	True	Found	%D
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Control Limits ±30

SAMPLE LOG-IN SHEET

Lab Name		Page __ of ___
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	Sample Delivery Group No.	Mod. Ref. No.

Remarks:	
1. Custody Seal(s)	Present/Absent* Intact/Broken
2. Custody Seal NOs.	_____ _____
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*
4. Airbill	Airbill/Sticker Present/Absent*
5. Airbill No.	_____ _____
6. Sample Tags Sample Tag Numbers	Present/Absent* Listed/Not Listed on Traffic Report/Chain of Custody Record
7. Sample Condition	Intact/Broken*/ Leaking
8. Cooler Temperature Indicator Bottle	Present/Absent*
9. Cooler Temperature	_____
10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree?	Yes/No*
11. Date Received at Lab	_____
12. Time Received	_____

Sample Transfer	
Fraction	Fraction
Area#	Area#
By	By
On	On

	EPA Sample #	Aqueous/ Water Sample pH	Corresponding		Remarks: Condition of Sample Shipment, etc.
			Sample Tag #	Assigned Lab #	
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____

CITY/STATE _____

CASE NO. _____ SDG NO. _____

SDG NOs. TO FOLLOW _____ _____ _____

MOD. REF. NO. _____

CONTRACT NO. _____

SOW NO. _____

All documents delivered in the Complete SDG File must be original documents where possible. (Reference - Exhibit B Section 2.6)

		PAGE NOs.		CHECK	
		FROM	TO	LAB	REGION
1.	Inventory Sheet (DC-2)				
2.	SDG Narrative				
3.	Sample Log-In Sheet (DC-1)				
4.	Traffic Report/Chain of Custody Record(s)				
5.	Cover Page				
Inorganic Analysis					
6.	Data Sheet (Form I-IN)				
7.	Initial & Continuing Calibration Verification (Form IIA-IN)				
8.	Blanks (Form III-IN)				
9.	ICP-AES Interference Check Sample (Form IVA-IN)				
10.	ICP-MS Interference Check Sample (Form IVB-IN)				
11.	Matrix Spike Sample Recovery (Form VA-IN)				
12.	Post-Digestion Spike Sample Recovery (Form VB-IN)				
13.	Duplicates (Form VI-IN)				
14.	Laboratory Control Sample (Form VII-IN)				
15.	ICP-AES and ICP-MS Serial Dilutions (Form VIII-IN)				
16.	Method Detection Limit (Annually) (Form IX-IN)				
17.	ICP-AES Interelement Correction Factors (Annually) (Form XA-IN)				
18.	ICP-AES Interelement Correction Factors (Annually) (Form XB-IN)				
19.	Internal Standard Association (Form XI-IN)				
20.	Preparation Log (Form XII-IN)				
21.	Analysis Run Log (Form XIII-IN)				
22.	ICP-MS Tune (Form XIV-IN)				

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

	PAGE NOs.		CHECK	
	FROM	TO	LAB	REGION
23. ICP-MS Internal Standards Relative Intensity Summary (Form XV-IN)				
24. Initial Calibration (Form XVI-IN)				
25. ICP-AES Raw Data				
26. ICP-MS Raw Data				
27. Mercury Raw Data				
28. Cyanide Raw Data				
29. Preparation Logs Raw Data				
30. Percent Solids Determination Log				
31. USEPA Shipping/Receiving Documents				
Airbill (No. of Shipments_____)				
Sample Tags				
Sample Log-In Sheet (Lab)				
32. Misc. Shipping/Receiving Records (list all individual records) Communication Logs				
_____				
_____				
33. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)				
_____				
_____				
34. Internal Original Sample Prep & Analysis Records (describe or list)				
Prep Records _____				
Analysis Records _____				
PE Instructions _____				
Description _____				
35. Other Records (describe or list)				
Communication Logs				
_____				
_____				

36. Comments:

_____

_____

_____

Completed by:
(CLP Lab) _____ _____ _____
(Signature) (Print Name & Title) (Date)

Audited by:
(USEPA) _____ _____ _____
(Signature) (Print Name & Title) (Date)

EXHIBIT C

INORGANIC TARGET ANALYTE LIST WITH CONTRACT REQUIRED QUANTITATION LIMITS

Exhibit C - Inorganic Target Analyte List With Contract Required Quantitation Limits

Table of Contents

Section Page

1.0 INORGANIC TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) 3

1.0 INORGANIC TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLS)

Table 1. CRQLS for ICP-AES

Analyte	CAS Number	ICP-AES CRQL for Water[1,2,3] (µg/L)	ICP-AES CRQL for Soil[1,2,3,4] (mg/kg)	ICP-AES for Wipes [1,2] (µg)	ICP-AES for Filters [1,2] (µg)
Aluminum	7429-90-5	200	20	20	2
Antimony	7440-36-0	60	6	6	0.6
Arsenic	7440-38-2	10	1	1	0.1
Barium	7440-39-3	200	20	20	2
Beryllium	7440-41-7	5	0.5	0.5	0.05
Cadmium	7440-43-9	5	0.5	0.5	0.05
Calcium	7440-70-2	5000	500	500	50
Chromium	7440-47-3	10	1	1	0.10
Cobalt	7440-48-4	50	5	5	0.5
Copper	7440-50-8	25	2.5	2.5	0.25
Iron	7439-89-6	100	10	10	1
Lead	7439-92-1	10	1	1	0.1
Magnesium	7439-95-4	5000	500	500	50
Manganese	7439-96-5	15	1.5	1.5	0.15
Nickel	7440-02-0	40	4	4	0.4
Potassium	7440-09-7	5000	500	500	50
Selenium	7782-49-2	35	3.5	3.5	0.35
Silver	7440-22-4	10	1	1	0.10
Sodium	7440-23-5	5000	500	500	50
Thallium	7440-28-0	25	2.5	2.5	0.25
Vanadium	7440-62-2	50	5	5	0.5
Zinc	7440-66-6	60	6	6	0.60

[1] The CRQLs are the minimum levels of quantitation acceptable under the contract Statement of Work (SOW).

[2] Subject to the restrictions specified in Exhibit D, any analytical method specified in ISM01.2 Exhibit D may be utilized as long as the documented MDLs are less than one-half the CRQLs.

[3] Changes to the Inorganic Target Analyte List (TAL) (e.g., adding an additional analyte) or CRQLs may be requested under the modified analysis clause in the contract.

[4] The CRQLs for soil/sediment are based on 100% solids and on the minimum weights and volumes specified in Exhibit D.

Table 2. CRQLS for ICP-MS

Analyte	CAS Number	ICP-MS CRQL for Water[1,2,3] (µg/L)	ICP-MS CRQL for Soil[1,2,3,4] (mg/kg)
Aluminum	7429-90-5	20	--
Antimony	7440-36-0	2	1
Arsenic	7440-38-2	1	0.5
Barium	7440-39-3	10	5
Beryllium	7440-41-7	1	0.5
Cadmium	7440-43-9	1	0.5
Calcium	7440-70-2	500	--
Chromium	7440-47-3	2	1
Cobalt	7440-48-4	1	0.5
Copper	7440-50-8	2	1
Iron	7439-89-6	200	--
Lead	7439-92-1	1	0.5
Magnesium	7439-95-4	500	--
Manganese	7439-96-5	1	0.5
Nickel	7440-02-0	1	0.5
Potassium	7440-09-7	500	--
Selenium	7782-49-2	5	2.5
Silver	7440-22-4	1	0.5
Sodium	7440-23-5	500	--
Thallium	7440-28-0	1	0.5
Vanadium	7440-62-2	5	2.5
Zinc	7440-66-6	2	1

[1] The CRQLs are the minimum levels of quantitation acceptable under the contract Statement of Work (SOW).

[2] Subject to the restrictions specified in Exhibit D, any analytical method specified in ISM01.2 Exhibit D may be utilized as long as the documented MDLs are less than one-half the CRQLs.

[3] Changes to the Inorganic Target Analyte List (TAL) (e.g., adding an additional analyte) or CRQLs may be requested under the modified analysis clause in the contract.

[4] The CRQLs for soil/sediment are based on 100% solids and on the minimum weights and volumes specified in Exhibit D.

Table 3. CRQLS for Mercury by Cold Vapor Atomic Absorption (CVAA)

Analyte	CAS Number	CRQL for Water[1,3] (µg/L)	CRQL for Soil[1,2,3] (mg/kg)
Mercury	7439-97-6	0.2	0.1

[1] The CRQLs are the minimum levels of quantitation acceptable under the contract Statement of Work (SOW).

[2] The CRQL for soil/sediment are based on 100% solids and on the minimum weights and volumes specified in Exhibit D.

[3] Changes to the CRQL may be requested under the Modified Analysis clause in the contract.

Table 4. CRQLS for Cyanide by Spectrophotometry

Analyte	CAS Number	CRQL for Water[1,2,3,5] (µg/L)	CRQL for Soil[1,2,3,4,5] (mg/kg)
Cyanide	57-12-5	10	0.5

[1] The CRQLs are the minimum levels of quantitation acceptable under the contract Statement of Work (SOW).

[2] Subject to the restrictions specified in Exhibit D, any analytical method specified in ISM01.2 Exhibit D may be utilized as long as the documented MDLs are less than one-half the CRQLs.

[3] Changes to the CRQL may be requested under the Modified Analysis clause in the contract.

[4] The CRQL for soil/sediment are based on 100% solids and on the minimum weights and volumes specified in Exhibit D.

[5] Use the CRQLs for cyanide regardless of the preparation or analysis method used to analyze the samples.

United States Environmental Protection Agency

Office of Solid Waste and Emergency Response

OSWER Document 9200.5-171-FS
EPA Publication 540-FS-07-001
August 2007

Multi-Media, Multi-Concentration, Organic Analytical Service for Superfund (SOM01.2)

Office of Superfund Remediation and Technology Innovation (OSRTI)
Analytical Services Branch (ASB) (5203P)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites are a series of volatile, semivolatile, pesticide, and Aroclor compounds that are analyzed using gas chromatography coupled with mass spectrometry (GC/MS) and gas chromatography with an electron capture detector (GC/ECD). The Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) offers an analytical service that provides data from the analysis of water and soil/sediment samples for organic compounds for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the organic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

This new organic analytical service provides a technical and contractual framework for laboratories to apply EPA/CLP analytical methods for the isolation, detection, and quantitative measurement of 52 volatile, 67 semivolatile, 21 pesticide, and 9 Aroclor target compounds in water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including Quality Assurance (QA), Quality Control (QC), and Standard Operating Procedures (SOPs), by which EPA evaluates the data. This service uses GC/MS and GC/ECD methods to analyze the target compounds.

Three data delivery turnarounds are available to CLP customers: 7-day, 14-day, and 21-day turnaround after laboratory receipt of the last sample in the set. In addition, there are 48-hour (for trace volatiles and volatiles) and 72-hour (for semivolatiles, pesticides, and Aroclors) preliminary data submission options available. Options under this service include a closed system purge-and-trap method for low-level volatile soil analysis and methanol preservation for medium-level volatile soil analysis. In addition, data users may request modifications to the SOW that may include, but are not limited to, additional compounds, sample matrices other than soil/sediment or water, lower quantitation limits, and other requirements to enhance method performance.

DATA USES

This analytical service provides data which EPA uses for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of a hazardous waste site including, but not limited to: site inspections; Hazard Ranking System (HRS) scoring; remedial investigations/Feasibility Studies (FSs); remedial design; treatability studies; and removal actions. In addition, this service provides data that will be available for use in Superfund enforcement/litigation activities.

TARGET COMPOUNDS

Table 1 lists the compounds for which this service is applicable and the corresponding quantitation limits. Specific quantitation limits are highly matrix-dependent.

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) for SOM01.2*

	Quantitation Limits				
	Trace Water by SIM (µg/L)	Trace Water (µg/L)	Low Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)
VOLATILES					
1. Dichlorodifluoromethane		0.50	5.0	5.0	250
2. Chloromethane		0.50	5.0	5.0	250
3. Vinyl Chloride		0.50	5.0	5.0	250
4. Bromomethane		0.50	5.0	5.0	250
5. Chloroethane		0.50	5.0	5.0	250
6. Trichlorofluoromethane		0.50	5.0	5.0	250
7. 1,1-Dicholoroethene		0.50	5.0	5.0	250
8. 1,1,2-Trichloro-1,2,2-trifluoroethane		0.50	5.0	5.0	250
9. Acetone		5.0	10	10	500
10. Carbon Disulfide		0.50	5.0	5.0	250
11. Methyl acetate		0.50	5.0	5.0	250
12. Methylene chloride		0.50	5.0	5.0	250
13. trans-1,2-Dichloroethene		0.50	5.0	5.0	250
14. Methyl tert-butyl ether		0.50	5.0	5.0	250
15. 1,1-Dichloroethane		0.50	5.0	5.0	250
16. cis-1,2-Dichloroethene		0.50	5.0	5.0	250
17. 2-Butanone		5.0	10	10	500
18. Bromochloromethane		0.50	5.0	5.0	250
19. Chloroform		0.50	5.0	5.0	250
20. 1,1,1-Trichloroethane		0.50	5.0	5.0	250
21. Cyclohexane		0.50	5.0	5.0	250
22. Carbon tetrachloride		0.50	5.0	5.0	250
23. Benzene		0.50	5.0	5.0	250
24. 1,2-Dichloroethane		0.50	5.0	5.0	250
25. 1,4-Dioxane			100	100	5000
26. Trichloroethene		0.50	5.0	5.0	250
27. Methylcyclohexane		0.50	5.0	5.0	250
28. 1,2-Dichloropropane		0.50	5.0	5.0	250
29. Bromodichloromethane		0.50	5.0	5.0	250
30. cis-1,3-Dichloropropene		0.50	5.0	5.0	250
31. 4-Methyl-2-pentanone		5.0	10	10	500
32. Toluene		0.50	5.0	5.0	250
33. trans-1,3-Dichloropropene		0.50	5.0	5.0	250
34. 1,1,2-Trichloroethane		0.50	5.0	5.0	250
35. Tetrachloroethane		0.50	5.0	5.0	250
36. 2-Hexanone		5.0	10	10	500
37. Dibromochloromethane		0.50	5.0	5.0	250
38. 1,2-Dibromochloroethane	0.050	0.50	5.0	5.0	250
39. Chlorobenzene		0.50	5.0	5.0	250

	Quantitation Limits				
	Trace Water by SIM (µg/L)	Trace Water (µg/L)	Low Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)
VOLATILES (CON'T)					
40. Ethylbenzene		0.50	5.0	5.0	250
41. o-Xylene		0.50	5.0	5.0	250
42. m, p-Xylene		0.50	5.0	5.0	250
43. Styrene		0.50	5.0	5.0	250
44. Bromoform		0.50	5.0	5.0	250
45. Isopropylbenzene		0.50	5.0	5.0	250
46. 1,1,2,2-Tetrachloroethane		0.50	5.0	5.0	250
47. 1,3-Dichlorobenzene		0.50	5.0	5.0	250
48. 1,4-Dichlorobenzene		0.50	5.0	5.0	250
49. 1,2-Dichlorobenzene		0.50	5.0	5.0	250
50. 1,2-Dibromo-3-chloropropane	0.050	0.50	5.0	5.0	250
51. 1,2,4-Trichlorobenzene		0.50	5.0	5.0	250
52. 1,2,3-Trichlorobenzene		0.50	5.0	5.0	250
SEMIVOLATILES	Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)
53. Benzaldehyde		5.0		170	5000
54. Phenol		5.0		170	5000
55. bis-(2-chloroethyl) ether		5.0		170	5000
56. 2-Chlorophenol		5.0		170	5000
57. 2-Methylphenol		5.0		170	5000
58. 2,2'-Oxybis (1-chloropropane)		5.0		170	5000
59. Acetophenone		5.0		170	5000
60. 4-Methylphenol		5.0		170	5000
61. N-Nitroso-di-n propylamine		5.0		170	5000
62. Hexachloroethane		5.0		170	5000
63. Nitrobenzene		5.0		170	5000
64. Isophorone		5.0		170	5000
65. 2-Nitrophenol		5.0		170	5000
66. 2,4-Dimethylphenol		5.0		170	5000
67. Bis (2-chloroethoxy) methane		5.0		170	5000
68. 2,4-Dichlorophenol		5.0		170	5000
69. Napthalene	0.10	5.0	3.3	170	5000
70. 4-Chloroaniline		5.0		170	5000
71. Hexachlorobutadiene		5.0		170	5000
72. Caprolactam		5.0		170	5000
73. 4-Chloro-3-methylphenol		5.0		170	5000
74. 2-Methylnapthalene	0.10	5.0	3.3	170	5000
75. Hexachlorocyclo-pentadiene		5.0		170	5000
76. 2,4,6-Trichlorophenol		5.0		170	5000
77. 2,4,5-Trichlorophenol		5.0		170	5000

* For volatiles, quantitation limits for medium soils are approximately 50 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 30 times the quantitation limits for low soils.

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) for SOM01.2* (Con't)

	Quantitation Limits				
	Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)
SEMIVOLATILES (CON'T)					
78. 1,1'-Biphenyl		5.0		170	5000
79. 2-Chloronapthalene		5.0		170	5000
80. 2-Nitroaniline		10		330	10000
81. Dimethylphthalate		5.0		170	5000
82. 2,6-Dinitrotoluene		5.0		170	5000
83. Acenaphthylene	0.10	5.0	3.3	170	5000
84. 3-Nitroaniline		10		330	10000
85. Acenaphthene	0.10	5.0	3.3	170	5000
86. 2,4-Dinitrophenol		10		330	10000
87. 4-Nitrophenol		10		330	10000
88. Dibenzofuran		5.0		170	5000
89. 2,4-Dinitrotoluene		5.0		170	5000
90. Diethylphthalate		5.0		170	5000
91. Fluorene	0.10	5.0	3.3	170	5000
92. 4-Chlorophenyl-phenyl ether		5.0		170	5000
93. 4-Nitroaniline		10		330	10000
94. 4,6-Dinitro-2-methylphenol		10		330	10000
95. N-Nitrosodiphenylamine		5.0		170	5000
96. 1,2,4,5-Tetra chlorobenzene		5.0		170	5000
97. 4-Bromophenyl-phenylether		5.0		170	5000
98. Hexachlorobenzene		5.0		170	5000
99. Atrazine		5.0		170	5000
100. Pentachlorophenol	0.20	10	6.7	330	10000
101. Phenanthrene	0.10	5.0	3.3	170	5000
102. Anthracene	0.10	5.0	3.3	170	5000
103. Carbazole		5.0		170	5000
104. Di-n-butylphthalate		5.0		170	5000
105. Fluoranthene	0.10	5.0	3.3	170	5000
106. Pyrene	0.10	5.0	3.3	170	5000
107. Butylbenzylphthalate		5.0		170	5000
108. 3,3'-Dichlorobenzidine		5.0		170	5000
109. Benzo (a) anthracene	0.10	5.0	3.3	170	5000
110. Chrysene	0.10	5.0	3.3	170	5000
111. Bis (2-ethylhexyl) phthalate		5.0		170	5000
112. Di-n-octylphthalate		5.0		170	5000
113. Benzo (b) fluoroanthene	0.10	5.0	3.3	170	5000
114. Benzo (k) fluoroanthene	0.10	5.0	3.3	170	5000

	Quantitation Limits				
	Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)
SEMIVOLATILES (CON'T)					
115. Benzo (a) pyrene	0.10	5.0	3.3	170	5000
116. Indeno (1,2,3-cd)-pyrene	0.10	5.0	3.3	170	5000
117. Dibenzo (a,h)-anthracene	0.10	5.0	3.3	170	5000
118. Benzo (g,h,i) perylene	0.10	5.0	3.3	170	5000
119. 2,3,4,6-Tetrachlorophenol		5.0		170	5000
PESTICIDES	**Water (µg/L)**			**Soil (µg/kg)**	
120. alpha-BHC	0.050			1.7	
121. beta-BHC	0.050			1.7	
122. delta-BHC	0.050			1.7	
123. gamma-BHC (Lindane)	0.050			1.7	
124. Heptachlor	0.050			1.7	
125. Aldrin	0.050			1.7	
126. Heptachlor epoxide	0.050			1.7	
127. Endosulfan I	0.050			1.7	
128. Dieldrin	0.10			3.3	
129. 4,4'-DDE	0.10			3.3	
130. Endrin	0.10			3.3	
131. Endosulfan II	0.10			3.3	
132. 4-4'-DDD	0.10			3.3	
133. Endosulfan sulfate	0.10			3.3	
134. 4-4'-DDT	0.10			3.3	
135. Methoxychlor	0.50			17	
136. Endrin ketone	0.10			3.3	
137. Endrin aldehyde	0.10			3.3	
138. alpha-Chlordane	0.050			1.7	
139. gamma-Chlordane	0.050			1.7	
140. Toxaphene	5.0			170	
AROCLORS	**Water (µg/L)**			**Soil (µg/kg)**	
141. Aroclor-1016	1.0			33	
142. Aroclor-1221	1.0			33	
143. Aroclor-1232	1.0			33	
144. Aroclor-1242	1.0			33	
145. Aroclor-1248	1.0			33	
146. Aroclor-1254	1.0			33	
147. Aroclor-1260	1.0			33	
148. Aroclor-1262	1.0			33	
149. Aroclor-1268	1.0			33	

* For volatiles, quantitation limits for medium soils are approximately 50 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 30 times the quantitation limits for low soils.

The TCL for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since the inception of the CLP, compounds have been added to and removed from the TCL, based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program. The SOM analytical service combines the previous OLM and OLC services into one method. For example, drinking water and ground water type samples may be analyzed using the Trace Volatiles method in SOM.

METHODS AND INSTRUMENTATION

For trace volatile water samples, 25 mL of water sample is added to a purge-and-trap device and purged with an inert gas at room temperature. For low/medium volatile water samples, 5 mL of water sample is added to a purge-and-trap device and purged with an inert gas at room temperature. Higher purge temperatures may be used for both trace and low/medium volatile analyses if all technical acceptance criteria is met for all standards, samples, and blanks. For low-level volatile soil samples, organic compounds are generally determined by analyzing approximately 5 g of sample in a closed-system purge-and-trap device at 40°C. For a medium-level soil sample, a soil sample of 5 g is collected, preserved, and/or extracted with methanol and an aliquot of methanol extract is added to 5 mL reagent water and purged at room temperature. For water and soil samples, the volatiles purged from the sample are trapped on a solid sorbent. The purged volatiles are subsequently desorbed by rapidly heating and backflushing with helium, and then introduced into a GC/MS system.

For semivolatile, pesticide, and Aroclor water samples, a 1 L aliquot of sample is extracted with methylene chloride using a continuous liquid-liquid extractor or separatory funnel (for pesticides and Aroclors only). For low-level semivolatile, pesticide, and Aroclor soil samples, a 30 g soil/sediment sample is extracted with methylene chloride/acetone using sonication, automated Soxhlet/Dean-Stark (SDS) extraction, or pressurized fluid extraction techniques. For medium-level semivolatile soil samples, a 1g aliquot is extracted with methylene chloride using the techniques mentioned above for low-level soil samples. For both water and soil samples, the extract is concentrated, subjected to fraction-specific cleanup procedures, and analyzed by GC/MS for semivolatiles or GC/ECD for pesticides and Aroclors. **Table 2** summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include hardcopy data reporting forms and supporting raw data. In addition to the hardcopy deliverable, contract laboratories must also submit the same data electronically. The laboratory must submit data to EPA within 7, 14, or 21-days after laboratory receipt of the last sample in set [or preliminary data within 48 hours (for trace volatiles and volatiles) or 72 hours (for semivolatiles, pesticides, and Aroclors)] after laboratory receipt of each sample. EPA then processes the data through an automated Data Assessment Tool (DAT). DAT provides EPA Regions with PC-compatible reports, spreadsheets, and electronic files within 24-48 hours from the receipt of the data for use in data validation. This automated tool also facilitates the transfer of analytical data into Regional databases. In addition to the Regional electronic reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has 6 business days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

QUALITY ASSURANCE (QA)

The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of known and documented quality.

During the implementation of the data collection effort, QA activities ensure that the Quality Control (QC) system is functioning effectively and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions.

Each contract laboratory prepares a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, and functional guidelines, as well as the QA and QC activities designed to achieve the data quality requirements in the contract.

QUALITY CONTROL (QC)

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analytical service are provided in **Table 3.**

Table 2. Methods and Instruments

Fraction	Water	Soil
Trace Volatiles	Purge-and-trap followed by GC/MS analysis	N/A
Volatiles	Purge-and-trap followed by GC/MS analysis	Purge-and-trap or closed-system purge-and-trap followed by GC/MS analysis
Semivolatiles	Continuous liquid-liquid extraction (CLLE) followed by GC/MS analysis	Sonication, automated SDS extraction, or pressurized fluid extraction followed by GC/MS analysis
Pesticides	CLLE or separatory funnel extraction followed by dual column GC/ECD analysis	Sonication, automated SDS extraction or pressurized fluid extraction followed by dual column GC/ECD analysis
Aroclors	CLLE or separatory funnel extraction followed by dual column GC/ECD analysis	Sonication, automated SDS extraction or pressurized fluid extraction followed by dual column GC/ECD analysis

Table 3. Quality Control (QC)

QC Operation	Frequency
Deuterated Monitoring Compounds (DMCs) (trace volatiles, volatiles, and semivolatiles)	Added to each sample, standard, and blank
Surrogates (pesticides and Aroclors)	Added to each sample, standard, and blank
Method Blanks (trace volatiles and volatiles)	Analyzed at least every 12 hours for each matrix and level
Method Blanks (semivolatiles, pesticides, and Aroclors)	Prepared with each group of 20 samples or less of same matrix and level, or each time samples are extracted by the same procedure
Instrument Blank (trace volatiles and volatiles)	Analyzed after a sample which contains compounds at concentrations greater than the calibration range
Instrument Blank (pesticides and Aroclors)	Every 12 hours on each GC column used for analysis
Storage Blanks (trace volatiles and volatiles)	Prepared and stored with each set of samples
GC/MS Mass Calibration and Ion Abundance Patterns (trace volatiles, volatiles, and semivolatiles)	Every 12 hours for each instrument used for analysis
GC Resolution Check (pesticides)	Prior to initial calibration, on each instrument used for analysis
Initial Calibration	Upon initial set up of each instrument, and each time continuing calibration fails to meet the acceptance criteria
Continuing Calibration	Every 12 hours for each instrument used for analysis
Internal Standards (trace volatiles, volatiles, and semivolatiles)	Added to each sample, standard, and blank
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	Once every 20 or fewer samples of same fraction, matrix, and level in a Sample Delivery Group (SDG)
Laboratory Control Samples (LCSs) (pesticides and Aroclors)	Once every 20 or fewer samples of same fraction, matrix, and level in an SDG
Method Detection Limit (MDL)	Determined annually, per matrix and level

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by ASB and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, GC/MS and/or GC/ECD tape audits, and evaluates laboratory performance through the use of blind Performance Evaluation (PE) samples.

CONTACTING EPA

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Modifications Updating ISM01.2 to ISM 1.3

Exhibit Section(s)	Revisions
Exhibit A, Section 4.2.1.2.3.4	The recording of cooler temperature has been revised as follows: "The Contractor shall record the temperature of the cooler on Form DC-1, under Item 9 - Cooler Temperature. BEGIN INSERTION If ice is present, that will be noted in the remarks column. END INSERTION (see Exhibit B)."
Exhibit Section(s)	Revisions
Exhibit B, Section 2.5.2.3.4, Table 2	The Codes for Labeling Data have been revised as follows: For Continuing Calibration Verification the laboratory shall use CCV##. For Continuing Calibration Blank the laboratory shall use CCB##. For Laboratory Control Sample the laboratory shall use LCS##. For Preparation Blank (Aqueous/Water) the laboratory shall use PBW##. For Preparation Blank (Soil/Sediment) the laboratory shall use PBS##. For Preparation Blank (Wipe/Filter) the laboratory shall use PBF##. Insert Footnote 3 "Within a fraction, the two-character suffix (##) should be unique for each instance of each sample type within an SDG. The Contractor may achieve this by replacing the suffix with one or two alpha-numeric characters".
Exhibit B, Section 2.12	The delivery of hardcopy data in PDF format has been revised as follows: In addition to all required deliverables identified in the Laboratory's contract and the ISM01.2 SOW, the laboratory shall provide a complete copy of the hardcopy deliverable in PDF via EXES. The Laboratory may upload CSF in PDF via EXES at: http://epasmoweb.fedcsc.com/scstr/ and follow the naming convention for the PDF file. The format of the PDF file name should be Case Number_SDG Number_Contract Number_Submission type. For Example: 40000_MAB123_EPW05000_FS. The following identifiers are utilized for submission type: First Submission = FS

	Replacement Submission = RS (if you have to send in a complete replacement of the first submission PDF) Reconciliation Submission = R# (# representing the recon submission number – for example the first recon will be R1) Additional Data Submission = A# (# representing the additional data submission number - for example the first additional data will be A1)

Exhibit Section(s)	Revisions
Exhibit B, Section 3.3.9.1	The language has been revised as follows: FROM: "NOTE: All results shall be transcribed to Inorganic Forms IIA-IN through XV-IN from..." TO: NOTE: All results shall be transcribed to Inorganic Forms IIA-IN through XVI-IN from...
Exhibit Section(s)	**Revisions**
Exhibit D – Part A, Analytical Methods for ICP – Atomic Emission Spectroscopy, Section 10.1.5.1.5	The sample preparation instructions have been revised as follows: From: "Filter the sample digestate... make to volume with reagent water, stopper, and mix." (BEGIN INSERTION) NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. (END INSERTION) "The sample is now ready for analysis."

Exhibit Section(s)	Revisions
Exhibit D – Part B, Analytical Methods for Inductively Coupled Plasma – Mass Spectrometry, Table 3	The Recommended Isotopes and Masses for Selected Elements have been revised as follows: For Element of Interest: Calcium Under "Analyte Masses – Choose One, or More – Calibrated" (BEGIN INSERTION) 40 (END INSERTION)
Exhibit Section(s)	Revisions
Exhibit D – Part B, Analytical Methods for Inductively Coupled Plasma – Mass Spectrometry, Table 6	The spiking Levels for Spike Sample Analysis have been revised as follows: The spike level for selenium is 100 ug/L and 10 mg/Kg for aqueous and soils respectively