USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

INORGANIC ANALYSIS

Multi-Media, Multi-Concentration

ILM05.2
December 2001

Method 00060

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STATEMENT OF WORK

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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 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 water/aqueous and/or soil/sediment samples. 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 and two appendices. 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 reporting data in computer-readable format appear in Exhibit H. Appendix A provides examples of the data format requirements specified in Exhibit H. Appendix B contains a description of the requirements for performing

modified analyses, as well as the analytical procedure for Graphite Furnace Atomic Absorption (GFAA).

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. 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. The Contractor shall communicate with SMO personnel by telephone 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 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) 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 and the USEPA Regional CLP Project Officer (CLP PO) regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO personnel and the USEPA Regional CLP PO 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 blank with each cooler shipped. The temperature blank will be clearly labeled: USEPA COOLER

TEMPERATURE INDICATOR. 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.

4.2.1.2.3.3 To determine the temperature of the cooler: the Contractor shall locate the cooler temperature indicator bottle in the sample shipping cooler, remove the cap, and insert a calibrated thermometer into the cooler temperature indicator bottle. 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.

4.2.1.2.3.4 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 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.

4.2.1.2.3.5 The Contractor shall record the temperature of the cooler on Form DC-1, under Item 9 - Cooler Temperature, and in the SDG Narrative (see Exhibit B).

4.2.1.2.4 The Contractor is required to retain unused sample volume, used sample containers, and empty sample bottle containers for a period of 60 days after data submission. From time of receipt until analysis, the Contractor shall maintain all water/aqueous (preserved and unpreserved) and/or soil/sediment samples at 4°C (±2°C) (see Exhibit B).

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 Case of field samples received, or
- 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).
- 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.

4.2.1.2.6.2 Samples may be assigned to SDGs by matrix (i.e., all soils in one SDG, all waters 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 six digits in length. If the Contractor receives a sample number of any other length, the Contractor shall contact SMO immediately. 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 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 a 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 a 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, analysis of additional analytes and/or lower quantitation limits. These requests will be made by the USEPA Regional CLP PO, USEPA OERR Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (PM), and Contracting Officer (CO) in writing, prior to sample scheduling. If the Contractor voluntarily elects to perform these modified analyses, these analyses will be performed with no increase in per sample price. All contract requirements specified in the SOW/Specifications will remain in effect unless the USEPA CO provides written approval for the modification(s) and a waiver for associated defects. The USEPA CO approval must be obtained prior to sample scheduling.

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 and the USEPA Regional CLP PO 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 Management 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 all 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 or 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, computer-readable data on diskette (or via an alternate means of electronic transmission approved in advance by USEPA) 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 Sample Data Package(s) provided that the forms are in **exact 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 computer-readable data submitted by the Contractor on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) shall contain identical information. If discrepancies are found during Government inspection, the Contractor shall be required to resubmit either the hardcopy forms or the computer-readable data, or both sets of data, at no additional cost to USEPA.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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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 USEPA Office of Emergency and Remedial Response (OERR) Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (AOC 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	CLP PO[D]	QATS
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]	Sample Data Package	1	XX[C] days after Validated Time of Sample Receipt (VTSR)[1] of last sample in SDG.	X			
C.[2]	Data in Computer-Readable Format	1	XX[C] days after VTSR of last sample in SDG.	X	X		
D.[2]	Results of Intercomparison Study/PE Sample Analysis Study	1	XX[C] days after VTSR of last sample in SDG.	X			X
E.[2,3]	Complete SDG File (CSF)[B]	1	XX[C] days after VTSR of last sample in SDG.		X		
F.[4]	Preliminary Results	1	Within 72 hours after receipt of each sample at laboratory, if requested.	X	X		
G.[5,6]	Quarterly Verification of ICP-AES/ICP-MS Linear Ranges and ICP-AES Interelement Correction Factors	1	Quarterly: 15th day of January, April, July, and October.	X		X	X
	Annual Verification of Method Detection Limits (MDLs)	1	Annually: 15th day of January.	X		X	X

TABLE 1 (Con't)

Item		No. of Copies[A]	Delivery Schedule	Distribution			
				SMO	Region	CLP PO[D]	QATS
H.[6,7]	Standard Operating Procedures (SOPs)	1	Revise within 30 days after contract award and receipt of USEPA comments. Submit within 7 days of receipt of written request to recipients as directed. (See Exhibit E, Section 6) Submit within 14 days of amended SOP(s) as directed in Exhibit E, Section 6.4.	As Directed Amended SOPs distributed to CLP PO and QATS			
I.[6,7]	Quality Assurance Management Plan (QAP)	1	Revise within 30 days after contract award and receipt of USEPA comments. Submit within 7 days of receipt of written request to recipients as directed. (See Exhibit E, Section 5) Submit within 14 days of amended QAP as directed in Exhibit E, Section 5.3.	As Directed Amended QAP distributed to CLP PO and QATS			
J.	Electronic Instrument 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			

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 OERR Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (AOC PM). Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the USEPA AOC PM.

[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.

[D]The CLP PO is the USEPA Regional Contract Laboratory Program (CLP) Project Officer (CLP PO) designated on the contract.

[1]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. Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less with the same laboratory turnaround and not exceeding 20 samples [excluding Performance Evaluation (PE) samples]. Data for all samples in the SDG are due concurrently. 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.

[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 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 Package plus all of the original documents described in Exhibit B, Section 2.6.

[4]If required at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of all Form Is (see Exhibit B, Section 2.9). Facsimile or electronic transmittal is required as requested by the Region. Electronic transmittals shall be transmitted as WordPerfect, MS Word, PDF, or other USEPA-approved formats. The Contractor will be notified of the format, fax numbers, or email address(es) at the time of sample scheduling. Sample Traffic Reports/Chain of Custody Records and SDG Cover Sheets shall be submitted with the Preliminary Results. The Contractor shall document all communication in a telephone log.

Preliminary Results Delivery Schedule:

If a sample requiring Preliminary Results arrives before 5 p.m., the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day.

[5]Also required in each Sample Data Package.

[6]See Exhibit E for description. Time is cited in calendar days.

Footnotes (Con't):

[7]The Contractor shall deliver both hardcopy and electronic (i.e., diskette) copies of the Standard Operating Procedures (SOPs) and Quality Assurance Management 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]
2000 Edmund Halley Drive
Reston, VA 20191-3400

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 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) Laboratory[2]
2700 Chandler Avenue, Building C
Las Vegas, NV 89120
Attn: Data Audit Staff

In addition, the mailing and delivery addresses for the USEPA AOC Inorganic Program Manager (AOC PM) are:

Mailing Address: USEPA OERR Analytical Operations/
Data Quality Center
Ariel Rios Building (5204G)
1200 Pennsylvania Avenue, N.W.
Washington, DC 20460
Attn: CLP Inorganic Program Manager

Fed-Ex/Overnight Delivery: USEPA OERR Analytical Operations/
Data Quality Center
1235 Jefferson Davis Highway
Crystal Gateway I, 12th Floor
Arlington, VA 22202
Attn: CLP Inorganic Program Manager

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[1]The 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.

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 section;
- Paginated sequentially according to instructions in this exhibit; and
- Double-sided.

NOTE: Complete Sample Delivery Group (SDG) Files (CSFs) need not be double-sided. (The CSF is composed of original documents.) However, Sample Data Packages delivered to the USEPA Contract Laboratory Program (CLP) Sample Management Office (SMO) and the Region, [and USEPA designated recipients, e.g., Quality Assurance Technical Support (QATS), upon written request] must be double-sided.

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 shall be specified in all correspondence.

2.1.2 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 field descriptors for reporting data in computer-readable format 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 4 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 the USEPA Regional CLP Project Officer (CLP PO) action, or through a 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 Region) and to USEPA's designated recipient (e.g., QATS) when a written request for the Sample Data Package 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 Contract Compliance Screening (CCS) review by SMO, the data shall be sent to the two contractual data recipients (SMO and Region) and to USEPA's designated recipient (e.g., QATS) when a written request for the Sample Data Package has been made. In all instances, the Contractor shall include a color-coded cover sheet (Laboratory

Response to Results of Contract Compliance Screening) provided by SMO. Electronic deliverables shall be submitted or resubmitted to SMO and the Region. Revised DC-1 and DC-2 forms shall be resubmitted to SMO and the 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 which consists of everything above the Chain of Custody Record section, and the bottom section which is the Chain of Custody Record. 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 receipt of samples.. The laboratory signature box is located at the bottom of the CLP Traffic Report/Chain of Custody Record 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 receipt of all samples identified within the CLP Traffic Report section and sign and date the signature box located in the upper half of the CLP Traffic Report/Chain of Custody Record. If a non-CLP Traffic Report/Chain of Custody Record is submitted with the samples, for example 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;
- Sample analysis price (full sample price from the contract);
- Case number; 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 six digits in length and continuous (without spaces or hyphens). If the Contractor receives sample numbers of any other length, the Contractor shall contact SMO immediately. 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 Package

The Sample Data Package 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 Package 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 Package shall include the following:

2.5.1 Cover Documentation

2.5.1.1 Cover Page for the inorganic analyses Data Package shall include: laboratory name; laboratory code; contract number; Case number; SDG number; Non-Routine Analytical Service (NRAS) number (if appropriate); EPA sample numbers in alphanumeric order showing EPA sample numbers cross-referenced with laboratory Sample ID numbers; and completion of the questions on 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 Sample 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 Sample Data Package and in the computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) 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, contract number, and detailed documentation of any Quality Control (QC), sample, shipment, and/or analytical problems encountered in processing the samples reported in the Sample Data Package. The Contractor shall include any technical and administrative problems encountered and the resolution or corrective actions taken. This 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 or curves (at least one equation or curve per method), to allow the recalculation of sample results from raw instrument output. The Contractor shall also include a discussion of any flexibility Statement of Work (SOW) modification. 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]

2.5.2 Sample 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, quarterly and annual verification of method and instrument parameters forms, raw data, and copies of the digestion and distillation logs.

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 micrograms per Liter (UG/L) for water samples and milligrams per kilogram (MG/KG) for solid samples. (No other units are acceptable.) Results for solid 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 one decimal place. The preceding discussion concerning significant numbers applies to Forms IA-IN, IB-IN, and IX-IN only. For other forms, follow the

instructions specific to those forms as discussed in this exhibit.

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.

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]

2.5.2.2.1.2 CRQL Check Standard [Form IIB-IN]

2.5.2.2.1.3 Blanks [Form III-IN]

2.5.2.2.1.4 ICP-AES Interference Check Sample [Form IVA-IN]

2.5.2.2.1.5 ICP-MS Interference Check Sample [Form IVB-IN]

2.5.2.2.1.6 Matrix Spike Sample Recovery [Form VA-IN]

2.5.2.2.1.7 Post-Digestion Spike Sample Recovery [Form VB-IN]

2.5.2.2.1.8 Duplicates [Form VI-IN]

2.5.2.2.1.9 Laboratory Control Sample [Form VII-IN]

2.5.2.2.1.10 ICP-AES and ICP-MS Serial Dilutions [Form VIII-IN]

2.5.2.2.1.11 Method Detection Limits (Annually) [Form IX-IN]

2.5.2.2.1.12 ICP-AES Interelement Correction Factors (Quarterly) [Form XA-IN]

2.5.2.2.1.13 ICP-AES Interelement Correction Factors (Quarterly) [Form XB-IN]

2.5.2.2.1.14 ICP-AES and ICP-MS Linear Ranges (Quarterly) [Form XI-IN]

2.5.2.2.1.15 Preparation Log [Form XII-IN]

2.5.2.2.1.16 Analysis Run Log [Form XIII-IN]

2.5.2.2.1.17 ICP-MS Tune [Form XIV-IN]

2.5.2.2.1.18 ICP-MS Internal Standards Relative Intensity Summary [Form XV-IN]

2.5.2.3 Raw Data

For each reported value, the Contractor shall include in the Sample Data Package all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the quarterly and annual verification of method and instrument parameters submitted as a part of each Sample Data Package. When 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 shall include, for all samples, 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, Section 7.2.4.4.1, 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., Batch Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the Method Detection Limit (MDL). Raw data shall not be corrected for dilutions or volume adjustments. All Atomic Absorption (AA), Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES), and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) instruments shall provide a legible hardcopy of the direct real-time instrument readout (i.e., strip charts, printer tapes, etc.) or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included. A hardcopy of the instrument's direct readout shall be included for cyanide if the instrumentation has the capability.

2.5.2.3.2 The order of raw data in the Sample Data Package for inorganic analyses shall be: ICP-AES, Graphite Furnace Atomic Absorption (GFAA), 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 The ICP-MS raw data shall also contain the turbidity measurements results [in Nephelolometric Turbidity Units (NTU)] for the field samples.

2.5.2.3.4 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.

2.5.2.3.5 Raw data shall be labeled with EPA sample numbers and appropriate codes, shown in Exhibit B, Table 2 - Codes for Labeling Data, following, 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 (PBs);
- Initial and Continuing Calibration Verification (ICV/CCV) standards, Interference Check Samples (ICSs), serial dilution samples, Contract Required Quantitation Limit (CRQL) Check Standard (CRI), 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 (source, concentration, 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. If the instrument does not automatically provide times of analysis, these shall be manually entered on all raw data (e.g., ICV/CCV, blanks, and the CRQL Check Standard).

Table 2
Codes for Labeling Data [1,2,3]

Sample	XXXXXX
Sample Not Part of the SDG	ZZZZZZ
Duplicate	XXXXXXD
Matrix Spike	XXXXXXS
Serial Dilution	XXXXXXL
Analytical Spike/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##
CRQL Check Standard	CRI##
Laboratory Control Samples:	
Aqueous (Water)	LCSW##
Solid (Soil/Sediment)	LCSS##
Preparation Blank (Water)	PBW##
Preparation Blank (Soil)	PBS##
Linear Range Analysis Standard	LRS##
Baseline Correction	BASELINE##
Reslope	RESLOPE##
Cyanide Mid-Range Standard	MIDRANGE##
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".

[3]The EPA sample number shall be unique for each ICV, ICB, CCV, CCB, ICSA, ICSAB, CRI, LCSW, LCSS, PBW, PBS, LRS, BASELINE, RESLOPE, MIDRANGE, and TUNE within an analysis or preparation method, within an SDG. The Contractor shall achieve this by replacing the two-character terminator (##) of the identifier with one or two characters, numbers, or a combination of both.

2.5.2.4 Digestion and Distillation Logs. The following logs shall be submitted as appropriate for each preparation procedure: digestion logs for ICP-AES, ICP-MS, mercury preparations, and 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, PB) correspond to each batch digested; (4) comments describing any significant sample changes or reactions which occur during preparation shall be entered in the log and noted in the SDG Narrative; (5) indication of pH less than 2 or greater than 12, as applicable; and (6) identification of the sample preparer(s) [signature(s)].

2.5.3 A copy of the Sample Traffic Reports/Chain of Custody Records submitted in Exhibit B, Section 2.4, for all of the samples in the SDG. The Traffic Reports/Chain of Custody Records shall be arranged in increasing EPA sample number order, considering both alpha and numeric designations. A legible photocopy of the SDG Cover Sheet shall also be submitted.

2.6 Complete SDG File (CSF)

As specified in the Delivery Schedule, one CSF (including the original Sample Data Package) shall be delivered to the Region concurrently with the delivery of a copy of the Sample Data Package to SMO. Delivery to USEPA's designated recipient (e.g., QATS) is only required upon written request.

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 Package.

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 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 Package

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); and
- Sample Tags (if present) sealed in plastic bags.

2.6.2.4 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.

2.6.2.5 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; and
- Internal sample and sample digestate and distillate transfer Chain of Custody Records.

2.6.2.6 All other original SDG-specific documents in the possession of the laboratory, including, but not limited to, the following documents:

- Telephone contact 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 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 Region only.

2.6.4 The Contractor shall retain a legible electronic (PDF) or hard copy of the CSF for 365 days after submission of the reconciled data package. After this time, the Contractor may dispose of the package.

2.7 Data in Computer-Readable Format

The Contractor shall provide a computer-readable copy for all samples in the SDG, as specified in Exhibit H, and delivered as specified in Exhibit B, Section 1.1. Computer-readable data deliverables shall be submitted on DOS formatted 3.5-inch high density 1.44 MB diskette(s) (or

via an alternate means of electronic transmission, if approved in advance by USEPA).

2.7.1 When submitted, diskette(s) shall be packaged and shipped in such a manner that the diskette(s) cannot be bent or folded and will not be exposed to extreme heat/cold or any type of electromagnetic radiation. The diskette(s) shall be included in the same shipment as the hardcopy data, and, at a minimum, be enclosed in a diskette mailer.

2.7.2 The data shall be recorded in the file format and adhere to the file, record, and field specifications listed in Exhibit H, "Data Dictionary and Format for Data Deliverables in Computer-Readable Format".

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 (e.g., 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 scanning 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 Quarterly Verification of Linear Ranges and Interelement Correction Factors and Annual Verification of MDLs

The Contractor shall perform and report quarterly verification of instrument linear range and annual verification of MDLs by the methods specified in Exhibit D for each instrument used under this contract. The Contractor shall also perform and report quarterly ICP-AES interelement correction factors (including method of determination), wavelengths used, and integration times. Forms reporting results for quarterly and annual verification of method and instrument parameters for the current quarter and year shall be submitted in each Sample Data Package, using Inorganic Forms IX, XA, XB, and XI. Submission of the quarterly and annual verification of method and instrument parameters shall include the raw data used to determine the values reported.

2.11 Electronic Instrument Data

The Contractor shall adhere to the requirements in Exhibit E.

2.12 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. Each form submitted shall be filled out completely for all analytes before proceeding to the next form of the same type. Do not submit multiple forms if the information on those forms can be submitted on one form.

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 computer-readable data formats specified in Exhibit H, "Data Dictionary and Format for Data Deliverables in Computer-Readable Format". The specific length of each variable for computer-readable data transmission purposes is given 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".

NOTE: On the hardcopy forms, the space provided for entries is greater in some instances than the length prescribed for the variable as written to the electronic deliverable (see Exhibit H). Greater space is provided on the hardcopy forms for the sake of visual clarity.

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 be identical to 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 OERR Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (AOC 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 (i.e., "Matrix (soil/water):" shall appear, not just "Matrix").

3.2.3 Alphabetic entries made onto the forms by the Contractor shall be in ALL UPPERCASE letters (i.e., "LOW", not "Low" or "low"). If an entry does not fill the entire blank space provided on the form, null characters shall be used to remove the remaining underscores that comprise the blank line (see Exhibit H for additional instructions). However, do **not** remove the underscores or vertical bar characters that delineate "boxes" on the forms.

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, Non-Routine Analytical Services (NRAS) number, and Sample Delivery Group (SDG) number. Except as noted for NRAS number, 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. It may not exceed 25 characters.

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 of up to six characters, assigned by USEPA, 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 shall not 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 SMO-assigned Case number (to five characters) associated with the sample, and reported on the Traffic Report/Chain of Custody Record.

3.3.5 NRAS Number. The "NRAS No." is the USEPA assigned number for analyses performed under Non-Routine Analytical Services (NRAS). If samples are to be analyzed under NRAS only, and reported on these forms, then enter the NRAS number and leave the Case number blank. If samples are analyzed according to the Routine Analytical Services (RAS) protocol and have additional NRAS requirements, list both the Case number and NRAS number on all forms. If the analyses have no NRAS requirements, leave the "NRAS 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 are an insufficient number of samples for assignment of SDG numbers, the contractor shall contact SMO for the assignment of a 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 triple-spaced box in the upper right-hand corner of a form, it shall be centered on the middle line of the three lines that form the box.

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 Other Common Fields. Other pieces of information are common to many of the data reporting forms. These include Matrix and Level.

- For "Matrix", enter "SOIL" for soil/sediment samples and "WATER" for water samples.

  NOTE: The matrix must be spelled out. Abbreviations such as "S" or "W" shall **not** be used.

- For "Level", enter the determination of concentration level. Enter as "LOW" or "MED", **not** "L" or "M".

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, round up; otherwise round down. 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 raw data to the specified number of decimal places that are 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. If there are not enough figures in the raw data result to enter in the specified space for that result, then zeros shall be used for decimal places to the specified number of reporting decimals for that result for a specific form. The following examples are provided:

Raw Data Result	Specified Format	Correct Entry on Form
95.99653	5.4 (to four decimal places)	95.9965
95.99653	5.3 (to three decimal places)	95.997
95.99653	5.2 (to two decimal places)	96.00
95.996	5.4 (to four decimal places)	95.9960
95.9	5.4 (to four decimal places)	95.9000

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 "ILM05.2" for the SOW Number.

3.4.1.2.2 Enter an EPA sample number including spikes and duplicates (to seven spaces) 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 MAB123 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 - MAB124, MAB125, MAC111, MA1111, MA1111D, etc.

3.4.1.2.3 A maximum of 20 field sample numbers (excluding PE samples) 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 (to ten spaces) 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 Spectrometer (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 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 (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 percent solids to one decimal place (i.e., 5.3%). If the percent solids is not required because the sample is fully aqueous, or is less than 1% solid, then enter "0.0".

3.4.2.2.3 Enter the appropriate concentration units (UG/L for water or MG/KG dry weight for soil). 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 is greater than or equal to the Method Detection Limit (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. The concentration 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 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: The water CRQL (in ug/L) and the MDL obtained from direct analysis (Preparation Method "NP1") for a given analysis method and instrument shall be used to qualify the results of samples and instrument QC standards that are not taken through a preparation procedure [e.g., ICP-MS samples with turbidity less than 1 Nephelolometric Turbidity Unit (NTU), ICB, CCB, and CRI for ICP-AES].

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: Manual Cold Vapor Atomic Absorption (AA)
AV: Automated Cold Vapor AA
AS: Semi-Automated Spectrophotometric
C: Manual Spectrophotometric
" ": Where no data have been entered
NR: If the analyte is not required to be analyzed

3.4.2.2.6 A brief physical description of the sample, both before and after digestion, shall be reported in the fields for color (before and after), clarity (before and after), texture, and artifacts. For water samples, report color and clarity. For soil 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, leave this field blank. Note any significant changes that occur during sample preparation (i.e., 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 two times the CRQL (-2xCRQL) in the "Comments" field and in the Sample Delivery Group (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]

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 (12 characters maximum) and the CCV Source (12 characters maximum). Enter sufficient information in the available 12 spaces to identify the manufacturer and the solution 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 micrograms per Liter (ug/L), to one decimal place] 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, to two decimal places), 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.

The values used in EQ. 1 for "True(ICV)" and "Found(ICV)" shall be exactly those reported on this form.

3.4.3.2.5 Under "Continuing Calibration Verification True", enter the value (in ug/L, to one decimal place) 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, to two decimal places) 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 each analyte, and "Found(CCV)" is the found concentration of the analyte in the CCV Solution.

The values used in EQ. 2 for "True(CCV)" and "Found(CCV)" shall be exactly those reported on this form.

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 or "NR", 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 CRQL Check Standard [Form IIB-IN]

3.4.4.1 Purpose. This form is used to report analyte recoveries from analyses of the CRQL Check Standards (CRIs).

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 the CRQL Check Standard Source (12 characters maximum) as explained in Exhibit B, Section 3.4.3.2.1.

3.4.4.2.2 Under "CRQL Check Standard True", enter the value (in ug/L, to one decimal place) of the concentration of each analyte in the CRQL Check Standard that was analyzed for analytical samples associated with the SDG.

3.4.4.2.3 Under "CRQL Check Standard Initial Found", enter the value (in ug/L, to two decimal places) of the concentration of each analyte measured in the CRQL Check Standard analyzed at the beginning of each run. Concentration units are ug/L. If applicable, enter the concentration qualifier "J" or "U" after the concentration (e.g., 1.96J for Lead), as specified in Exhibit B, Section 3.4.2.2.5.1.

3.4.4.2.4 Under "CRQL Check Standard Initial %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 3 CRQL Check Standard Initial Percent Recovery

$$\%R = \frac{\text{CRQL Check Standard Initial Found}}{\text{CRQL Check Standard True}} \times 100$$

3.4.4.2.5 Under "CRQL Check Standard Final Found", enter the value (in ug/L, to two decimal places) of the concentration of each analyte measured in the CRQL Check Standard analyzed at the end of each run. If applicable, enter the concentration qualifier "J" or "U" after the concentration (e.g., 1.96J for Lead), as specified in Exhibit B, Section 3.4.2.2.5.1.

3.4.4.2.6 Under "CRQL Check Standard Final %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 4 CRQL Check Standard Final Percent Recovery

$$\%R = \frac{\text{CRQL Check Standard Final Found}}{\text{CRQL Check Standard True}} \times 100$$

3.4.4.2.7 All percent recovery values reported in EQs. 3 and 4 shall be calculated using the exact true and found values reported on this form.

NOTE: For every initial solution reported there must be a final one. However, the opposite is not true. If a CRQL Check Standard was required to be analyzed in the middle of a run, it shall be reported in the "Final Found" section of this form.

3.4.4.2.8 If more CRI analyses were required or analyses were performed using more than one wavelength per analyte, submit additional Form(s) IIB-IN as appropriate.

3.4.4.2.9 The order of reporting CRIs for each analyte shall follow the chronological order in which the standards were run starting with the first Form IIB-IN and continuing to the following Forms IIB-IN as appropriate. When multiple wavelengths are used for one analyte, all the results of one wavelength shall be reported before proceeding to the next wavelength.

3.4.5 Blanks [Form III-IN]

3.4.5.1 Purpose. This form is used to report analyte concentrations found in the Initial Calibration Blank (ICB), Continuing Calibration Blanks (CCB), and the Preparation Blank (PB).

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.

3.4.5.2.1 Enter "SOIL" or "WATER" as appropriate as the matrix of the PB. No abbreviations or other matrix descriptors may be used.

3.4.5.2.2 According to the matrix specified for the PB, enter the PB concentration units as "UG/L" for water or "MG/KG" for soil.

3.4.5.2.3 Under "Initial Calibration Blank", enter the concentration (in ug/L, to one decimal place) of each analyte in the most recent ICB, as described in Exhibit B, Section 3.4.5.2.8, below.

3.4.5.2.4 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 water but greater than or equal to the

MDL that was obtained from direct analysis (Preparation Method "NP1") using that method and instrument.

For prepared calibration blanks (e.g., mercury), the CRQL for water and the MDL 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 obtained from direct analysis or the preparation method.

3.4.5.2.5 Under "Continuing Calibration Blank 1", enter the concentration (in ug/L, to one decimal place) of each analyte detected in the first required CCB analyzed after the ICB, as described in Exhibit B, Section 3.4.5.2.8, 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.

3.4.5.2.6 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.

3.4.5.2.7 Under "Preparation Blank", enter the concentration in ug/L (to three decimal places) for a water blank, or mg/kg (to three decimal places) for a soil blank, of each analyte in the PB, as described in Exhibit B, Section 3.4.5.2.8, 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.

3.4.5.2.8 For all blanks, enter the concentration (positive or negative) for each analyte, if the absolute value of the concentration is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration is less than the appropriate MDL.

For example, arsenic has a MDL of 3 ug/L for Preparation Method "NP1" [CRQL for arsenic is 15 ug/L (water)]. Therefore, a CCB instrument reading of -4.2485 ug/L will be reported as -4.2J; a CCB instrument reading of -2.4356 ug/L will be reported as 15.0U; a CCB instrument reading of 4.3586 ug/L will be reported as 4.4J; and a CCB instrument reading of 2.1584 ug/L will be reported as 15.0U.

3.4.5.2.9 Under "M", enter the method used, as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.5.2.10 If more than one wavelength/mass is used to analyze an analyte, submit additional Form(s) III-IN as appropriate.

3.4.5.2.11 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.

3.4.6 ICP-AES and ICP-MS Interference Check Sample (ICS) [Forms IVA-IN and IVB-IN]

3.4.6.1 Purpose. These forms are used to report ICS results for each ICP-AES or ICP-MS instrument used in SDG analyses.

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. The instructions for Forms IVA-IN and IVB-IN are identical except where specified.

3.4.6.2.1 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.

3.4.6.2.2 Enter "ICS Source" (12 characters maximum) 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., EPA-LV87).

3.4.6.2.3 Under "True Sol. A", enter the true concentration (in ug/L, to the nearest whole number) of each analyte present in Solution A. Enter "0" for each analyte with no specified true value in Solution A.

3.4.6.2.4 Under "True Sol. AB", enter the true concentration (in ug/L, to the nearest whole number) of each analyte present in Solution AB. Enter "0" for each analyte with no specified true value in Solution AB.

3.4.6.2.5 Under "Initial Found Sol. A" on Form IVA-IN (ICP-AES), and "Found Sol. A" on Form IVB-IN (ICP-MS), enter the concentration (positive, negative, or zero, in ug/L, to the nearest whole number) of each analyte and interferent in the initial analysis of Solution A as required in Exhibit D.

3.4.6.2.6 Under "Initial Found Sol. A %R" on Form IVA-IN (ICP-AES), and "Found Sol. A %R" on Form IVB-IN (ICP-MS), 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. 5 Initial Found Sol. A Percent Recovery

$$\%R = \frac{\text{Initial Found Solution A}}{\text{True Solution A}} \times 100$$

Leave the field blank if "True Solution A" equals zero.

3.4.6.2.7 Under "Initial Found Sol. AB" on Form IVA-IN (ICP-AES), and "Found Sol. AB" on Form IVB-IN (ICP-MS), enter the concentration (positive, negative, or zero, in ug/L, to one decimal place) of each analyte and interferent in the initial analysis of Solution AB as required in Exhibit D.

3.4.6.2.8 Under "Initial Found Sol. AB %R" on Form IVA-IN (ICP-AES), and "Found Sol. AB %R" on Form IVB-IN (ICP-MS), 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. 6 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.

3.4.6.2.9 Under "Final Found Sol. A", enter the concentration (positive, negative, or zero, in ug/L, to the nearest whole number) of each analyte and interferent in the final analysis of Solution A as required in Exhibit D. ICP-MS analysis (Form IVB-IN) does not require a final analysis.

3.4.6.2.10 Under "Final 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. 7 Final Found Sol. A Percent Recovery

$$\%R = \frac{\text{Final Found Solution A}}{\text{True Solution A}} \times 100$$

Leave the field blank if "True Solution A" equals zero.

3.4.6.2.11 Under "Final Found Sol. AB", enter the concentration (positive, negative, or zero, in ug/L, to one decimal place) of each analyte and interferent in the final analysis of Solution AB as required in Exhibit D. ICP-MS analysis (Form IVB-IN) does not require a final analysis.

3.4.6.2.12 For all found values of Solutions A and AB, enter the concentration (positive, negative, or zero) of each analyte and interferent at each wavelength used for analysis by ICP.

3.4.6.2.13 Under "Final 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. 8 Final Found Sol. AB Percent Recovery

$$\%R = \frac{\text{Final Found Solution AB}}{\text{True Solution AB}} \times 100$$

Leave the field empty if "True Solution AB" equals zero.

All percent recovery values reported shall be calculated using the exact true and found values reported on this form.

NOTE: For ICP-AES (Form IVA-IN), for every initial solution reported there must be a final solution reported. However, the opposite is <u>not</u> true. If an ICS was required to be analyzed in

the middle of a run, it shall be reported in the "Final Found" section of this form.

3.4.6.2.14 If more ICS analyses were required, submit additional Form(s) IVA-IN and/or IVB-IN as appropriate.

3.4.6.2.15 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.

3.4.7 Matrix Spike Sample Recovery [Form VA-IN]

3.4.7.1 Purpose. This form is used to report results for the pre-digest spike.

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 Indicate the appropriate matrix, level, and concentration units (ug/L for water and mg/kg dry weight for soil) as explained in Exhibit B, Sections 2.5.2.1.1 and 3.3.8.

3.4.7.2.2 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.

3.4.7.2.3 In the "EPA Sample No." box, enter an EPA sample number (7 places maximum) of the sample from which the spike results on this form were obtained. The number shall be centered in the box.

3.4.7.2.4 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.

3.4.7.2.5 Under "Spiked Sample Result (SSR)", enter the measured value (to four decimal places), in appropriate units, for each relevant analyte in the matrix spike sample. 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.

3.4.7.2.6 Under "Sample Result (SR)", enter the measured value (to four decimal places) for each required analyte in the sample (reported in "EPA Sample No." box) on which the matrix spike was performed. 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.

3.4.7.2.7 Under "Spike Added (SA)", enter the value (to two decimal places) for the concentration of each analyte added to the sample. The same concentration units shall be used for "SSR", "SR", and "SA". If the "Spike Added" concentration is specified in the contract, the value added and reported shall

be the specific concentration in appropriate units, corrected for spiked sample weight and percent solids (soils) or spiked sample volume (waters).

3.4.7.2.8 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. 9 Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Percent recovery shall be reported, whether it is negative, positive or zero.

The values for "SSR", "SR", and "SA" must be exactly those reported on this form. A value of zero shall be used in calculations for "SSR" or "SR" if the analyte value is less than the MDL.

3.4.7.2.9 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.

3.4.7.2.10 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.

3.4.7.2.11 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.

3.4.8 Post-Digestion Spike Sample Recovery [Form VB-IN]

3.4.8.1 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 digested sample.

3.4.8.2 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.

3.4.8.2.1 In the "EPA Sample No." box, enter an EPA sample number (seven characters maximum) of the sample from which the spike results on this form were obtained. The number shall be centered in the box.

3.4.8.2.2 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.

3.4.8.2.3 Under "Spiked Sample Result (SSR)", enter the measured value (in ug/L, to two decimal places) for each analyte in the post-digest spike sample. 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.

3.4.8.2.4 Under "Sample Result (SR)", enter the measured value (in ug/L, to two decimal places) for the concentration of each analyte in

the sample (reported in "EPA Sample No." box) on which the spike was performed. 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.

3.4.8.2.5 Under "Spike Added (SA)", enter the value (in ug/L, to one decimal place) 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.

3.4.8.2.6 Under "%R", enter the value (to the nearest whole number) of the percent recovery for all spiked analytes computed according to EQ. 9 in Exhibit B, Section 3.4.7.2.8. Percent recovery shall be reported, whether it is negative, positive, or zero. The values for "SSR", "SR", and "SA" must be exactly those reported on this form. A value of zero shall be substituted for "SSR" or "SR" if the analyte value is less than the MDL.

3.4.8.2.7 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.

3.4.8.2.8 If different samples were used for spike sample analysis of different analytes, additional Form(s) VB-IN shall be submitted.

3.4.9 Duplicates [Form VI-IN]

3.4.9.1 Purpose. The duplicates form is used to report results of duplicate analyses. Duplicate analyses are required for percent solids values and all analyte results.

3.4.9.2 Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.9.2.1 Indicate the appropriate matrix, level, and concentration units (ug/L for water and mg/kg dry weight for soil) as explained in Exhibit B, Sections 2.5.2.1.1 and 3.3.8.

3.4.9.2.2 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.

3.4.9.2.3 For "% Solids for Duplicate", enter the percent solids (as explained in Exhibit B, Section 3.4.2.2.2) for the duplicate sample of the EPA sample number reported on the form.

3.4.9.2.4 In the "EPA Sample No." box, enter EPA sample number (seven characters maximum) of the sample from which the duplicate sample results on this form were obtained. The number shall be centered in the box.

3.4.9.2.5 Under "Control Limit", enter the CRQL (in appropriate units, ug/L for water or mg/kg dry weight 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.

3.4.9.2.6 Under "Sample (S)", enter the original measured value (to four decimal places) 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 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.

3.4.9.2.7 Under "Duplicate (D)", enter the measured value (to four decimal places) for each analyte in the duplicate sample. Concentration units are those specified on the form. 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.

3.4.9.2.8 For solid 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.

3.4.9.2.9 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. 10 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

The values for "S" and "D" shall be exactly those reported on this form. 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.

3.4.9.2.10 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.

3.4.9.2.11 If both values are below the CRQL, then no control limit is applicable.

3.4.9.2.12 Under "M", enter method used as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.10 Laboratory Control Sample [Form VII-IN]

3.4.10.1 Purpose. This form is used to report results for the solid and aqueous LCSs.

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 For the Solid LCS Source (12 characters maximum), enter the appropriate EPA sample number if EPA provided standard was used. Substitute an appropriate number provided by EPA for LCS solutions prepared in the future. If other sources were used, identify the source. For the aqueous LCS Source, enter the source name (12 characters maximum) as explained in Exhibit B, Section 3.4.3.2.1.

3.4.10.2.2 Under "Aqueous True", enter the value (in ug/L, to one decimal place) of the concentration of each analyte in the Aqueous LCS Standard Source.

3.4.10.2.3 Under "Aqueous Found", enter the measured concentration (in ug/L, to two decimal places) of each analyte found in the Aqueous LCS solution.

3.4.10.2.4 Under "Aqueous %R", enter the value of the percent recovery (to the nearest whole number) computed according to the following equation:

EQ. 11 Aqueous LCS Percent Recovery

$$\%R = \frac{\text{Aqueous LCS Found}}{\text{Aqueous LCS True}} \times 100$$

3.4.10.2.5 Under "Solid True", enter the value (in mg/kg, to one decimal place) of the concentration of each analyte in the solid LCS Source.

3.4.10.2.6 Under "Solid Found", enter the measured value (in mg/kg, to one decimal place) of each analyte found in the solid LCS solution.

3.4.10.2.7 Under "C", enter "J" or "U" or leave empty, to describe the found value of the solid LCS as explained in Exhibit B, Section 3.4.2.2.5.1.

3.4.10.2.8 Under "Limits", enter the lower limit (in mg/kg, to one decimal place) in the left column, and the upper limit (in mg/kg, to one decimal place) in the right column, for each analyte in the solid LCS solution.

3.4.10.2.9 Under "Solid %R", enter the value of the percent recovery (to the nearest whole number) computed according to the following equation:

EQ. 12 Solid LCS Percent Recovery

$$\%R = \frac{\text{Solid LCS Found}}{\text{Solid LCS True}} \times 100$$

3.4.10.2.10 The values for true and found aqueous and solid LCSs used in EQs. 11 and 12 shall be exactly those reported on this form. If the analyte concentration is less than the MDL, a value of zero shall be substituted for the aqueous and solid LCS found.

3.4.10.2.11 Submit additional Form(s) VII-IN as appropriate if more than one aqueous LCS or solid LCS was required.

3.4.11 ICP-AES and ICP-MS Serial Dilutions [Form VIII-IN]

3.4.11.1 Purpose. This form is used to report results for ICP-AES and ICP-MS serial dilutions.

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 In the "EPA Sample No." box, enter EPA sample number (7 places maximum) of the sample for which serial dilution analysis results on this form were obtained. The number shall be centered in the box.

3.4.11.2.2 Under "Initial Sample Result (I)", enter the instrument measured value (in ug/L to two decimal places) for each ICP analyte. This value shall not be corrected for any dilution. 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.

NOTE: The initial sample concentration for an analyte does not have to equal the value for that analyte reported on Form IA-IN for that sample. It is the value of the analyte's instrument measured value (uncorrected for dilution) that is within the linear range of the instrument.

3.4.11.2.3 Under "Serial Dilution Result (S)", enter the instrument measured value corrected for a five-fold dilution (in ug/L to two decimal places) for each ICP analyte in the diluted sample. 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.

NOTE: The "Serial Dilution Result (S)" is obtained by multiplying by five the instrument measured value (in ug/L) of the serially diluted sample. The "C" qualifier for the serial dilution shall be established based on the serial dilution result before correcting it for the five-fold dilution regardless of the value reported on Form VIII-IN.

For example, if the instrument readout value for the "Initial Sample Result (I)" for silver in a two-fold diluted sample MAX123 is 1164.36 ug/L, and the instrument readout value for the "Serial Dilution Result (S)" for silver in a ten-fold diluted sample MAX123 (MAX123L) is 241.67 ug/L, then the concentration reported for silver in the "Initial Sample Result (I)" column will be 1164.36 ug/L (not 2 times the instrument readout value which equals 2328.72 ug/L), and the concentration reported for silver in the "Serial Dilution Result (S)" column will be five times the instrument readout value which equals 1208.35 ug/L (not 10 times the instrument readout value which equals 2416.70 ug/L).

3.4.11.2.4 Under "% Difference", enter the absolute value (to the nearest whole number) of the percent difference in concentration of required analytes, between the original sample and the diluted sample (adjusted for dilution) according to the following formula:

EQ. 13 Serial Dilution Percent Difference

$$\% \text{ Difference} = \frac{|I - S|}{I} \times 100$$

The values for "I" and "S" used to calculate percent difference in EQ. 13 shall be exactly those reported on this form. 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.

3.4.11.2.5 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.

3.4.11.2.6 Under "M", enter the method of analysis for each analyte as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.12 Method Detection Limits (Annually) [Form IX-IN]

3.4.12.1 Purpose. This form documents the Method Detection Limits (MDLs) 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 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 Sample Data Package on Forms IX-IN.

3.4.12.2 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.

3.4.12.2.1 Enter the Analysis Method qualifier as specified in Exhibit B, Section 3.4.2.2.5.3, in the "Instrument Type" field.

3.4.12.2.2 Enter the Instrument ID in the "Instrument ID" field (12 characters maximum). These instrument IDs are used to uniquely identify each instrument that the laboratory used to perform the analysis.

3.4.12.2.3 Enter the date (formatted MM/DD/YYYY) on which the MDL analysis was performed in the "Date" field.

3.4.12.2.4 For "Preparation Method", enter the method of preparation (three characters maximum) for which the MDLs listed on Form IX-IN were established. Use appropriate sample preparation codes as specified below:

HW1: Hotplate/Block digestion for ICP-AES analysis of water samples.
HW2: Hotplate/Block digestion for ICP-MS analysis of water samples.
MW1: Microwave digestion for ICP-AES analysis of water samples.
MW2: Microwave digestion for ICP-AES analysis of water samples.
HS1: Hotplate/Block digestion for ICP-AES analysis of soil samples.
HS2: Hotplate/Block digestion for ICP-AES analysis of soil samples.
MS1: Microwave digestion for ICP-AES analysis of soil samples.
CW1: Preparation for the Manual Cold Vapor AA analysis of water samples.
CS1: Preparation for the Manual Cold Vapor AA analysis of soil samples.
CW2: Preparation for the Automated Cold Vapor AA analysis of water samples.
DW1: Distillation for the manual and semi-automated spectrophotometric analysis of water samples.
DW2: Midi-distillation for the semi-automated spectrophotometric analysis of water samples.
DS1: Distillation for the manual and semi-automated spectrophotometric analysis of soil samples.
DS2: Midi-distillation for the semi-automated spectrophotometric analysis of soil samples.
NP1: No preparation.

3.4.12.2.5 Enter the concentration units (UG/L for water or MG/KG wet weight for soil) for the results reported on Form IX-IN in the "Concentration Units" field. Enter "UG/L" for MDL results obtained from direct analysis (Preparation Method "NP1").

3.4.12.2.6 Under "Wavelength/Mass", enter the wavelength in nanometers (nm) to two decimal places or the mass in atomic mass units (amu) to two decimal places for each analyte for which an MDL has been established and is listed in the MDL column. If more than one wavelength or mass is used for an analyte, use additional Form(s) IX-IN as appropriate to report the MDL.

3.4.12.2.7 Contract Required Quantitation Limits (in ug/L for water and mg/kg for soil) as established in Exhibit C, shall be reported in the column headed "CRQL". The CRQL shall be reported in ug/L on Form(s) IX-IN associated with Preparation Method "NP1".

3.4.12.2.8 Under "MDL", enter the MDL (in ug/L for water and direct analysis, or mg/kg for soil, 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 up 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.

3.4.12.2.9 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 Sample Data Package or precede them by more than twelve months.

3.4.12.2.10 Use the "Comments" section to indicate alternative wavelengths and the conditions under which they are used.

3.4.13 ICP-AES Interelement Correction Factors (Quarterly) [Form XA-IN]

3.4.13.1 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 quarterly, a copy of the results of the quarterly interelement correction factors shall be included with each Sample Data Package on Form XA-IN and Form XB-IN as appropriate.

3.4.13.2 Instructions. Complete the header information according to instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.13.2.1 Enter the ICP-AES Instrument ID (12 characters maximum), which is a unique number designated by the Contractor to identify each ICP-AES instrument used to produce data in the Sample Data Package. If more than one ICP-AES instrument is used, submit additional Form(s) XA-IN as appropriate.

3.4.13.2.2 Report the date (formatted as MM/DD/YYYY) on which these correction factors were determined for use. This date shall not exceed the ICP-AES analysis dates in the Sample Data Package or precede them by more than three calendar months.

3.4.13.2.3 Under "Wavelength", list the wavelength in nm (to two decimal places) 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.

3.4.13.2.4 Under "Al", "Ca", "Fe", and "Mg", enter the correction factor (negative, positive or zero, to seven decimal places, 10 characters maximum) 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.

3.4.13.2.5 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).

3.4.14 ICP-AES Interelement Correction Factors (Quarterly) [Form XB-IN]

3.4.14.1 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.

3.4.14.2 Instructions. Complete this form following the instructions for Form XA-IN (see Exhibit B, Section 3.4.13) by listing the chemical symbol for additional analytes in the heading of the empty columns in the two-space fields provided.

3.4.14.2.1 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.

3.4.15 ICP-AES and ICP-MS Linear Ranges (Quarterly) [Form XI-IN]

3.4.15.1 Purpose. This form documents the quarterly linear range analysis for each ICP instrument that the Contractor used to obtain data for the SDG.

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 Enter the ICP Instrument ID (12 characters maximum), which is a unique number designated by the Contractor to identify each ICP instrument used to produce data for the SDG. If more than one ICP instrument is used, submit additional Form(s) XI-IN as appropriate.

3.4.15.2.2 Report the date (formatted as MM/DD/YYYY) on which these linear ranges were analyzed. This date shall not exceed the dates of analysis by ICP in the Sample Data Package and shall not precede the analysis dates by more than three calendar months.

3.4.15.2.3 Under "Integ. Time (Sec.)", enter the integration time (in seconds to two decimal places) used for each measurement taken from the ICP instrument.

3.4.15.2.4 Under "Concentration", enter the concentration (in ug/L) that is the upper limit of the ICP instrument linear range as determined in Exhibit D. Any measurement above it is out of the linear range, and thus, is an estimated value and shall be diluted into the linear range.

3.4.15.2.5 Under "M", enter the method of analysis for each analyte as explained in Exhibit B, Section 3.4.2.2.5.3.

3.4.15.2.6 If more instruments or analyte wavelengths/masses are used, submit additional Form(s) XI-IN as appropriate.

3.4.16 Preparation Log [Form XII-IN]

3.4.16.1 Purpose. This form is used to report the preparation run log.

3.4.16.1.1 All field samples and all Quality Control (QC) preparations (including duplicates, matrix spikes, LCSs, PBs, and re-preparations) associated with the SDG shall be reported on Form XII-IN.

3.4.16.1.2 Submit one Form XII-IN per batch, per method, if no more than thirty-two 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.

3.4.16.1.3 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.

3.4.16.2 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 "Preparation Method", enter the method of preparation (three characters maximum) for which the preparations listed on Form XII-IN were made, as specified in Exhibit B, Section 3.4.12.2.4.

3.4.16.2.2 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). All EPA sample numbers shall be listed in ascending alphanumeric order, continuing to the next Form XII-IN if applicable.

3.4.16.2.3 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.

3.4.16.2.4 Under "Weight", enter the wet weight (in grams, to two decimal places) of each soil sample prepared for analysis by the method indicated in the header section of the form. If the sample matrix is water, then leave the field empty.

3.4.16.2.5 Under "Volume", enter the final volume (in mL, to the nearest whole number) 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.

3.4.17 Analysis Run Log [Form XIII-IN]

3.4.17.1 Purpose. This form is used to report the sample analysis run log.

3.4.17.1.1 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.

3.4.17.1.2 All field samples and all QC analyses (including tunes, calibration standards, ICVs, CCVs, ICBs, CCBs, CRIs, ICSs, LRSs, LCSs, PBs, 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.

3.4.17.1.3 Submit one Form XIII-IN per run if no more than thirty-two (32) analyses, including instrument calibration, were analyzed in the run. If more than thirty-two analyses were performed in the run, submit additional Form(s) XIII-IN as appropriate.

3.4.17.1.4 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, 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.

3.4.17.2 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 "Instrument ID", enter the Instrument ID (12 characters maximum) 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, and XV.

3.4.17.2.2 For "Analysis Method", enter the method code (two characters maximum) according to the specifications in Exhibit B, Section 3.4.2.2.5.3.

3.4.17.2.3 For "Start Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was started.

3.4.17.2.4 For "End Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was ended.

3.4.17.2.5 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".

3.4.17.2.6 Under "D/F", enter the dilution factor (to two significant figures) 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.

3.4.17.2.7 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.

3.4.17.2.8 For USEPA supplied solutions such as ICVs, ICSs, and LCSs, 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 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. 14 ICV/CCV Correction for Dilution

Found value on Form II = Instrument readout (ug/L) x D/F

3.4.17.2.9 Under "Time", enter the time (in military format - HHMM) at which each analysis was performed.

3.4.17.2.10 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.

3.4.17.2.11 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 Inorganic 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.

3.4.17.2.12 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.

3.4.18 ICP-MS Tune [Form XIV-IN]

3.4.18.1 Purpose. This form is used to report the tuning results for each ICP-MS instrument used in SDG analyses.

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 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 Sample Data Package.

3.4.18.2.3 For "Avg. Measured Mass (amu)", enter the average mass calculated from the five tune analyses (in atomic mass units, to two decimals places) measured for that isotope.

3.4.18.2.4 For "Avg. Peak Width at 5% Peak Height (amu)" enter the average peak width calculated from the analysis (in atomic mass units, to two decimal places) at 5% of the peak height.

3.4.18.2.5 For "%RSD", enter percent Relative Standard Deviation of the absolute signals (intensities) for each isotope calculated from the five tune analyses.

3.4.19 ICP-MS Internal Standards Relative Intensity Summary [Form XV-IN]

3.4.19.1 Purpose. This form is used to report the relative internal standard intensity levels during a run for ICP-MS. 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.

3.4.19.2 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.

3.4.19.2.1 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.

3.4.19.2.2 For "Start Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was started.

3.4.19.2.3 For "End Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was ended.

3.4.19.2.4 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 EPA sample number of "ZZZZZZ." Samples identified as "ZZZZZZ" need not have intensities reported for internal standards.

3.4.19.2.5 Under "Time", enter the time (in military format - HHMM) at which each analysis was performed.

3.4.19.2.6 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.

3.4.19.2.6.1 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 "ZZZZZZ"). The internal standard relative intensity (%RI) is calculated using the following formula:

EQ. 15 Internal Standard Percent Relative Intensity

$$\% RI = \frac{I_n}{I_o} x\ 100$$

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 EPA sample number in the same units.

3.4.19.2.7 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.

3.4.19.2.8 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.

3.4.19.3 All field samples and all QC samples (including calibration standards, ICVs, CCVs, ICBs, CCBs, CRIs, ICSs, LCS, PB, 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.

3.4.19.4 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.

3.5 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 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.

3.5.2 Instructions

3.5.2.1 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).

3.5.2.2 Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in Item 1.

3.5.2.3 Record the custody seal numbers in Item 2.

3.5.2.4 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.

3.5.2.5 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.

3.5.2.6 Record the presence or absence of a cooler temperature indicator bottle in Item 8.

3.5.2.7 Record the cooler temperature in Item 9.

3.5.2.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and mark the appropriate answer in Item 10.

3.5.2.9 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.

3.5.2.10 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.

3.5.2.11 Record the pH for all aqueous samples received.

3.5.2.12 Record the appropriate sample tags and assigned laboratory numbers, if applicable.

3.5.2.13 Any comments should be made in the "Remarks" column.

3.5.2.14 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.

3.5.2.15 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.

3.5.2.16 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. (Do not number Form DC-2). 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 33, 34, 35, or 36. Category 36 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.

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EXHIBIT B

INORGANIC FORMS

USEPA - CLP

COVER PAGE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS 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)	_____	_____

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 computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) 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.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Level: (low/med) _____ Date Received: _____

% Solids: _____

Concentration Units (ug/L 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:

_____

_____

_____

_____

USEPA - CLP

1B-IN
INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Level: (low/med) _____ Date Received: _____

% Solids: _____

Concentration Units (ug/l 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.: _____ NRAS 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 80-120; Other Metals 90-110; Cyanide 85-115

USEPA - CLP

2B-IN
CRQL CHECK STANDARD

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

CRQL Check Standard Source: _____

Concentration Units: ug/L

Analyte	CRQL Check Standard				
	Initial			Final	
	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: 70-130 with the following exceptions:
ICP-AES - Antimony, Lead, and Thallium: 50-150.
ICP-MS - Cobalt, Manganese, and Zinc: 50-150.

* If applicable, enter the concentration qualifier "J" or "U" after the concentration in these columns (e.g., 0.20U for Mercury).

USEPA - CLP

3-IN
BLANKS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Preparation Blank Matrix (soil/water): _____

Preparation Blank Concentration Units (ug/L or mg/kg): _____

Analyte	Initial Calibration Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Preparation Blank	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.: _____ NRAS No.: _____ SDG No.: _____

ICP-AES Instrument ID: _____ ICS Source: _____

Concentration Units: ug/L

Analyte	True		Initial Found				Final Found			
	Sol. A	Sol. AB	Sol. A	%R	Sol. AB	%R	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.: _____ NRAS No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ ICS 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						
Carbon						
Chloride						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Molybdenum						
Nickel						
Phosphorus						
Potassium						
Selenium						
Silver						
Sodium						
Sulfur						
Thallium						
Titanium						
Vanadium						
Zinc						

USEPA - CLP

5A-IN
MATRIX SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

% 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.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

Concentration Units: ug/L

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.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

% Solids for Sample: _____ % Solids for Duplicate: _____

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.: _____ NRAS No.: _____ SDG No.: _____

Solid LCS Source: _____

Aqueous LCS Source: _____

Analyte	Aqueous (ug/L)			Solid (mg/kg)					
	True	Found	%R	True	Found	C	Limits		%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.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

Concentration Units: ug/L

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 LIMITS (ANNUALLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Instrument Type: _____ Instrument ID: _____ Date: _____

Preparation Method: _____

Concentration Units (ug/L or mg/kg): _____

Analyte	Wavelength /Mass	CRQL	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 (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS 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 (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS 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
ICP-AES and ICP-MS LINEAR RANGES (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP Instrument ID: _____ Date: _____

Analyte	Integ. Time (Sec.)	Concentration (ug/L)	M
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

12-IN
PREPARATION LOG

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Preparation Method: _____

EPA Sample No.	Preparation Date	Weight (gram)	Volume (mL)

USEPA - CLP

13-IN
ANALYSIS RUN LOG

Lab Name: ______________________________ Contract: ____________________

Lab Code: _______ Case No.: _______ NRAS 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.: _____ NRAS No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ Date: _____

Element - Mass	Avg. Measured Mass (amu)	Avg. Peak Width at 5% Peak Height (amu)	%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.: _____ NRAS 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

FORM XV-IN ILM05.2

SAMPLE LOG-IN SHEET

Lab Name					Page __ of __
Received By (Print Name)					Log-in Date
Received By (Signature)					
Case Number		Sample Delivery Group No.			NRAS Number

Remarks:		EPA Sample #	Aqueous Sample pH	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken					
2. Custody Seal Nos.	_____ _____					
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*					
4. Airbill	Airbill/Sticker Present/Absent*					
5. Airbill No.	_____ _____					
6. Sample Tags	Present/Absent*					
Sample Tag Numbers	Listed/Not Listed on Traffic Report/Chain of Custody Record					
7. Sample Condition	Intact/Broken*/ Leaking					
8. Cooler Temperature Indicator Bottle	Present/Absent*					
9. Cooler Temperature	_____					
10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree?	Yes/No*					
11. Date Received at Lab	_____					
12. Time Received	_____					
Sample Transfer						
Fraction	Fraction					
Area #	Area #					
By	By					
On	On					

* Contact SMO and attach record of resolution

Reviewed By		Logbook No.
Date		Logbook Page No.

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____

CITY/STATE _____

CASE NO. _____ SDG NO. _____

SDG NOs. TO FOLLOW _____ _____ _____

NRAS 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) (Do not number)	____	____	____	____
2.	Sample Log-In Sheet (DC-1)	____	____	____	____
3.	Traffic Report/Chain of Custody Record	____	____	____	____
4.	Cover Page	____	____	____	____
5.	SDG Narrative	____	____	____	____
	Inorganic Analysis				
6.	Data Sheet (Form I-IN)	____	____	____	____
7.	Initial & Continuing Calibration Verification (Form IIA-IN)	____	____	____	____
8.	CRQL Standard (Form IIB-IN)	____	____	____	____
9.	Blanks (Form III-IN)	____	____	____	____
10.	ICP-AES Interference Check Sample (Form IVA-IN)	____	____	____	____
11.	ICP-MS Interference Check Sample (Form IVB-IN)	____	____	____	____
12.	Matrix Spike Sample Recovery (Form VA-IN)	____	____	____	____
13.	Post-Digestion Spike Sample Recovery (Form VB-IN)	____	____	____	____
14.	Duplicates (Form VI-IN)	____	____	____	____
15.	Laboratory Control Sample (Form VII-IN)	____	____	____	____
16.	ICP-AES and ICP-MS Serial Dilutions (Form VIII-IN)	____	____	____	____
17.	Method Detection Limits (Annually) (Form IX-IN)	____	____	____	____
18.	ICP-AES Interelement Correction Factors (Quarterly) (Form XA-IN)	____	____	____	____
19.	ICP-AES Interelement Correction Factors (Quarterly) (Form XB-IN)	____	____	____	____
20.	ICP-AES and ICP-MS Linear Ranges (Quarterly) (Form XI-IN)	____	____	____	____
21.	Preparation Log (Form XII-IN)	____	____	____	____
22.	Analysis Run Log (Form XIII-IN)	____	____	____	____

	PAGE NOs. FROM	TO	CHECK LAB	REGION
23. ICP-MS Tune (Form XIV-IN)	____	____	____	____
24. ICP-MS Internal Standards Relative Intensity Summary (Form XV-IN)	____	____	____	____
25. ICP-AES Raw Data	____	____	____	____
26. GFAA Raw Data (If Applicable)	____	____	____	____
27. ICP-MS Raw Data	____	____	____	____
28. Mercury Raw Data	____	____	____	____
29. Cyanide Raw Data	____	____	____	____
30. Preparation Logs Raw Data	____	____	____	____
31. Percent Solids Determination Log	____	____	____	____
32. USEPA Shipping/Receiving Documents				
Airbill (No. of Shipments ____)	____	____	____	____
Sample Tags			____	____
Sample Log-In Sheet (Lab)	____	____	____	____
33. Misc. Shipping/Receiving Records (list all individual records)				
Telephone Logs	____	____	____	____
_____	____	____	____	____
_____	____	____	____	____
34. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)				
_____	____	____	____	____
_____	____	____	____	____
35. Internal Original Sample Prep & Analysis Records (describe or list)				
Prep Records _____	____	____	____	____
Analysis Records _____	____	____	____	____
Description _____	____	____	____	____
36. Other Records (describe or list)				
Telephone Communications Log	____	____	____	____
_____	____	____	____	____
_____	____	____	____	____

37. Comments: _____

_____

_____

_____

Completed by:

(CLP Lab) _____ _____ _____

(Signature) (Print Name & Title) (Date)

Audited by:

(USEPA) _____ _____ _____

(Signature) (Print Name & Title) (Date)

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EXHIBIT C

INORGANIC TARGET ANALYTE LIST
WITH CONTRACT REQUIRED
QUANTITATION LIMITS

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EXHIBIT C - INORGANIC TARGET ANALYTE LIST WITH CONTRACT REQUIRED QUANTITATION LIMITS

Table of Contents

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1.0 INORGANIC TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)

Analyte	CAS Number	ICP-AES CRQL for Water[1,2,3,4] (µg/L)	ICP-AES CRQL for Soil[1,2,3,4,5] (mg/kg)	ICP-MS CRQL for Water[1,2,4] (µg/L)
Aluminum	7429-90-5	200	40	30
Antimony	7440-36-0	60	12	2
Arsenic	7440-38-2	15	3	1
Barium	7440-39-3	200	40	10
Beryllium	7440-41-7	5	1	1
Cadmium	7440-43-9	5	1	1
Calcium	7440-70-2	5000	1000	--
Chromium	7440-47-3	10	2	2
Cobalt	7440-48-4	50	10	0.5
Copper	7440-50-8	25	5	2
Iron	7439-89-6	100	20	--
Lead	7439-92-1	10	2	1
Magnesium	7439-95-4	5000	1000	--
Manganese	7439-96-5	15	3	0.5
Mercury	7439-97-6	0.2	0.1	--
Nickel	7440-02-0	40	8	1
Potassium	7440-09-7	5000	1000	--
Selenium	7782-49-2	35	7	5
Silver	7440-22-4	10	2	1
Sodium	7440-23-5	5000	1000	--
Thallium	7440-28-0	25	5	1
Vanadium	7440-62-2	50	10	1
Zinc	7440-66-6	60	12	1
Cyanide	57-12-5	10	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 ILM05.2 Exhibit D may be utilized as long as the documented Method Detection Limits (MDLs) are less than one-half the CRQLs.

[3]Mercury is analyzed by cold vapor atomic absorption. Cyanide is analyzed by colorimetry/spectrophotometry.

[4]Changes to the Inorganic Target Analyte List (TAL) (e.g., adding an additional analyte) or CRQLs may be requested under the flexibility clause in the contract.

[5]The CRQLs for soil are based on 100% solids and on the exact weights and volumes specified in Exhibit D. Samples with less than 100% solids may have CRQLs greater than those listed in the table above.

EXHIBIT D

INTRODUCTION TO ANALYTICAL METHODS

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Exhibit D - Analytical Methods

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1.0 INTRODUCTION

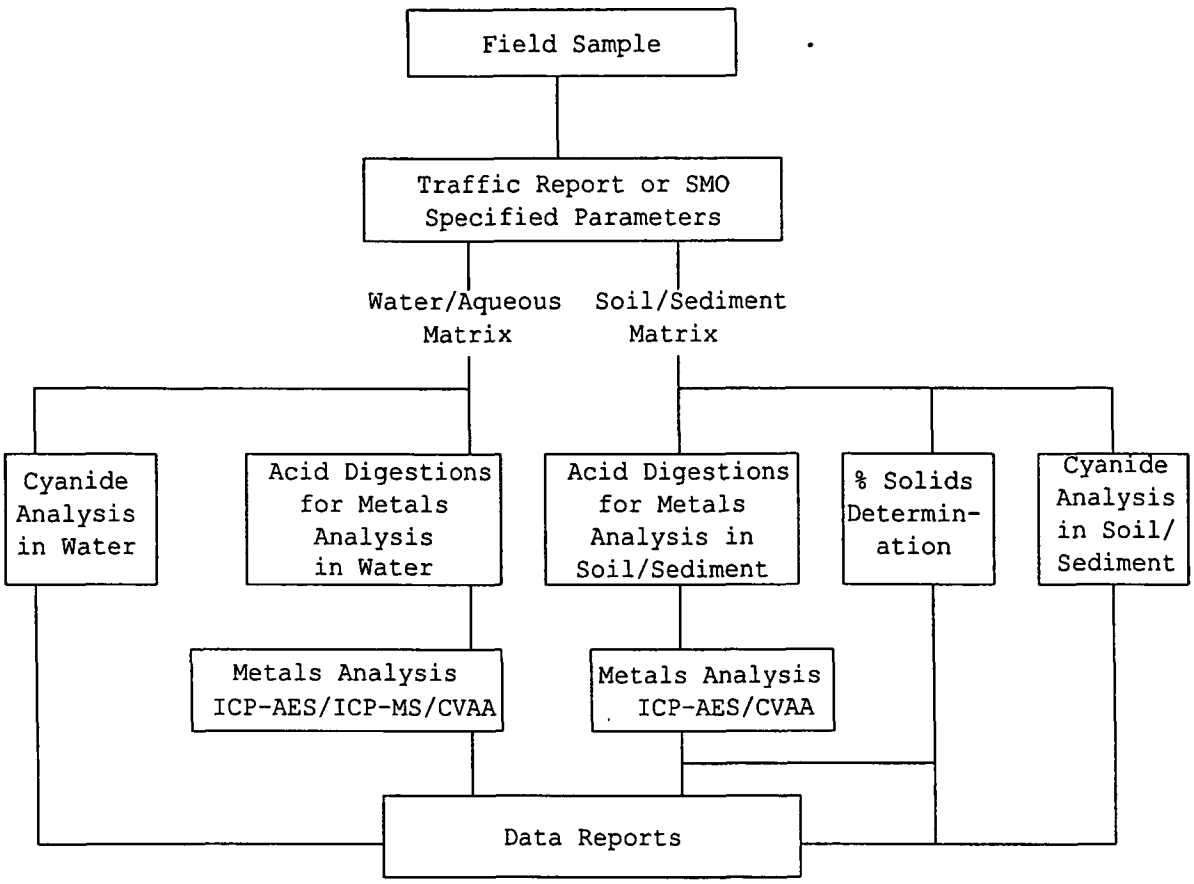
The inorganic analytical service provides a contractual framework for laboratories. This framework applies USEPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 23 metals (including mercury) and cyanide in water/aqueous and/or soil/sediment samples.

The analytical methods that follow are designed to analyze water and sediment/soil 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. Analytical techniques in the inorganic methodologies include Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma - Mass Spectroscopy (ICP-MS), Cold Vapor Atomic Absorption Spectroscopy, and Spectrophotometry. Graphite Furnace Atomic Absorption (GFAA) may be requested by the flexibility clause in the contract.

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 or an equivalent procedure. An electronic version can be found via USEPA's National Environmental Publications Internet Site (NEPIS) at http://www.epa.gov/cincl.

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 Sample Preservation

1.5.1 At the time of sample receipt, the Contractor shall check the pH of the sample and note in a preparation log if the pH is less than 2 for metals. In addition, it should be noted if the pH is greater than 12 for a cyanide sample. Unless instructed by the USEPA Regional CLP Project Officer (CLP PO), the Contractor shall not perform any pH adjustment action if the sample has not been properly preserved. If the sample has not been properly preserved, contact Sample Management

Office (SMO) for further instructions before proceeding with the preparation and analysis. The Contractor may adjust the pH of a sample for metals if SMO provides written documentation to the Contractor from the USEPA Regional CLP PO or USEPA OERR Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (AOC PM) authorizing the adjustment.

1.5.2 Before preparation is initiated for an aqueous 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 (which has been pre-moistened with pH 4 acetate buffer solution). If the test strip turns black, the Contractor shall treat the total volume of sample with powdered cadmium carbonate or lead 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 Sample Delivery Group (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 weighing dish. Weigh and record the weight to the nearest 0.01 g.

1.6.2 Place weighing dish plus sample, with the cover tipped to allow for moisture escape, in a drying oven maintained at 103-105°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 weighing dish cover in place before weighing. Weigh and record weight to nearest 0.01 g. Do not analyze the dried sample.

1.6.4 Duplicate percent solids determinations are required at the same frequency as other analytical determinations. Duplicate results are to be recorded on Form VI-IN.

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[1]Drying time is defined as the elapsed time in the oven; thus raw data must record 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.

1.6.5 For the duplicate percent solids determination, designate one sample aliquot as the "original" sample and the other aliquot as the "duplicate" sample. Calculate dry weight using the results of the "original" sample aliquot.

1.6.6 Calculate percent solids by the formula below. The value thus obtained will be reported on the appropriate Forms I 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

$$\%\ Solids = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

1.6.7 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.

1.7 Insufficient Sample Volume

If insufficient sample volume (less than the required amount) is received to perform the analysis, the Contractor shall contact the SMO to apprise them of the problem. SMO will contract the 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 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.

1.9 Undiluted Analysis

1.9.1 All samples shall be run undiluted for multi-analyte analysis (i.e., the final product of the sample preparation procedure) unless the dilution adjusted detection limits for all analytes are below the CRQL. When an analyte concentration exceeds the calibrated or linear 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 half of the calibrated or linear 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, 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 half of the calibration 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

1.10.1 If dissolved metals are requested by USEPA Regional Offices, the Contractor shall follow the instructions provided on the Traffic Report(s)/Chain of Custody Record(s). If there are no instructions on the Traffic Report/Chain of Custody Record, the Contractor shall digest the samples designated as dissolved metals.

1.10.2 If the Regional Office indicates on the Traffic Report/Chain of Custody Record that a digestion is not to be performed when analyzing field samples for dissolved metals, then a aqueous Laboratory Control Sample (LCSW) and a post-digestion spike sample (hardcopy Form VB-IN and diskette QC codes PDO and PDF) are not required.

1.11 Replicate Exposure

If the Contractor analyzes samples using multiple injections or exposures, the Contractor must use the data obtained from all injections or exposures to calculate the final sample result even if more than the minimum number of injections or 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 Region and notify the Contractor of the Regional decision.

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

1.15.1 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.

1.15.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington D.C., 20036, (202) 872-4477.

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. For further information on waste management consult "The Waste Management Manual for Laboratory Personnel", available from the American Chemical Society at the address listed in Section 1.15.2.

EXHIBIT D - PART A

ANALYTICAL METHODS FOR INDUCTIVELY COUPLED PLASMA - ATOMIC EMISSION SPECTROSCOPY

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Exhibit D - Analytical Methods for ICP-AES

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1.0 SCOPE AND APPLICATION

The following method is an inductively coupled atomic plasma-atomic emission spectroscopy procedure that is used to analyze water, sediment, sludge, and soil samples taken from hazardous waste sites. All metals (except mercury) which are contained in the Inorganic Target Analyte List (TAL) in Exhibit C are quantitated by this Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) method.

2.0 SUMMARY OF METHOD

Water and soil samples are treated with acids and heat or microwave energy 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.

3.0 DEFINITIONS

See Exhibit G 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 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 milligrams per Liter (mg/L) and when total elements are determined after the appropriate digestion procedures are performed. Several types of interferences are summarized below:

4.1 Spectral Interferences

Spectral interferences can be categorized as: overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, 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.

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 Spectrometer (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 analyte 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 milliliter (mL) beaker or other appropriate vessel

6.1.2 Watch glasses

6.1.3 Funnels

6.1.4 Graduated cylinders

6.1.5 Various volumetric flasks (Type A)

6.1.6 Thermometer that covers a range of 0-200°C

6.1.7 Whatman No. 42 filter paper or equivalent

6.1.8 Hot plate, block digester, or other heating source

6.1.9 Equipment and supplies for microwave digestion

6.1.9.1 Whatman No. 41 filter paper (or equivalent)

6.1.9.2 Disposable polypropylene filter funnel

6.1.9.3 Polyethylene bottles, 125 mL, with caps

6.1.9.4 Microwave oven with programmable power settings up to at least 600 watts.

6.1.9.5 The system must use PTFE PFA digestion vessels (120 mL capacity) capable of withstanding pressure of up to 110 (±10) pounds per square inch (psi) [7.5 (±0.7 atm)]. These vessels are capable of controlled pressure relief at pressures exceeding 110 psi.

6.1.9.6 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).

6.1.10 Balances - Analytical Balance, 300 gram (g) capacity, and minimum ±0.01 g.

6.2 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

Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. (Redistilled acids are acceptable.)

7.1.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-77). Use this preparation for all reagents, standards, and dilutions of solutions.

7.1.2 Acetic acid - Concentrated (specific gravity 1.06).

7.1.3 Hydrochloric acid - Concentrated (specific gravity 1.19).

7.1.4 Hydrochloric acid, (1+1) - Add 500 milliliters (mL) conc. HCl (specific gravity 1.19) to 400 mL reagent water and dilute to 1 Liter (L).

7.1.5 Nitric acid - Concentrated (specific gravity 1.41).

7.1.6 Nitric acid, (1+1) - Add 500 mL conc. HNO_3 (specific gravity 1.41) to 400 mL reagent water and dilute to 1 L.

7.1.7 Hydrogen peroxide (30%)

7.1.8 Nitric acid, 5% (v/v) - Add 50 mL conc. HNO_3 to 500 mL reagent water; dilute to 1 L.

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards 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 or prepared from ultra high purity grade chemicals or metals. 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) Typical stock solution preparation procedures follow.

7.2.2.2 Aluminum solution, stock (1 mL = 100 µg Al) - Dissolve 0.100 grams (g) of aluminum metal in an acid mixture of 4 mL of (1+1) HCl and 1 mL of conc. HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask, add an additional 10 mL of (1+1) HCl and dilute to 1000 mL with reagent water.

7.2.2.3 Antimony solution, stock (1 mL = 100 µg Sb) - Dissolve 0.2669 g $K(SbO)C_4H_4O_6$ in reagent water, add 10 mL (1+1) HCl and dilute to 1000 mL with reagent water.

7.2.2.4 Arsenic solution, stock (1 mL = 100 µg As) - Dissolve 0.1320 g of As_2O_3 in 100 mL of reagent water containing 0.4 g NaOH. Acidify the solution with 2 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.5 Barium solution, stock (1 mL = 100 µg Ba) - Dissolve 0.1516 g $BaCl_2$ (dried at 250°C for 2 hours) in 10 mL reagent water with 1 mL (1+1) HCl. Add 10.0 mL (1+1) HCl and dilute to 1000 mL with reagent water.

7.2.2.6 Beryllium solution, stock (1 mL = 100 µg Be) - Do not dry. Dissolve 1.966 grams (g) $BeSO_4 \cdot 4H_2O$, in reagent water, add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.7 Cadmium solution, stock (1 mL = 100 µg Cd) - Dissolve 0.1142 g CdO in a minimum amount of (1+1) HNO_3. Heat to increase rate of dissolution. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.8 Calcium solution, stock (1 mL = 100 µg Ca) - Suspend 0.2498 g $CaCO_3$ dried at 180°C for 1 hour before weighing in reagent water and dissolve cautiously with a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.9 Chromium solution, stock (1 mL = 100 µg Cr) - Dissolve 0.1923 g of CrO_3 in reagent water. When solution is complete acidify with 10 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.10 Cobalt solution, stock (1 mL = 100 µg Co) - Dissolve 0.1000 g of cobalt metal in a minimum amount of (1+1) HNO_3. Add 10.0 mL (1+1) HCl and dilute to 1000 mL with reagent water.

7.2.2.11 Copper solution, stock (1 mL = 100 µg Cu) - Dissolve 0.1252 g CuO in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.12 Iron solution, stock (1 mL = 100 µg Fe) - Dissolve 0.1430 g Fe_2O_3 in a warm mixture of 20 mL (1+1) HCl and 2 mL of conc. HNO_3. Cool, add an additional 5 mL of conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.13 Lead solution, stock (1 mL = 100 µg Pb) - Dissolve 0.1599 g $Pb(NO_3)_2$ in a minimum amount of (1+1) HNO_3. Add 10.0 mL of conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.14 Magnesium solution, stock (1 mL = 100 µg Mg) - Dissolve 0.1658 g MgO in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.15 Manganese solution, stock (1 mL = 100 µg Mn) - Dissolve 0.1000 g of manganese metal in the acid mixture, 10 mL conc. HCl and 1 mL conc. HNO_3, and dilute to 1000 mL with reagent water.

7.2.2.16 Nickel solution, stock (1 mL = 100 µg Ni) - Dissolve 0.1000 g of nickel metal in 10 mL hot conc. HNO_3, cool and dilute to 1000 mL with reagent water.

7.2.2.17 Potassium solution, stock (1 mL = 100 µg K) - Dissolve 0.1907 g KCl, dried at 110°C, in reagent water. Dilute to 1000 mL.

7.2.2.18 Selenium solution, stock (1 mL = 100 µg Se) - Do not dry. Dissolve 0.1727 g H_2SeO_3 (actual assay 94.6%) in reagent water and dilute to 1000 mL.

7.2.2.19 Silver solution, stock (1 mL = 100 µg Ag) - Dissolve 0.1575 g $AgNO_3$ in 100 mL of reagent water and 10 mL conc. HNO_3. Dilute to 1000 mL with reagent water.

7.2.2.20 Sodium solution, stock (1 mL = 100 µg Na) - Dissolve 0.2542 g NaCl in reagent water. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.21 Thallium solution, stock (1 mL = 100 µg Tl) - Dissolve 0.1303 g $TlNO_3$ in reagent water. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.22 Vanadium solution, stock (1 mL = 100 µg V) - Dissolve 0.2297 NH_4VO_3 in a minimum amount of conc. HNO_3. Heat to increase rate of dissolution. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

7.2.2.23 Zinc solution, stock (1 mL = 100 µg Zn) - Dissolve 0.1245 g ZnO in a minimum amount of dilute HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

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 acidified reagent water 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 The calibration blank is prepared by diluting 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to 100 mL with reagent water. Prepare a sufficient quantity to be used to flush the system between standards and samples.

7.2.4.2 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

The concentration of the analytes in the CRI shall be at the respective CRQLs. Information regarding the CRI shall be reported on Form IIB-IN.

7.2.4.3 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.

7.2.4.4 Method Detection Limit (MDL) Solution

The MDL solution shall be at a concentration of 3 to 5 times the expected MDL.

7.2.4.5 Mixed Calibration Standard Solutions

7.2.4.5.1 Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (see Sections 7.2.4.5.2 through 7.2.4.5.7). Add 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl and dilute to 100 mL with reagent water (see Note in Section 7.2.4.5.6). 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 elements are compatible and stable. Transfer the mixed standard solutions to a FEP fluorocarbon or unused polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change with aging. Although not specifically required, some typical calibration standard combinations follow.

7.2.4.5.2 Mixed standard solution I - manganese, beryllium, cadmium, lead, and zinc.

7.2.4.5.3 Mixed standard solution II - barium, copper, iron, vanadium, and cobalt.

7.2.4.5.4 Mixed standard solution III - arsenic and selenium.

7.2.4.5.5 Mixed standard solution IV - calcium, sodium, potassium, aluminum, chromium, and nickel.

7.2.4.5.6 Mixed standard solution V - antimony, magnesium, silver and thallium.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of reagent water and warm the flask until the solution clears. Cool and dilute to 100 mL with reagent water. For this acid combination, the silver concentration should be limited to 2 milligrams per Liter (mg/L). Silver under these conditions is stable in a tap water matrix for 30 days. Higher concentrations of silver require additional HCl.

7.2.4.5.7 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 samples must be collected in glass or polyethylene containers. Water/aqueous samples must be preserved with nitric acid to pH less than 2 immediately after collection. 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 (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 2 immediately after filtration.

8.2 Procedures for Sample Storage

The 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 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 365 days 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, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where correction factors are valid. It is the responsibility of the analyst to verify that 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 Microwave Calibration Procedure

9.2.1 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.

9.2.2 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.

9.2.3 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 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 and 25°C. The beaker is circulated continuously through the field for at least two 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. 1 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.

9.3 Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES) Instrument Calibration Procedure

9.3.1 Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument standardization date and time shall be included in the raw data.

9.3.2 The calibration standards shall be prepared as in Section 7.2.4.5.

9.3.3 Calibrate the ICP-AES instruments according to instrument manufacturer's recommended procedures. At least two standards shall be used for ICP-AES calibration. One of the standards shall be a blank.

9.3.4 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 of the ICP-AES systems have 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.

9.4.2 Only if the ICV solution(s) is(are) not available from USEPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a

concentration other than that used for instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

9.4.3 The ICV solution(s) shall be run at each wavelength used for analysis. The values for the ICV shall be reported on Form IIA-IN.

9.5 Continuing Calibration Verification (CCV)

9.5.1 To ensure calibration accuracy during each analysis run, one of the following standards is to be used for the CCV and shall be analyzed and reported for every wavelength used for the analysis of each analyte, at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The standard shall also be analyzed and reported for every wavelength used for analysis at the beginning of the run and after the last analytical sample. The analyte concentrations in the CCV standard shall be different than the concentration used for the ICV and shall be one of the following solutions at or near one-half of the calibration standard:

- USEPA Solutions
- NIST Standards
- A Contractor-prepared standard solution

The same CCV standard shall be used throughout the analysis runs for a Sample Delivery Group (SDG) of samples received.

9.5.2 Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses, and other related operations that may affect the CCV measured result may not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it, as well as the difference in time between the CCV and the analytical sample immediately preceding it, may not exceed the lowest difference in time between any two consecutive analytical samples associated with the CCV.

9.5.3 Information regarding the CCV shall be reported on Form IIA-IN.

9.6 Initial and Continuing Calibration Blank (ICB/CCB)

A calibration blank shall be analyzed at each wavelength used for analysis immediately after every ICV and CCV, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. The blank shall be analyzed at the beginning of the run and after the last analytical sample.

NOTE: A CCB shall be analyzed immediately after the last CCV, and the last CCV shall be analyzed immediately after the last analytical sample of the run. The results for the calibration blanks shall be reported on Form III-IN.

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 Region for instructions. The 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 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 sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the Region. If all phases of the sample are amenable to analysis, the 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 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 Region's decision in the SDG Narrative.

10.1.3 Water/Aqueous Sample Preparation

10.1.3.1 Preparation Method/Code (HW1) (USEPA Method 200.7, December 1982)

Shake sample and transfer 50-100 milliliter (mL) of well-mixed sample to a 250 mL heating vessel, add 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to the sample. Cover with watch glass or similar cover and heat on a hot plate, block digester, or equivalent heating source which is adjustable and capable of maintaining a temperature of 92-95°C for 2 hours or until sample volume is reduced to between 25 and 50 mL, making certain sample does not boil. Cool sample and filter to remove insoluble material.

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.

Adjust sample volume to 50-100 mL with reagent water. The sample is now ready for analysis. Concentrations so determined shall be

reported as "total". If volumes less than 100 mL are used, all other reagents shall be reduced appropriately (e.g., if 50 mL is used, reduce reagent volumes by one-half). The final volume of the digestate must equal the initial volume of the sample aliquot.

10.1.3.2 Preparation Method/Code (MW1) (USEPA SW-846 Method 3015)

10.1.3.2.1 A 45 mL aliquot of the sample is measured into PTFE digestion vessels.

10.1.3.2.2 5 mL of high purity concentrated HNO_3 is added to the digestion vessels.

10.1.3.2.3 The caps with the pressure release valves are placed on the vessels hand tight and then tightened, using constant torque, to 12 ft/lbs. The weight of each vessel is recorded to 0.02 gram (g).

10.1.3.2.4 Place 5 sample vessels in the carousel, evenly spaced around its periphery in the microwave unit. Venting tubes connect each sample vessel with a collection vessel. Each sample vessel is attached to a clean, double-ported overflow vessel to collect any sample expelled from the sample vessel in the event of over pressurization. Assembly of the vessels into the carousel may be done inside or outside the microwave.

10.1.3.2.5 This procedure is energy balanced for five 45 mL water samples (each with 5 mL of acid) to produce consistent conditions. When fewer than five samples are digested, the remaining vessels must be filled with 45 mL of tap, deionized, or reagent water and 5 mL of concentrated nitric acid.

10.1.3.2.6 Newer microwave ovens may be capable of higher power settings which may allow a larger number of samples. If the analyst wishes to digest more than 5 samples at a time, the analyst may use different power settings as long as they result in the same time temperature conditions defined in the power programming for this method.

10.1.3.2.7 The initial temperature of the samples should be 24°C (±1°C). The Preparation Blank (PB) must have 45 mL of deionized water and the same amount (5 mL) of acid that is added to the samples.

10.1.3.2.8 The microwave unit first-stage program must be set to give 545 watts for 10 minutes and the second-stage program to give 344 watts for 10 minutes. This sequence brings the samples to 160°C (±4°C) in 10 minutes and permits a slow rise to 165-170°C during the second 10 minutes.

10.1.3.2.9 Following the 20 minute program, the samples are left to cool in the microwave unit for 5 minutes, with the exhaust fan on. The samples and/or carousel may then be removed from the microwave unit. Before opening the vessels, let cool until they are no longer hot to the touch.

10.1.3.2.10 After the sample vessel has cooled, weigh the sample vessel and compare to the initial weight as reported on the preparation log. Any sample vessel exhibiting a less than or equal to 0.5 g loss into the overflow vessel must have any excess sample from the associated collection vessel added to the original sample vessel before proceeding with the sample preparation.

Any sample vessel exhibiting a greater than 0.5 g loss must be identified in the preparation log and the sample redigested.

10.1.3.2.11 Sample Filtration - The digested samples are shaken well to mix in any condensate within the digestion vessel before being opened. The digestates are then filtered into 50 mL glass volumetric flasks through Whatman No. 41 (or equivalent) filter paper and diluted to 50 mL (if necessary). The samples are now ready for analysis. The sample results must be corrected by a factor of 1.11 in order to report final concentration values based on an initial volume of 45 mL. Concentrations so determined shall be reported as "total".

10.1.3.3 Preparation Method/Code (MW2) (ASTM Standard D4309-91)

10.1.3.3.1 A 50 mL aliquot of the sample is measured into PTFE digestion vessels.

10.1.3.3.2 3 mL of high purity concentrated HNO_3 and 2 mL of concentrated HCl is added to the digestion vessels.

10.1.3.3.3 Proceed as in Preparation Method/Code "MW1", Sections 10.1.3.2.3 through 10.1.3.2.11.

10.1.3.3.4 Sample Filtration - The digested samples are shaken well to mix in any condensate within the digestion vessel before being opened. If necessary, the digestates are then filtered through filter paper and diluted to 55 mL. The samples are now ready for analysis. The sample results must be corrected by a factor of 1.1 in order to report final concentration values based on an initial volume of 50 mL. Concentrations so determined shall be reported as "total".

10.1.4 Soil/Sediment Sample Preparation

10.1.4.1 Preparation Method/Code (HS1) (USEPA Method 200.7, December 1982)

10.1.4.1.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a beaker.

10.1.4.1.2 Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 92-95°C on hot plate or block digester, and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.

10.1.4.1.3 After the second reflux step has been completed and the sample has cooled, add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the heat source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.

Continue to add 30% H_2O_2 in 1 mL aliquots 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 30% H_2O_2.

10.1.4.1.4 Add 5 mL of 1:1 HCl and 10 mL of reagent water, return the covered heating vessel to the heat source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 mL with reagent water.

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.

Dilute the digestate 1:1 (200 mL final volume) with acidified water [prepared by diluting 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to 100 mL] to maintain constant acid strength. The sample is now ready for analysis.

10.1.4.2 Preparation Method/Code (HS2) (USEPA SW-846 Method 3050B)

10.1.4.2.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 2.0 g portion of sample and transfer to a beaker.

10.1.4.2.2 Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 92-95°C on hot plate, block digester, or equivalent heating source, and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel. Add an additional 5 mL of concentrated HNO_3 and reflux. Repeat this step until sample oxidation is complete (no brown fumes generated).

10.1.4.2.3 After the reflux steps have been completed and the sample has cooled, add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the heat source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.

10.1.4.2.4 Continue to add 30% H_2O_2 in 1 mL aliquots 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 30% H_2O_2.

10.1.4.2.5 Add 10 mL of concentrated HCl and return the covered heating vessel to the heat source and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 mL with reagent water.

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.

The sample is now ready for analysis.

10.1.4.3 Preparation Method/Code (MS1) (USEPA SW-846 Method 3051)

10.1.4.3.1 Add a representative 0.50 g (±0.01 g) of sample to the PTFE PFA vessel.

10.1.4.3.2 Add 10 mL of concentrated nitric acid. If a vigorous reaction occurs, allow the reaction to stop before capping the vessel.

10.1.4.3.3 Cap the vessel, then tighten using constant torque to 12 ft/lbs, according to the manufacturer's direction.

10.1.4.3.4 Connect the sample vessel to the overflow vessel using PTFE PFA tubing.

10.1.4.3.5 Weigh the vessel assembly to the nearest 0.01 g.

10.1.4.3.6 Place sample vessels in groups of 2 sample vessels or 6 sample vessels in the carousel, evenly spaced around its periphery in the microwave unit. If fewer than the recommended number of samples are to be digested (i.e., 3 samples plus 1 blank) then the remaining vessels must be filled with 10 mL of nitric acid to achieve the full complement of vessels.

10.1.4.3.7 Each sample vessel must be attached to a clean, double-ported vessel to collect any sample expelled from the sample vessel in the event of over pressurization. Assembly of the vessels into the carousel may be done inside or outside the microwave. Connect the overflow vessel to the center well of the oven.

10.1.4.3.8 The PB must have 0.5 mL of reagent water and the same amount (10 mL) of acid that is added to the samples. The PB must later be diluted to 50 mL in the same manner as the samples.

10.1.4.3.9 Irradiate the 2 sample vessel group at 344 watts for 10 minutes, or the 6-sample vessel group at 574 watts for 10 minutes.

10.1.4.3.10 This program brings the samples to 175°C in 5.5 minutes; the temperature remains between 170-180°C for the balance of the 10 minute irradiation period. The pressure should peak at less than 6 atmospheres (atm) for most samples. The pressure may exceed these limits in the case of high concentrations of carbonate or organic compounds. In these cases, the pressure will be limited by the relief pressure of the vessel to 7.5 (±0.7 atm).

10.1.4.3.11 Allow the vessels to cool for a minimum of 5 minutes before removing them from the microwave unit, with exhaust fan on. Allow the vessels to cool to room temperature before opening. The vessels must be carefully vented and uncapped in a fume hood.

10.1.4.3.12 Weigh each vessel assembly. If the weight of acid plus the sample has decreased by more than 10% from the original weight, discard the digests. Determine the reason for the loss. Losses typically are attributed to use of digestion time longer than ten minutes, using too large of a sample, or having improper heating conditions. Once the source of the losses has been corrected, prepare a new set of samples for digestion.

10.1.4.3.13 Sample Filtration: Shake the sample well to mix in any condensate within the digestion vessel before being opened.

Filter the digestion vessel into a 50 mL glass volumetric flask through filter paper. Rinse the sample digestion vessel, cap, connecting tube, and (if venting occurred) the overflow vessel into the 50 mL glass flask. Dilute to 50 mL. The samples are now ready for analysis. Concentrations so determined shall be reported as "total".

10.1.5 Non-Prepared Samples

10.1.5.1 Preparation Method/Code (NP1)

10.1.5.1.1 This code shall be used to report samples that are not digested prior to analysis (e.g., dissolved metal samples that the Contractor was instructed not to digest).

10.1.5.1.2 This Preparation Method/Code shall also be used to report the non-prepared Method Detection Limit (MDL). The concentration of this MDL shall be used to determine the appropriate concentration qualifier for the results of non-prepared samples and instrument Quality Control (QC) analyses.

10.2 Microwave Digestion Cleaning Procedure

10.2.1 Initial Cleaning of the PTFE PFA Digestion Vessels

10.2.1.1 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.

10.2.1.2 Rinse in reagent water.

10.2.1.3 Immerse in 1:1 HCl for a minimum of 3 hours after the cleaning bath has reached a temperature just below boiling.

10.2.1.4 Rinse in reagent water.

10.2.1.5 Immerse in 1:1 HNO_3 for a minimum of 3 hours after the cleaning bath has reached a temperature just below boiling.

10.2.1.6 The vessels are then rinsed with copious amounts of reagent water prior to use for any analyses under this contract.

10.2.2 Cleaning Procedure between Sample Digestions

10.2.2.1 Wash entire vessel in hot water using laboratory-grade non-phosphate detergent.

10.2.2.2 Rinse with 1:1 nitric acid.

10.2.2.3 Rinse 3 times with reagent water.

10.3 Sample Analysis

10.3.1 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.

10.3.2 Initiate appropriate operating configuration of computer.

10.3.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using mixed calibration standard solutions such as those described in Section 7.2.4.5.1.

10.3.4 A minimum of two replicate exposures is required for standardization and all QC and sample analyses. The average result of the multiple exposures for the standardization and all QC and sample analyses shall be used.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Water/Aqueous Sample Calculation

The concentrations determined in the digestate are to be reported in units of microgram per Liter (µg/L):

EQ. 2 Aqueous Sample Concentration

$$\text{Concentration } (\mu g/L) = C \times \frac{V_f}{V_i} \times DF$$

WHERE,

- C = Instrument value in µg/L
- V_f = Final digestion volume (mL)
- V_i = Initial digestion volume (mL)
- DF = Dilution Factor

11.2 Soil 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 milligrams per kilogram (mg/kg):

EQ. 3 Soil Sample Concentration

$$\text{Concentration (dry wt.) } (mg/kg) = \frac{C \times V}{W \times S} \times DF$$

WHERE,

- C = Concentration (mg/L)
- V = Final sample volume in Liters (L)
- W = Wet sample weight (kg)
- 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 water/aqueous samples, substitute the value of the MDL (µg/L) or CRQL (µg/L) into the "C" term in Equation 2 above.

Calculate the adjusted MDL or adjusted CRQL for soil samples as follows:

EQ. 4 Adjusted Soil MDL/Adjusted Soil CRQL Concentration

$$\text{Adjusted Concentration (dry wt.) (mg/kg)} = C \times \frac{W_M}{W_R} \times \frac{V_R}{V_M} \times \frac{1}{S} \times DF$$

WHERE,

C	=	MDL or CRQL concentration (mg/kg)
W_M	=	Minimum method required wet sample weight (g)
W_R	=	Reported wet sample weight (g)
V_M	=	Method required final sample volume (mL)
V_R	=	Reported final sample volume (mL)
S	=	% Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).
DF	=	Sample 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% (low) and 110% (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. Information regarding the ICV shall be reported on Form IIA-IN.

12.2 Continuing Calibration Verification (CCV)

The CCV standard shall be prepared by combining compatible elements at a concentration equivalent to the mid-points of their respective calibration curves. If the deviation of the CCV is greater than the control limits specified of 90% (low) and 110% (high), the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and the re-analysis of preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration verification shall be performed for the analytes affected. Information regarding the CCV shall be reported on Form IIA-IN.

12.3 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

12.3.1 To verify linearity near the CRQL, a standard at the CRQL (CRI) shall be prepared, in the same acid matrix as the calibration standards, and analyzed at the beginning and end of each sample analysis run, immediately preceding the Interference Check Sample (ICS) analyses, but not before the ICV. In addition, the Contractor shall analyze the CRI at a frequency of not less than once per 20 analytical samples[1] per analysis run. These analyses of the CRI sample shall be immediately followed by the ICS analyses. [That is, the analytical run sequence shall be CRI, ICS Solution A (ICSA), ICS Solution AB (ICSAB), CCV and Continuing Calibration Blank (CCB), in that order].

12.3.2 The CRI shall be run for every wavelength used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na, and K. Information regarding the CRI shall be reported on Form IIB-IN.

12.3.3 If the percent recovery of the CRI falls outside the control limits of 70-130% (50-150% for antimony, lead, and thallium) for one or more analytes, the CRI shall be re-analyzed immediately for those analytes only. If the results of the re-analysis for those analytes fall within the control limits, no further corrective action is required. If the results of the re-analysis for those analytes do not fall within the control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the CRI analyzed, and the samples associated with the CRI re-analyzed.

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 while the Preparation Blank is used to monitor for possible contamination.

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[1]As defined in Exhibit G, CRI is an analytical sample.

12.4.1 Initial and Continuing Calibration Blank (ICB/CCB)

The ICB and CCB are prepared with acids and reagent water. If the absolute value of the calibration blank (ICB/CCB) result exceeds the CRQL (see Exhibit C), the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank shall be performed for the elements affected.

12.4.2 Preparation Blank (PB)

12.4.2.1 The PB shall contain all the reagents and in the same volumes as used in processing the samples. The PB 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 PB, 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 [2] 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. (see Form III-IN). Each Sample Data Package shall contain the results of all PB analyses associated with the samples in that SDG.

12.4.2.4 The PB is 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 any analyte concentration in the blank is above the CRQL, the lowest concentration of that analyte in the associated samples shall be greater than or equal to 10 times the blank concentration. Otherwise, all samples associated with the blank, with the analyte concentration less than 10 times the blank concentration and above the CRQL, shall be redigested and re-analyzed with appropriate new Quality Control (QC) for that analyte. The only exception to this shall be an identified field blank. 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 re-analyzed with appropriate new QC.

12.4.2.4.4 The values for the PB shall be reported on Form III-IN.

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[2] A group of samples prepared at the same time.

12.5 Interference Check Sample (ICS)

12.5.1 The ICS is prepared by the analyst or obtained from USEPA, if available.

12.5.2 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), at the beginning and end of each analysis run, but not before the ICV. In addition, the Contractor shall analyze and report the results for the ICS at a frequency of not less than once per 20 analytical samples[3] per analysis run. These analyses 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 not dilute the ICS more than is necessary to meet the linear 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, and Solution AB consists of the analytes mixed with the interferents. 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 of ±2 times the CRQL of the analyte's true value or ±20% of the analyte's true value, whichever is greater (the true value shall be zero unless otherwise stated) in the ICSA. For example, if the analysis result(s) for Arsenic (CRQL = 15 µg/L, ICSA true value = 0 µg/L) in the ICSA analysis during the run is 29 µg/L, then the analytical result for Arsenic falls within the ±2 times the CRQL window for Arsenic in the ICSA. If the analytical results of the ICSA do not fall within the control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and re-analysis of the analytical samples analyzed since the last compliant ICSA shall be performed. For analytes with CRQLs less than 5000 µg/L, the ICSA results shall be reported from an undiluted sample analysis.

12.5.5 Results for the ICS Solution AB (ICSAB) during the analytical runs shall fall within the control limit of ±2 times the CRQL of the true value or ±20% of the true value, whichever is greater, for the analytes included in the ICSAB. If the analytical results of the ICSA do not fall within the control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and re-analysis of the analytical samples analyzed since the last compliant ICSAB shall be performed.

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 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

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[3]As defined in Exhibit G, ICSA and ICSAB are analytical samples.

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 on Form IVA-IN. Results shall fall within the control limit of ±2 times the CRQL of the established mean value or ±20% 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 on Form IVA-IN for all Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) analytes.

12.6 Spike Sample Analysis

12.6.1 The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology. If a digestion is performed, 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., water, soil) or for each SDG.[4]

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 2 - Spiking Levels for Spike Sample Analysis, for each element analyzed.

NOTE: See Table 2 footnotes for concentration levels and applications.

12.6.4 If the spike recovery is not at or within the limits of 75-125%, the data of all samples received and associated with that spike sample shall be flagged with the letter "N" on Forms IA/IB-IN and VA-IN. An exception to this rule is granted when the sample concentration exceeds the spike added 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 elements 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 sample at two times the indigenous level or two times the CRQL, whichever is greater. Results of the post-digestion spike shall be reported on Form VB-IN.

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[4]USEPA may require additional spike sample analyses, upon USEPA Regional CLP Project Officer (CLP PO) request.

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, flag all the samples of the same matrix and method in the SDG. Individual component percent recoveries are calculated as follows:

EQ. 5 Spike Percent Recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

WHERE, SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

12.6.7 When sample concentration is less than the Method Detection Limit (MDL), use SR = 0 only for purposes of calculating percent recovery. The Spike Sample Results (SSRs), Sample Results (SRs), Spike Added (SA), and percent recovery (positive or negative) shall be reported on Form VA-IN.

12.6.8 The units used for reporting SSRs will be identical to those used for reporting sample results on Form IA-IN.

12.7 Duplicate Sample Analysis

12.7.1 One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., water, soil) or for each SDG. [5] Duplicates cannot be averaged for reporting on Form IA-IN.

12.7.2 Duplicate sample analyses are 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 component 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)

D = Duplicate Result

12.7.3 The results of the duplicate sample analyses shall be reported on Form VI-IN. 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 of the CRQL value shall be entered in the "Control Limit" column on Form VI-IN 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

[5]USEPA may require additional duplicate sample analyses, upon USEPA Regional CLP PO request.

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 the MDL, the RPD is not calculated on Form VI-IN. For solid sample or solid 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 and percent solids), 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 "*" on Forms IA/IB-IN and VI-IN. 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 element. Specific control limits for each element will be added to Form VI-IN at a later date based on these precision results.

12.8 Laboratory Control Sample (LCS) Analysis

12.8.1 Water/aqueous and solid LCS 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.

12.8.1.1 The aqueous LCS solution (LCSW) shall be obtained from USEPA [if unavailable, the ICV solution(s) may be used]. One LCSW shall be prepared and analyzed for every group of aqueous samples in a SDG, or for each batch of aqueous samples digested, whichever is more frequent.

12.8.1.2 The USEPA provided solid LCS (LCSS) shall be prepared and analyzed using each of the procedures applied to the solid samples received (exception: percent solids determination not required). If the USEPA LCSS is unavailable, other USEPA QC Check Samples or other certified materials may be used. The control limits for these materials and samples must be documented. One LCSS shall be prepared and analyzed for every group of solid samples in a SDG, or for each batch of samples digested, whichever is more frequent.

12.8.2 All LCS and percent recovery results shall be reported on Form VII-IN. If the percent recovery for the LCSW falls outside the control limits of 80-120% (exception: Ag and Sb), the analyses shall be terminated, the problem corrected, and the samples associated with that LCSW redigested and re-analyzed with appropriate new QC.

12.8.3 If the results for the LCSS fall outside the control limits established by USEPA, the analyses shall be terminated, the problem corrected, and the samples associated with that LCSS redigested and re-analyzed with appropriate new QC.

12.9 ICP-AES Serial Dilution Analysis

12.9.1 Prior to reporting concentration data for the analyte elements, 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., water, soil) 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%, 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" on Form VIII-IN and Forms IA/IB-IN.

12.9.3 The percent differences for each component are calculated as follows:

EQ. 7 Serial Dilution Percent Differences

$$\% \text{ Difference} = \frac{|I - S|}{I} \times 100$$

WHERE, I = Initial Sample Result (Instrument reading)

S = Serial Dilution Result (Instrument reading x5)

12.9.4 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 on Form VIII-IN.

12.10 Method Detection Limit (MDL) Determination

12.10.1 Before any field samples are analyzed under this contract, the MDLs shall be determined for non-prepared analyses (Preparation Method/Code "NP1"), each digestion procedure and instrument used, prior to the start of contract analyses, and annually thereafter, and 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.

12.10.1.1 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. The Contractor shall also analyze the non-prepared MDL samples on each instrument used.

12.10.1.2 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C.

12.10.1.3 The concentration of the non-prepared MDL (Preparation Method/Code "NP1") shall be used to determine the appropriate concentration qualifier for the results of non-prepared samples and instrument QC analyses.

12.10.1.4 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).

12.10.1.5 The MDL results shall be reported on Form IX-IN.

12.11 Interelement Corrections

12.11.1 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 quarterly 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 quarterly and/or at multiple concentrations comparable to the sample interferent levels.

12.11.2 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 interelement correction factors determination shall be reported on Form XA-IN and Form XB-IN for all ICP-AES analytes.

12.12 Linear Range Analysis Standard (LRS)

12.12.1 A linear range verification check standard shall be analyzed and reported quarterly (i.e., January, April, July and October) for each element on Form XI-IN. The standard shall be analyzed during a routine analytical run performed under this contract. The analytically determined concentration of this standard shall be within 5% of the true value. This concentration is the upper limit of the ICP-AES linear range beyond which results cannot be reported under this contract without dilution of the analytical sample.

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 200.7. December 1982.

16.2 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3050B. Third Edition, Update III. December 1996.

16.3 American Society for Testing and Materials. Standard Practice for Sample Digestion Using Closed Vessel Microwave Heating Technique for the Determination of Total Recoverable Metals in Water. D4309-91. October 1991.

16.4 US Government Printing Office. 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

16.5 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3015. Third Edition, Update II. September 1994.

16.6 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 3051. Third Edition, Update II. September 1994.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1: Interferent and Analyte Elemental Concentrations Used for ICP-AES Interference Check Sample (ICS)

Analytes	(mg/L)	Interferents	(mg/L)
Ag	0.2	Al	250
As	0.1	Ca	250
Ba	0.5	Fe	100
Be	0.5	Mg	250
Cd	1.0		
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	0.05		
Sb	0.6		
Se	0.05		
Tl	0.1		
V	0.5		
Zn	1.0		

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.

TABLE 2: Spiking Levels for Spike Sample Analysis

Element	Water (µg/L)	Soil[1] (mg/kg)	Element	Water (µg/L)	Soil[1] (mg/kg)
Aluminum	2,000	*	Magnesium	*	*
Antimony	100	20	Manganese	500	100
Arsenic	40	8	Nickel	500	100
Barium	2,000	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	1,000	*			
Lead	20	4			

*No spike required. NOTE: Elements 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 wet 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 adjustment shall be made for microwave digestion procedures where 0.5 grams of sample or 50 mL (45 mL of sample plus 5 mL of acid) or 55 mL (50 mL of sample plus 5 mL of acid) of aqueous sample are required for analysis.

EQ. 8 Spiking Level Adjustment

$$\text{mg/kg} = \text{µg/L} \times \frac{\text{final volume (L)}}{\text{sample weight (g)}}$$

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EXHIBIT D - PART B

ANALYTICAL METHODS FOR INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY

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Exhibit D - Analytical Methods for ICP-MS

Table of Contents

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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 dissolved and total recoverable elements in water/aqueous 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.

2.0 SUMMARY OF METHOD

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 the use of internal standards.

3.0 DEFINITIONS

See Exhibit G 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 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* (krypton) has an isobaric elemental interference. 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 should be included with the report of 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. The 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.

4.3 Isobaric Polyatomic Ion Interferences

These 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 1 - 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 the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions.

4.4 Physical Interferences

These 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 should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard, containing the elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the Method Detection Limit (MDL) should be noted. 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 re-analyzed after a long 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 milliliter (mL) beaker or other appropriate vessel

6.1.2 Watch glasses

6.1.3 Funnels

6.1.4 Graduated cylinders

6.1.5 Various volumetric flasks (Type A)

6.1.6 Thermometer that covers range of 0-200°C

6.1.7 Whatman No. 42 filter paper or equivalent

6.1.8 Hot plate, block digester, or other heating source capable of maintaining 92-95°C.

6.1.9 Balances - Analytical Balance, 300 gram (g) capacity, and minimum ±0.1 milligram (mg).

6.2 Inductively Coupled Plasma Mass Spectrometer (ICP-MS) consisting of:

- An instrument capable of scanning the mass range 5-250 atomic mass unit (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. All acids used must be of ultra high-purity grade. 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 (HCl) is used, however, it should be noted that HCl is required to maintain stability in solutions containing antimony and silver. When HCl 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 D1193-77). Use this preparation for all reagents, standards, and dilutions of solutions.

7.1.2 Nitric Acid - Concentrated (specific gravity 1.41).

7.1.3 Nitric acid (1+1) - Add 500 milliliters (mL) conc. HNO_3 to 400 mL of reagent water and dilute to 1 Liter (L).

7.1.4 Nitric acid (1+9) - Add 100 mL conc. nitric acid to 400 mL of reagent water and dilute to 1 L.

7.1.5 Hydrochloric acid - Concentrated (specific gravity 1.19).

7.1.6 Hydrochloric acid (1+1) - Add 500 mL conc. HCl to 400 mL of reagent water and dilute to 1 L.

7.1.7 Hydrochloric acid (HCl) (1+4) - Add 200 mL conc. HCl to 400 mL reagent water and dilute to 1 L.

7.1.8 Ammonium hydroxide - Concentrated (specific gravity 0.902).

7.1.9 Tartaric acid - (CASRN 87-69-4).

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards 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 commercial source or prepared from ultra high-purity grade chemicals or metals (99.99-99.999% pure). All salts should 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 water, dried and weighed until the desired weight is achieved.

7.2.2.2 Aluminum solution, stock [1 mL = 1000 micrograms (µg) Al] - Pickle aluminum metal in warm (1+1) HCl to an exact weight of 0.100 g. Dissolve in 10 mL conc. HCl and 2 mL conc. nitric acid, heating to effect solution. Continue heating until the volume is reduced to 4 mL. Cool and add 4 mLs of reagent water. Heat until volume is reduced to 2 mL. Cool and dilute to 100 mL with reagent water.

7.2.2.3 Antimony solution, stock (1 mL = 1000 µg Sb) - Dissolve 0.100 g antimony powder in 2 mL (1+1) nitric acid and 0.5 mL conc. HCl, heating to effect solution. Cool, add 20 mL reagent water and 0.15 g tartaric acid. Warm the solution to dissolve the white precipitate. Cool and dilute to 100 mL with reagent water.

7.2.2.4 Arsenic solution, stock (1 mL = 1000 µg As) - Dissolve 0.1320 g As_2O_3 in a mixture of 50 mL reagent water and 1 mL conc. ammonium hydroxide. Heat gently to dissolve. Cool and acidify solution with 2 mL conc. nitric acid. Dilute to 100 mL with reagent water.

7.2.2.5 Barium solution, stock (1 mL = 1000 µg Ba) - Dissolve 0.1437 g $BaCO_3$ in a solution mixture of 10 mL reagent water and 2 mL conc. nitric acid. Heat and stir to effect solution and degassing. Dilute to 100 mL with reagent water.

7.2.2.6 Beryllium solution, stock (1 mL = 1000 µg Be) - Dissolve 1.965 g $BeSO_4 \cdot 4H_2O$ (DO NOT DRY) in 50 mL reagent water. Add 1 mL conc. nitric acid. Dilute to 100 mL with reagent water.

7.2.2.7 Bismuth solution, stock (1 mL = 1000 µg Bi) - Dissolve 0.1115 g Bi_2O_3 in 5 mL conc. nitric acid. Heat to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.8 Cadmium solution, stock (1 mL = 1000 µg Cd) - Pickle cadmium metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.9 Chromium solution, stock (1 mL = 1000 µg Cr) - Dissolve 0.1923 g CrO_3 in a solution mixture of 10 mL reagent water and 1 mL conc. nitric acid. Dilute to 100 mL with reagent water.

7.2.2.10 Cobalt solution, stock (1 mL = 1000 µg Co) - Pickle cobalt metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.11 Copper solution, stock (1 mL = 1000 µg Cu) - Pickle copper metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.12 Indium solution, stock (1 mL = 1000 µg In) - Pickle indium metal in (1+1) nitric acid to an exact weight of 0.100 g. Dissolve in 10 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.13 Lead solution, stock (1 mL = 1000 µg Pb) - Dissolve 0.1599 g $PbNO_3$ in 5 mL (1+1) nitric acid. Dilute to 100 mL with reagent water.

7.2.2.14 Magnesium solution, stock (1 mL = 1000 µg Mg) - Dissolve 0.1658 g MgO in 10 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.15 Manganese solution, stock (1 mL = 1000 µg Mn) - Pickle manganese flake in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.16 Nickel solution, stock (1 mL = 1000 µg Ni) - Dissolve 0.100 g nickel powder in 5 mL conc. nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.17 Scandium solution, stock (1 mL = 1000 µg Sc) - Dissolve 0.1534 Sc_2O_3 in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.18 Selenium solution, stock (1 mL = 1000 µg Se) - Dissolve 0.1405 g SeO_2 in 20 mL reagent water and dilute to 100 mL with reagent water.

7.2.2.19 Silver solution, stock (1 mL = 1000 µg Ag) - Dissolve 0.100 g silver metal in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water. Protect from the light.

7.2.2.20 Terbium solution, stock (1 mL = 1000 µg Tb) - Dissolve 0.1176 g Tb_4O_7 in 5 mL conc. nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.21 Thallium solution, stock (1 mL = 1000 µg Tl) - Dissolve 0.1303 g $TlNO_3$ in a solution mixture of 10 mL reagent water and 1 mL conc. nitric acid. Dilute to 100 mL with reagent water.

7.2.2.22 Vanadium solution, stock (1 mL = 1000 µg V) - Pickle vanadium metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.23 Yttrium solution, stock (1 mL = 1000 µg Y) - Dissolve 0.1270 g Y_2O_3 in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.2.24 Zinc solution, stock (1 mL = 1000 µg Zn) - Pickle zinc metal in (1+9) nitric acid to an exact weight of 0.100 g. Dissolve in 5 mL (1+1) nitric acid, heating to effect solution. Cool and dilute to 100 mL with reagent water.

7.2.3 Secondary Dilution Standards

Prepare mixed secondary dilution standard solutions by diluting the appropriate volumes of stock standards with acidified reagent water 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 elements are compatible and stable. Fresh calibration standards should be prepared from mixed standard solutions every two weeks or as needed. Dilute the mixed standards to levels appropriate to the operating range of the instrument using reagent water containing 1% (v/v) nitric acid. The element concentrations in the calibration standards should be sufficiently high to produce good measurement precision and to accurately define the slope of the response curve. If the direct addition procedure is being used, add internal standards.

7.2.4.2 Internal Standard Solution

Prepare mixed standard by diluting 10 mL each of the chosen element's stock standards to 100 mL with reagent water. Use this solution for additions to blanks, calibration standards, and samples, or dilute by an appropriate amount using 1% (v/v) nitric acid if the internal standards are being added by a peristaltic pump.

7.2.4.3 Tuning Solution

This solution is used for instrument tuning and mass calibration prior to analysis. Prepare mixed standard by diluting beryllium, magnesium, cobalt, indium, and lead stock standards to 100 µg/L with 1% (v/v) nitric acid. Do not add internal standard to this solution.

7.2.4.4 Interference Check Sample (ICS)

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. If the direct addition procedure is being used, add internal standards.

7.2.4.4.1 Solution A - Contains 100 milligrams per Liter (mg/L) of aluminum, calcium, iron, magnesium, potassium, sodium, phosphorus (as orthophosphate), sulfur (as sulfate), 200 mg/L carbon, 1000 mg/L chloride, and 2 mg/L molybdenum and titanium.

7.2.4.4.2 Solution AB - Contains all of the elements in Solution A plus all target analytes at a concentration of 20 µg/L.

7.2.4.5 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

The concentrations of the analytes in the CRI shall be at the CRQL. Information regarding the CRI shall be reported on Form IIB-IN.

7.2.4.6 Method Detection Limit (MDL) Solution

The MDL solution shall be at a concentration of 3 to 5 times the expected MDL.

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 (PB) (see Section 12.5.2) 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 in order to reduce memory interferences.

7.3.1 Calibration Blank - Consists of 1% (v/v) nitric acid in reagent water. If the direct addition procedure is being used, add internal standards.

7.3.2 Preparation Blank - Must contain all the reagents in the same volumes as used in preparing the samples. The PB must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.3.3 Rinse Blank - Consists of 2% (v/v) nitric acid in reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All samples must be collected in glass or polyethylene containers. Water/aqueous samples must be preserved with nitric acid to pH less than 2 immediately after collection. 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 (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 2 immediately after filtration.

8.2 Procedures for Sample Storage

The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after the 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 365 days 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, linear dynamic range, and interference effects must be investigated and established for each individual element 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 that 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 - 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 0.75 atomic mass unit (amu) at 5% peak height 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 a minimum of five times consecutively. The percent relative standard deviation of the absolute signals for all analytes in the tuning solution must be less than 5%.

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 4 - Internal Standards. For full range mass scans, a minimum of three internal standards shall be used. The masses of the internal standards shall bracket the masses of the analyte. Internal standards shall be present in all samples, standards, and blanks at identical levels. This may be achieved by directly adding an aliquot of the internal standards 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 concentration range of 20 µg/L to 200 µg/L of each internal standard is recommended. Internal standards should be added to samples, standards, and blanks in a similar manner, in order for dilution effects to be disregarded.

9.2.3 Calibration

Instruments shall be calibrated daily, once every 24 hours, or each time the instrument is set up. 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 two standards, one of which must be a blank standard.

A minimum of three replicate integrations are required for data acquisition. Use the average of the integrations for instrument calibration and data reporting.

NOTE: 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.

9.3.2 Only if the ICV solution(s) is(are) not available from USEPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the calibration range. An independent standard 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. The values for the ICV shall be reported on Form IIA-IN.

9.4 Continuing Calibration Verification (CCV)

9.4.1 To ensure calibration accuracy during each analysis run, one of the following standards shall be used for the CCV for each mass used for reporting final results for each element, at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The standard shall also be analyzed and reported for each mass used for reporting final results for each element at the beginning of the run and after the last analytical sample. The analyte concentrations in the CCV standard(s) shall be different from the concentrations for the ICV and shall be one of the following solutions at or near one-half of the calibration standard:

- USEPA Solutions
- NIST Standards
- A Contractor-prepared standard solution

The same CCV standard shall be used throughout the analysis runs 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 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses, and other related operations which may affect the CCV measured result may not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it, as well as the difference in time between the CCV and the analytical sample immediately preceding it, may not exceed the lowest difference in time between any two consecutive analytical samples associated with the CCV.

9.4.3 Information regarding the CCV shall be reported on Form IIA-IN.

9.5 Initial and Continuing Calibration Blank (ICB/CCB)

A calibration blank shall be analyzed for each mass used for reporting final results for each element immediately after every ICV and CCV, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. The blank shall be analyzed at the beginning of the run and after the last analytical sample.

NOTE: A CCB shall be analyzed immediately after the last CCV, and the last CCV shall be analyzed immediately after the last analytical sample of the run. The results of the calibration blanks shall be reported on Form III-IN.

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 Region for instructions. The 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 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 sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the Region. If all phases of the sample are amenable to analysis, the 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 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 Region's decision in the SDG Narrative.

10.1.3 Sample Preparation Procedures

10.1.3.1 Direct Analyses - Preparation Method/Code (NP1)

10.1.3.1.1 For the analysis of dissolved analytes in water samples, transfer 20 milliliters (mL) of the filtered, acid-preserved sample to a clean, closeable container such as a centrifuge

tube. Add sufficient (1+1) nitric acid to make the sample 1% (v/v) acid. If the direct addition procedure is being used, add internal standards, close the container and mix. The sample is now ready for analysis. If a precipitate is formed during acidification, transport, or storage, sample digestion is required as described in Section 10.1.3.2.

10.1.3.1.2 This Preparation Method/Code shall also be used to report the direct analysis Method Detection Limit (MDL). The concentration of this MDL shall be used to determine the appropriate concentration qualifier for the results of non-prepared samples and instrument Quality Control (QC) analyses.

10.1.3.1.3 Prior to the analysis of aqueous samples for total recoverable analytes (excluding CLP Quarterly Blind (QB) samples), determine the turbidity and report the results of these measurements, in Nephelolometric Turbidity Units (NTU), in the raw data. If the turbidity is less than 1 NTU, direct analysis of the sample shall be performed using the procedure in Section 10.1.3.1.1. If the turbidity is greater than or equal to 1 NTU, digest the sample as follows:

10.1.3.2 Preparation Method/Code (HW2)

Shake and transfer a 100 mL aliquot of the sample to a 250 mL heating vessel, add 2 mL (1+1) nitric acid and 1 mL of (1+1) hydrochloric acid (HCl) to the sample. Cover with a ribbed watch glass and heat on either a hot plate, block digester, or equivalent heating source which is adjustable and capable of maintaining a temperature of 92-95°C for 2 hours, or until the sample volume is reduced to about 20 mL (DO NOT BOIL). Cover with a watch glass to prevent additional evaporation and reflux for 30 minutes. Cool sample, transfer to a 50 mL volumetric flask, and adjust sample volume to 50 mL with reagent water. Mix and allow any solids present to settle by gravity overnight or centrifuge (if after settling or centrifuging, the sample contains suspended solids, a portion of the sample may be filtered prior to analysis).

10.1.3.2.1 Prior to analysis, adjust the chloride concentration by pipetting 20 mL of the digestate into a 50 mL volumetric flask and dilute to volume with reagent water and mix. If the direct addition method is being used, add internal standards and mix. The sample is now ready for analysis.

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. 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 linear 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.

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 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 or a minimum of one minute. Samples should be aspirated for a sufficient period of time to obtain a stable response prior to the collection of data.

10.2.4 Samples having concentrations higher than the established linear dynamic range should be diluted into range and re-analyzed. The sample 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 should then be diluted for the determination of the remaining elements. Alternatively, the dynamic range may be adjusted by selecting an alternative isotope of lower natural abundance, provided QC data for that isotope have been established. The dynamic range must not be adjusted by altering instrument conditions to an uncharacterized state.

10.2.5 All masses which might affect data quality must be monitored during the analytical run. At a minimum, those masses prescribed in Table 2 - Mass Choices for Elements that Must Be Monitored During the Analytical Run, 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.

10.2.6 During the analysis of samples, the laboratory must comply with the required QC described in Section 12. For the determination of dissolved analytes or the direct analysis of aqueous samples with turbidity less than 1 NTU, the Preparation Blank (PB) and Laboratory Control Sample (LCS) are not required.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Recommended Elemental Equations

Elemental expressions recommended for sample data calculations are listed in Table 3 - Recommended Elemental Expressions for Isobaric Interferences. Do not report element concentrations below the determined Method Detection Limit (MDL).

11.2 Data Value Corrections

Data values 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 (HCl), 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 Direct Analysis

EQ. 1 Non-Prepared Sample Concentration

$$\text{Concentration } (\mu g/L) = C \times DF$$

WHERE,
- C = Instrument value in µg/L. (The average of all replicate integrations).
- DF = Dilution Factor

11.5 Prepared Sample Analysis

EQ. 2 Prepared Sample Concentration

$$\text{Concentration } (\mu g/L) = C \times \frac{V_f}{V_i} \times \frac{V_f}{20} \times DF$$

WHERE,
- C = Instrument value in µg/L (The average of all replicate integrations).
- V_f = Final digestion volume (50 mL)
- V_i = Initial digestion volume (100 mL)
- DF = Dilution Factor

11.6 Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation

To calculate the adjusted CRQL or adjusted MDL, multiply the value of the CRQL (µg/L) or MDL (µg/L) by the sample 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 at least 5 times consecutively. If the peak width at 5% peak height is not within 0.75 atomic mass units (amu) for each isotope, 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 the absolute signals of the analytes exceeds 5%, the analysis shall be terminated, the problem corrected, and the instrument re-tuned. 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 measurements exceed the control limits of 90% (low) and 110% (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. Information regarding the ICV shall be reported on Form IIA-IN.

12.3 Continuing Calibration Verification (CCV)

The CCV standard shall be prepared by combining compatible elements at a concentration equivalent to the mid-points of their respective calibration curves. If the deviation of the CCV is greater than the specified control limits of 90% (low) and 110% (high), the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration verification shall be performed for the elements affected. Information regarding the CCV shall be reported on Form IIA-IN.

12.4 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

12.4.1 To verify linearity near the CRQL, a standard at the CRQL (CRI) shall be prepared, in the same acid matrix as the calibration standards, and analyzed at the beginning and end of each sample analysis run, but not before the ICV. In addition, the contractor shall analyze the CRI at a frequency of not less than once per 20 analytical samples[1] per analysis run. The initial analysis of the CRI shall be immediately followed by the Interference Check Samples (ICS) analyses.

12.4.2 The CRI shall be run for every required isotope used for the analysis of all Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) analytes. Information regarding the CRI shall be reported on Form IIB-IN.

12.4.3 If the percent recovery of the CRI falls outside the control limits of 70-130% (50-150% for cobalt, manganese, and zinc) for one or more analytes, the CRI shall be re-analyzed immediately for those analytes

[1]As defined in Exhibit G, CRI is an analytical sample.

only. If the results of the re-analysis for those analytes fall within the control limits, no further corrective action is required. If the results of the re-analysis for those analytes do not fall within the control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the CRI analyzed, and the samples associated with the CRI re-analyzed.

12.5 Blank Analyses

There are two different types of blanks required by this method. The calibration blank is used in establishing the analytical curve while the preparation blank is used to monitor for possible contamination.

12.5.1 Initial and Continuing Calibration Blank (ICB/CCB)

The ICB and CCB are prepared with acid and reagent water. If the absolute value of the calibration blank (ICB/CCB) result exceeds the CRQL (see Exhibit C), the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank shall be performed for the elements affected.

12.5.2 Preparation Blank (PB)

12.5.2.1 The PB shall contain all the reagents and in the same volumes as used in processing the samples. The PB shall be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

12.5.2.2 At least one PB, 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 [2] of samples digested, whichever is more frequent.

12.5.2.3 The first batch of samples in an SDG is to be assigned to Preparation Blank one, the second batch to Preparation Blank two, etc. (see Form III-IN). Each Sample Data Package shall contain the results of all PB analyses associated with the samples in that SDG.

12.5.2.4 The PB is to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

12.5.2.4.1 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.

12.5.2.4.2 If the analyte concentration in the blank is above the CRQL, the lowest concentration of that analyte in the associated samples shall be greater than or equal to 10 times the blank concentration. Otherwise, all samples, associated with the blank, with the analyte concentration less than 10 times the blank concentration and above the CRQL, shall be redigested and re-analyzed with appropriate new Quality Control (QC) for that analyte. The only exception to this shall be an identified field blank. The sample concentration is not to be corrected for the blank value.

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[2] A group of samples prepared at the same time.

12.5.2.4.3 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 re-analyzed with appropriate new QC.

12.5.2.4.4 The values for the PB shall be reported on Form III-IN.

12.6 Interference Check Sample (ICS)

12.6.1 The ICS is prepared by the analyst or obtained from USEPA, if available.

12.6.2 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 for all interferents (target and non-target), at the beginning of each analysis run, but not before the ICV. This analysis of the ICS shall be immediately followed by 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 not dilute the ICS (for the higher concentration elements) more than is necessary to meet the linear range values of the instrument.

12.6.3 The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A.

12.6.4 The analytical results of ICS Solution A (ICSA) shall fall within the control limit of ±3 times the CRQL of the analyte's true value or ±20% of the analyte's true value (the true value shall be zero unless otherwise stated) in the ICSA, whichever is greater. If not, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and re-analysis of the analytical samples analyzed since the last compliant ICSA shall be performed. The ICSA results for these analytes shall be reported from an undiluted sample analysis.

12.6.5 Results for the ICS Solution AB (ICSAB) during the analytical runs shall fall within the control limit of ±3 times the CRQL of the true value or ±20% of the true value, whichever is greater, for the analytes included in the ICSAB. If not, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and re-analysis of the analytical samples analyzed since the last compliant ICSAB shall be performed.

NOTE: The control limits and concentrations for the ICSAB are being monitored. These may be adjusted to provide greater control of interferences.

12.6.6 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 Sections 7.2.4.4.1 and 7.2.4.4.2. The mean value and standard deviation shall be established by initially analyzing

the Check Samples at least five times repetitively for each analyte listed on Form IVB-IN. Results shall fall within the control limit of ±3 times the CRQL of the established mean value or ±20% 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 on Form IVB-IN for all ICP-MS parameters.

12.7 Spike Sample Analysis

12.7.1 The spike sample analysis is designed to provide information about the effect of sample matrix on the digestion and/or measurement methodology. If a digestion is performed, 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 for each SDG[3].

12.7.2 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 "original sample" (see Section 12.8). 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.

12.7.3 The analyte spike shall be added in the amount given in Table 5 - Spiking Levels for Spike Sample Analysis, for each element analyzed.

12.7.4 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 shall be flagged with the letter "N" on Forms IA/IB-IN and VA-IN. 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.

12.7.5 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 elements 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 shall be used for the post-digestion spike analysis. Spike an unspiked aliquot of the digestate at two times the indigenous level or two times the CRQL, whichever is greater. Results of the post-digestion spike shall be reported on Form VB-IN.

12.7.6 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, flag all the samples in the SDG. Individual component percent recoveries are calculated as follows:

EQ. 3 Spike Percent Recovery

$$\%\text{Recovery} = \frac{SSR - SR}{SA} \times 100$$

[3]USEPA may require additional spike sample analyses, upon USEPA Regional CLP Project Officer (CLP PO) request.

WHERE, SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

12.7.7 When sample concentration is less than the Method Detection Limit (MDL), use SR = 0 only for purposes of calculating percent recovery. The Spike Sample Results (SSRs), Sample Results (SRs), Spike Added (SA), and percent recovery (positive or negative) shall be reported on Form VA-IN.

12.7.8 The units used for reporting SSRs will be identical to those used for reporting sample results on Form IA-IN.

12.8 Duplicate Sample Analysis

12.8.1 One duplicate sample shall be analyzed for each SDG[4]. Duplicates cannot be averaged for reporting on Form IA-IN.

12.8.2 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. 4 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

WHERE, RPD = Relative Percent Difference

S = Sample Result (original)

D = Duplicate Result

12.8.3 The results of the duplicate sample analyses shall be reported on Form VI-IN. 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 on Form VI-IN 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.

12.8.4 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 MDL, the RPD is not calculated on Form VI-IN. If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*" on Forms IA/IB-IN and VI-IN. 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 in the SDG. The percent

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[4]USEPA may require additional duplicate sample analyses, upon USEPA Regional CLP PO request.

difference data will be used by USEPA to evaluate the long-term precision of the methods for each element. Specific control limits for each element may be added to Form VI-IN at a later date based on these precision results.

12.9 Laboratory Control Sample (LCS) Analysis

12.9.1 A water/aqueous LCS (LCSW) shall be analyzed for each analyte using the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures employed for USEPA samples received.

12.9.2 The LCSW solution must be obtained from USEPA (if unavailable, the ICV solution(s) may be used). One aqueous LCS shall be prepared and analyzed for each group of samples in an SDG, or for each batch of samples digested, whichever is more frequent.

12.9.3 All LCSW and percent recovery results shall be reported on Form VII-IN. If the percent recovery for the LCSW falls outside the control limits of 80-120%, the analyses shall be terminated, the problem corrected, and the samples associated with that LCSW redigested and re-analyzed with appropriate new QC.

12.10 ICP-MS Serial Dilution Analysis

12.10.1 Prior to reporting concentration data for the analyte elements, 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 SDG. Samples identified as field blanks and PE samples shall not be used for serial dilution analysis.

12.10.2 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" on Forms IA/IB-IN and VIII-IN.

12.10.3 The percent differences for each component are calculated as follows:

EQ. 5 Serial Dilution Percent Difference

$$\% \text{ Difference} = \frac{|I - S|}{I} \times 100$$

WHERE, I = Initial Sample Result (Instrument Reading)

S = Serial Dilution Result (Instrument Reading x5)

12.10.4 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 in the SDG. Serial dilution results and "E" flags shall be reported on Form VIII-IN.

12.11 Internal Standards

12.11.1 The analyst shall monitor the responses from the internal standards throughout the sample set being analyzed. Ratios of the 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% of the original response in the calibration blank. If deviations greater than these are observed, the laboratory shall monitor the calibration blank internal standard responses by re-analyzing the calibration blank. If these are within the limits, the original sample shall be diluted by a factor of two, internal standards added, and the sample re-analyzed. If the internal standard responses for the calibration blank are not within the limits, terminate the analysis, correct the problem, recalibrate, verify the calibration and re-analyze all analytical samples analyzed since the last compliant calibration blank. If the internal standard responses for the diluted sample analysis are not within the limits, note this in the SDG Narrative.

12.12 Method Detection Limit (MDL) Determination

12.12.1 Before any field samples are analyzed under this contract, the MDLs shall be determined for each instrument used, prior to the start of contract analyses, and annually thereafter, and 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.

12.12.2 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. The Contractor shall also analyze non-prepared MDL samples on each instrument used.

12.12.3 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C.

12.12.4 The direct analysis MDL (Preparation Method/Code "NP1") shall be used to determine the appropriate concentration qualifier for the results of instrument QC.

12.12.5 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).

12.12.6 The MDL results shall be reported on Form IX-IN.

12.13 Linear Dynamic Range (LDR)

12.13.1 Before any field samples are analyzed under this contract, the upper limit of the linear calibration range shall be established for each analyte by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. The linear calibration range used for the analysis of samples shall be determined from the resulting data. The upper LDR limit shall be an observed signal no more than 10% below the level extrapolated from lower standards. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and re-analyzed. The LDRs must be verified whenever a change in instrument hardware operating

conditions indicate they should be redetermined, or verified quarterly.

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 5.4. 1994.

16.2 US Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Method 6020A. Third Edition, Update IV-A. 1986.

16.3 US Government Printing Office. 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Table 1. 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							

Table 1. Isobaric Molecular-Ion Interferences (Con't)

Analyte	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{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	
^{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}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo^{++}
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	S_2	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	S_2	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}

Table 1. Isobaric Molecular-Ion Interferences (Con't)

Analyte	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{67}Zn	VO	TiOH	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	Cl_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 2. Mass Choices for Elements that Must Be Monitored During the Analytical Run

Mass	Element of Interest
27	Aluminum
121	Antimony
75	Arsenic
134, 135, 136, 137	Barium
9	Beryllium
111, 114	Cadmium
52, 53	Chromium
59	Cobalt
63, 65	Copper
206, 207, 208	Lead
24, 25, 26	Magnesium
55	Manganese
60, 61, 62	Nickel
77, 78, 80, 82	Selenium
107, 109	Silver
203, 205	Thallium
51	Vanadium
66, 67, 68	Zinc

NOTE: Underlined isotopes are preferred for measurements. Where possible, alternative isotopes are indicated. 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 3. Recommended Elemental Expressions for Isobaric Interferences

Element	Isobaric Correction	Expression Proportional to Elemental Concentration
Al	none	(1.0000) (^{27}C)
Sb	none	(1.0000) (^{121}C)
As	ArCl, Se	(1.0000) (^{75}C) - (3.127) [(^{77}C) - (0.815) (^{82}C)]
Ba	none	(1.0000) (^{137}C)
Be	none	(1.0000) (^{9}C)
Cd	MoO, Pd	(1.000) (^{111}C) - (1.073) [(^{108}C) - (0.712) (^{106}C)]
Cr	none	(1.0000) (^{52}C)
Co	none	(1.0000) (^{59}C)
Cu	none	(1.0000) (^{63}C)
Pb	none	(1.0000) (^{206}C) + (1.0000) (^{207}C) + (1.0000) (^{208}C)
Mg	none	(1.0000) (^{25}C)
Mn	none	(1.0000) (^{55}C)
Ni	none	(1.0000) (^{60}C)
Se	none	(1.0000) (^{78}C)
Ag	none	(1.0000) (^{107}C)
Tl	none	(1.0000) (^{205}C)
V	ClO, Cr	(1.0000) (^{51}C) - (3.127) [(^{53}C) - (0.113) (^{52}C)]
Zn	none	(1.0000) (^{66}C)
Sc	none	(1.0000) (^{45}C)
Y	none	(1.0000) (^{89}C)
Rh	none	(1.0000) (^{103}C)
In	Sn	(1.0000) (^{115}C) - (0.0140) (^{118}C)
Tb	none	(1.0000) (^{159}C)
Ho	none	(1.0000) (^{165}C)
Bi	none	(1.0000) (^{209}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.

The correction equations shall not be applied if appropriate interference check sample measurement demonstrates absence of interference above the CRQL.

Table 4. Internal Standards (must use at least three)

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
Indium	115	7440-74-6
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 5. Spiking Levels for Spike Sample Analysis

Analyte	Spike (µg/L)
Al	2000
Sb	100
As	40
Ba	2000
Be	50
Cd	50
Cr	200
Co	500
Cu	250
Pb	20
Mn	500
Ni	500
Se	10
Ag	50
Tl	50
V	500
Zn	500

EXHIBIT D - PART C

ANALYTICAL METHODS
FOR
COLD VAPOR MERCURY ANALYSIS

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Exhibit D - Analytical Methods for Cold Vapor Mercury Analysis

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Exhibit D - Analytical Methods for Cold Vapor Mercury Analysis

Table of Contents (Con't)

1.0 SCOPE AND APPLICATION

The analytical method that follows is designed to analyze water, sediment, sludge, and soil 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 mercurials 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 organic mercurials, 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 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.

The range of the method may be varied through instrument and/or recorder expansion. Using a 100 milliliters (mL) sample, a detection limit of less than 0.1 micrograms per Liter (µg/L) can be achieved.

The range of the method for soil/sediments is 0.05 milligrams per kilogram (mg/kg) to 5 mg/kg. The range may be extended above or below the normal range by increasing or decreasing sample size or through instrument and recorder control.

2.0 SUMMARY OF METHOD

2.1 Water by Automated and Manual Techniques

This is a physical method based on the absorption of radiation at 253.7 nanometers (nm) by mercury vapor. Free mercury atoms can exist at room temperature; therefore, mercury can be measured by Atomic Absorption (AA) without a heated sample cell. Organic compounds are oxidized, and in the cold vapor mercury technique, mercury is chemically reduced to the free atomic state by reacting the sample with a strong reducing agent like stannous chloride or sodium borohydride 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 AA spectrophotometer. Sometimes the cell is heated slightly to avoid water condensation; otherwise the cell is completely unheated. 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 will be taken as the analytical signal.

2.2 Soil/Sediment by Manual Technique

2.2.1 A weighed portion of the sample is acid digested for 2 minutes at 95°C, followed by oxidation with potassium permanganate and potassium persulfate. Mercury in the digested sample is then measured by the conventional cold vapor technique.

2.2.2 An alternate digestion involving the use of an autoclave is described in Section 10.1.4.2.1.2.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES

4.1 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 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 Formation of a heavy precipitate, in some wastewaters and effluents, has been reported upon addition of concentrated sulfuric acid. If this is encountered, the problem sample cannot be analyzed by this method.

4.1.3 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 milligram per Liter (mg/L) of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

4.1.4 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.

4.1.5 Samples containing solids must be blended and then mixed while being sampled if total mercury values are to be reported.

4.2 Soil/Sediment

4.2.1 The same types of interferences that may occur in water samples are also possible with sediments (i.e., sulfides, high copper, high chlorides, etc.).

4.2.2 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 weight of the original sample or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

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 General Information for Water and Soils (Automated and Manual Techniques)

6.1.1 Atomic Absorption (AA) Spectrophotometer - Any AA unit having an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed.

NOTE: Instruments designed specifically for the measurement of mercury using the cold vapor technique are commercially available and may be substituted for the AA spectrophotometer.

NOTE: All cold vapor mercury analyzers shall be equipped with all manufactured required equipment (i.e., dryers) to ensure that the specified CRQLs are met.

6.1.2 Mercury Hollow Cathode Lamp

6.1.3 Recorder - Any multi-range variable speed recorder that is compatible with the UV detection system is suitable.

6.2 Water by Automated Technique

6.2.1 Automated Analyzer instrumentation consisting of:

6.2.1.1 Sampler with provision for sample mixing

6.2.1.2 Manifold

6.2.1.3 Proportioning Pump(s)

6.2.1.4 High temperature heating bath with distillation coil(s)

6.2.1.5 Vapor-liquid separator

6.2.1.6 Absorption cell with quartz windows

6.3 Water and Soil/Sediment by Manual Technique

6.3.1 Absorption Cell - Standard spectrophotometer cells

6.3.2 Air Pump - Any device capable of delivering 1 Liter (L) of air per minute may be used.

6.3.3 Flowmeter - Capable of measuring an air flow of 1 L per minute.

6.3.4 Aeration Tubing - Tygon tubing is used for transporting the mercury vapor from the sample bottle to the absorption cell and for its return.

6.3.4.1 Straight glass tubing terminating in a coarse porous frit is used for sparging air into the sample.

6.3.5 Drying Tube - 6" X 3/4" diameter tube containing 20 grams (g) of magnesium perchlorate.

NOTE: In place of the magnesium perchlorate drying tube, a small reading lamp with a 60-watt bulb may be used to prevent condensation of moisture inside the cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10°C above ambient temperature.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Water by Automated Technique

7.1.1.1 Reagent Water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-77). 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, 2N - Dilute 56 milliliters (mL) of concentrated sulfuric acid to 1 Liter (L) with reagent water.

7.1.1.2.2 Sulfuric acid, 10% - Dilute 100 mL concentrated sulfuric acid to 1 L with reagent water.

7.1.1.3 Nitric acid, concentrated - Reagent grade of low mercury content.

Nitric acid, 0.5% wash solution - Dilute 5 mL of concentrated nitric acid to 1 L with reagent water.

7.1.1.4 Stannous sulfate - Add 50 grams (g) stannous sulfate to 500 mL of 2N sulfuric acid (see Section 7.1.1.2.1). This mixture is a suspension and should be stirred continuously during use.

NOTE: Stannous chloride may be used in place of stannous sulfate.

7.1.1.5 Sodium chloride-hydroxylamine sulfate solution - Dissolve 30 g of sodium chloride and 30 g of hydroxylamine sulfate in reagent water to 1 L.

NOTE: Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.

7.1.1.6 Potassium permanganate ($KMnO_4$) - 0.5% solution, w/v. Dissolve 5 g of potassium permanganate in 1 L of reagent water.

7.1.1.7 Potassium permanganate, 0.1N - Dissolve 3.16 g of potassium permanganate in reagent water and dilute to 1 L.

7.1.1.8 Potassium persulfate - 0.5% solution, w/v. Dissolve 5 g of potassium persulfate in 1 L of reagent water.

7.1.1.9 Air scrubber solution - Mix equal volumes of 0.1N potassium permanganate (see Section 7.1.1.6) and 10% sulfuric acid (see Section 7.1.1.2.2).

7.1.2 Water and Soil/Sediment by Manual Technique

7.1.2.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-77). Use this preparation for all reagents, standards, and dilutions of solutions.

7.1.2.2 Sulfuric acid, concentrated - Reagent grade.

7.1.2.2.1 Sulfuric acid, 0.5N - Dilute 14.0 mL of concentrated sulfuric acid to 1 L. (Water technique only.)

7.1.2.3 Nitric acid, concentrated - Reagent grade of low mercury content. If a high Preparation Blank (PB) is obtained, it may be necessary to distill the nitric acid.

7.1.2.4 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.

7.1.2.5 Sodium chloride-hydroxylamine sulfate solution - Dissolve 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.

7.1.2.6 Potassium permanganate ($KMnO_4$) - 5% solution, w/v. Dissolve 5 g of potassium permanganate in 100 mL of reagent water.

7.1.2.7 Potassium persulfate - 5% solution, w/v. Dissolve 5 g of potassium persulfate in 100 mL of reagent water.

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards 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 or prepared from ultra high purity grade chemicals or metals.

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 micrograms (µg) per mL. This working standard and the dilutions of the stock mercury solution should 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.

7.2.2 Working Standards

7.2.2.1 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

The concentration of the CRI for mercury shall be at the CRQL. Information regarding the CRI shall be reported on Form IIB-IN.

7.2.2.2 Method Detection Limit (MDL) Solution

The MDL solution shall be at a concentration of 3 to 5 times the expected MDL.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All samples must be collected in glass or polyethylene containers. Water/aqueous samples must be preserved with nitric acid to pH less than 2 immediately after collection. 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 (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 2 immediately after filtration.

8.2 Procedure for Sample Storage

The 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 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 Cold Vapor Atomic Absorption (AA) Instrument Calibration Procedure

9.1.1 Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument standardization date and time shall be included in the raw data.

9.1.2 The date and time of preparation and analysis shall be given in the raw data.

9.1.3 Calibration standards shall be prepared fresh with each preparation batch. Prepare a minimum of five calibration standards (which includes a blank) in graduated amounts in the appropriate range. One of the standards must be at the Contract Required Quantitation Limit (CRQL).

9.1.4 Aspirate the standards and record the readings. Results for these standards shall be within 5% of the true value. Each standard concentration and the calculations to show that the 5% criteria has been met shall be given in the raw data. If the values do not fall within this range, recalibration is necessary. The 5% criteria does not apply to the calibration standard at the CRQL. The acceptance criteria for the initial calibration curve is a correlation coefficient more than or equal to 0.995.

9.1.5 Baseline correction is acceptable as long as it is performed after every sample or after the Continuing Calibration Verification (CCV) and Blank (CCB) check. Resloping is acceptable as long as it is immediately preceded and immediately followed by a compliant CCV and CCB.

9.2 Initial Calibration Verification (ICV)

9.2.1 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.

9.2.2 Only if the ICV solution is not available from USEPA, or where a certified solution of the analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analyte from a different source than that used in the standards for the instrument calibration. The value for the ICV shall be reported on Form IIA-IN.

9.3 Continuing Calibration Verification (CCV)

9.3.1 To ensure calibration accuracy during each analysis run, one of the following standards is to be used for the CCV and shall be analyzed and reported at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The standard shall also be analyzed and reported at the beginning of the run and after the last analytical sample. The analyte concentration in the CCV standard shall be different than the concentration used for the ICV and shall be one of the following solutions at or near the mid-range level of the calibration curve:

- USEPA Solutions
- NIST Standards
- A Contractor-prepared standard solution

The same CCV standard shall be used throughout the analysis runs for a Sample Delivery Group (SDG) of samples received.

9.3.2 Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses, and other related operations that may affect the CCV measured result may not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it, as well as the difference in time between the CCV and the analytical sample immediately preceding it, may not exceed the lowest difference in time between any two consecutive analytical samples associated with the CCV.

9.3.3 Information regarding the CCV shall be reported on Form IIA-IN.

9.4 Initial and Continuing Calibration Blank (ICB/CCB)

A calibration blank shall be analyzed at each wavelength used for analysis immediately after every ICV and CCV, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. The blank shall be analyzed at the beginning of the run and after the last analytical sample.

NOTE: A CCB shall be analyzed immediately after the last CCV, and the last CCV shall be analyzed immediately after the last analytical sample of the run. The results for the calibration blanks shall be reported on Form III-IN.

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 Region for instructions. The 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 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 sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the Region. If all phases of the sample are amenable to analysis, the 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 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 Region's decision in the SDG Narrative.

10.1.3 Water Preparation of Standards and Samples (Manual Technique)

10.1.3.1 Standards Preparation

10.1.3.1.1 Transfer aliquots of the working mercury solution to a series of 300 milliliters (mL) BOD bottles. Add enough reagent water to each bottle to make a total volume of 100 mL.

10.1.3.1.2 Mix thoroughly and add 5 mL of concentrated sulfuric acid (see Section 7.1.2.2) and 2.5 mL of concentrated nitric acid (see Section 7.1.2.3) to each bottle. Add 15 mL of $KMnO_4$ (see Section 7.1.2.6) solution to each bottle and allow to stand at least 15 minutes. Add 8 mL of potassium persulfate (see Section 7.1.2.7) to each bottle and heat for 2 hours in a water bath maintained at 95°C. (If an autoclave is employed, cover the BOD bottles with foil and heat in the autoclave for 15 minutes at 120°C and 15 PSI instead of heating for 2 hours in a waterbath at 95°C). Cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution (see Section 7.1.2.5) to reduce the excess permanganate. When the solution has been

decolorized, wait 30 seconds, add 5 mL of the stannous sulfate solution (see Section 7.1.2.4) and immediately attach the bottle to the aeration apparatus to form a closed system. At this point the sample is allowed to stand quietly without manual agitation.

10.1.3.1.3 The circulating pump, which has previously been adjusted to a rate of 1 Liter (L) per minute, is allowed to run continuously (see Note 1). The absorbance will increase and reach maximum within 30 seconds. As soon as the response levels off, open the bypass valve and continue the aeration until the absorbance returns to its minimum value (see Note 2). Close the bypass valve, remove the stopper and frit from the BOD bottle and continue the aeration. Proceed with the standards and construct a standard curve by plotting instrument response at 253 nanometers (nm) versus micrograms (µg) of mercury.

NOTE 1: An open system where the mercury vapor is passed through the absorption cell only once may be used instead of the closed system.

NOTE 2: Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system to either vent the mercury vapor into an exhaust hood or pass the vapor through some absorbing media, such as equal volumes of 0.1 M $KMnO_4$, and 10% H_2SO_4 or 0.25% iodine in a 3% KI solution. A specially treated charcoal that will adsorb mercury vapor is commercially available.

10.1.3.2 Sample Preparation

10.1.3.2.1 Preparation Method/Code (CW1)

10.1.3.2.1.1 Transfer 100 mL, or an aliquot diluted to 100 mL, containing not more than 1.0 µg of mercury, to a 300 mL BOD bottle and continue as described in Section 10.1.3.1.2.

NOTE: The same amount of $KMnO_4$ added to the samples should be present in standards and blanks.

10.1.3.2.1.2 Cool and add 6 mL of sodium chloride-hydroxylamine sulfate (see Section 7.1.2.5) to reduce the excess permanganate. Purge the headspace in the BOD bottle for at least 1 minute and add 5 mL of stannous sulfate (see Section 7.1.2.4) and immediately attach the bottle to the aeration apparatus.

NOTE: Add reductant in 6 mL increments until $KMnO_4$ is completely reduced (until the color is no longer purple).

10.1.4 Soil/Sediment Preparation of Standards and Samples (Manual)

10.1.4.1 Standards Preparation

10.1.4.1.1 Transfer aliquots of the working mercury solutions (see Section 7.2.1.3) to a series of 300 mL BOD bottles. Add enough reagent water to each bottle to make a total volume of 10 mL.

10.1.4.1.2 Add 5 mL of concentrated H_2SO_4 (see Section 7.1.2.2) and 2.5 mL of concentrated HNO_3 (see Section 7.1.2.3) and heat 2 minutes in a water bath at 95°C. Allow the sample to cool and add 50 mL reagent water, 15 mL of $KMnO_4$ solution (see Section 7.1.2.6) and 8 mL of potassium persulfate solution (see Section 7.1.2.7)

to each bottle and return to the water bath for 30 minutes. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution (see Section 7.1.2.5) to reduce the excess permanganate. Add 50 mL of reagent water (final volume of reagent water = 100 mL). Treating each bottle individually, add 5 mL of stannous sulfate solution (see Section 7.1.2.4) and immediately attach the bottle to the aeration apparatus. At this point the sample is allowed to stand quietly without manual agitation. If an autoclave is used, the standards shall be prepared in the same way as the samples (see Section 10.1.4.2.1.2).

10.1.4.1.3 The circulating pump, which has previously been adjusted to a rate of 1 L per minute, is allowed to run continuously. The absorbance, as exhibited either on the spectrophotometer or the recorder, will increase and reach maximum within 30 seconds. As soon as the response levels off, open the bypass valve and continue the aeration until the absorbance returns to its minimum value. Close the bypass valve, remove the fritted tubing from the BOD bottle and continue the aeration. Proceed with the standards and construct a standard curve by plotting peak height versus µg of mercury.

10.1.4.2 Sample Preparation

10.1.4.2.1 Preparation Method/Code (CS1)

10.1.4.2.1.1 Weigh a representative 0.20 g (±0.01 g) portion of wet sample and place in the bottom of a BOD bottle. Add enough reagent water to each sample to make a total volume of 10 mL. Continue as described in Section 10.1.4.1.2.

10.1.4.2.1.2 If an autoclave is used, add 5 mL of concentrated H_2SO_4 and 2 mL of concentrated HNO_3 to the 0.20 g (±0.01 g) of sample. Add 5 mL of saturated $KMnO_4$ solution and 8 mL of potassium persulfate solution and cover with a piece of aluminum foil. The sample is autoclaved at 120°C and 15 PSI for 15 minutes. Cool, make up to a volume of 100 mL with reagent water, and add 6 mL of sodium chloride-hydroxylamine sulfate solution (see Section 7.1.2.5) to reduce the excess permanganate. Purge the headspace of the sample bottle for at least one minute and continue as described under Section 10.1.4.1.2.

10.1.5 Preparation of Standards for Automated Cold Vapor Analysis Technique (Analysis Method - AV)

10.1.5.1 Standards Preparation

Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 µg per mL. This working standard and the dilutions of the stock mercury solution should 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.

10.2 Sample Analysis

10.2.1 Set up instrument with proper operating parameters.

10.2.2 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using calibration standard

solutions mentioned in Section 9.1. Samples prepared by a certain method must be analyzed with calibration and QC standards prepared by the same method. Therefore, only one Preparation Method/Code can be associated with each run.

10.2.3 Analyze the Continuing Calibration Verification (CCV) instrument check standard and the Continuing Calibration Blank (CCB) after every 10 analytical samples.

10.2.4 Analysis of Water/Aqueous Samples by the Automated Cold Vapor Technique (AV) Preparation Method/Code (CW2)

10.2.4.1 Set up manifold.

10.2.4.2 Feed all the reagents through the system with acid wash solution (see Section 7.1.1.3) through the sample line, adjusting the heating bath to 105°C.

10.2.4.3 Turn on the Atomic Absorption (AA) Spectrophotometer, adjust instrument settings as recommended by the manufacturer, align absorption cell in light path for maximum transmittance and place heat lamp directly over absorption cell.

10.2.4.4 Arrange working mercury standards in sampler and start sampling. Complete loading of sample tray with unknown samples.

10.2.4.5 After the analysis is complete, put all lines except the H_2SO_4 line in reagent water to wash out system. After flushing, wash out the H_2SO_4 line. Also flush the coils in the high temperature heating bath by pumping stannous sulfate (see Section 7.1.1.4) through the sample lines followed by reagent water. This will prevent build-up of oxides of manganese.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Water/Aqueous by Automated Technique

11.1.1 Prepare a standard curve by plotting the instrumental response of processed standards against true concentration values. Use a linear regression equation to determine the concentration of field and Quality Control (QC) samples.

11.1.2 If samples were diluted for analysis, multiply the results from the linear regression by the dilution factor.

11.2 Water/Aqueous by Manual Technique

11.2.1 Determine the instrumental response of the unknown and determine the mercury value from the standard curve.

11.2.2 Calculate the mercury concentration in the sample by the formula:

EQ. 1 Aqueous Sample Concentration (Manual)

$$\text{Hg Concentration } (\mu g/L) = \frac{\mu g \text{ Hg, curve}}{\text{aliquot volume, mL}} \times \frac{1000 \text{ mL}}{1 \text{ L}}$$

11.3 Soil by Manual Technique

11.3.1 Measure the instrumental response of the unknown and determine the mercury value from the standard curve.

11.3.2 Calculate the mercury concentration in the sample by the formula:

EQ. 2 Soil Sample Concentration (Manual)

$$\text{Hg Concentration } (mg/kg) = \text{Hg } \mu g/g = \frac{C}{W \times S} \times (0.1L)$$

WHERE, C = Concentration from curve (µg/L)

W = Wet sample weight (g)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

11.4 Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation

To calculate the adjusted MDL or adjusted CRQL for water/aqueous samples, multiply the value of the MDL (µg/L) or CRQL (µg/L) by the Dilution Factor. Calculate the adjusted MDL or adjusted CRQL for soil samples as follows:

EQ. 3 Adjusted Soil MDL/Adjusted Soil CRQL Concentration

$$\text{Adjusted Concentration (dry wt.) (mg/kg)} = C \times \frac{W_M}{W_R} \times \frac{1}{S} \times DF$$

WHERE,

C	=	MDL or CRQL concentration (mg/kg)
W_M	=	Method required wet sample weight (g)
W_R	=	Reported wet sample weight (g)
S	=	% Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).
DF	=	Dilution Factor

12.0 QUALITY CONTROL

12.1 Initial Calibration Verification (ICV)

The ICV Standard shall be prepared in the same acid matrix as the samples and carried through the entire preparation and analysis procedure. If measurements exceed the control limits of 80% (low) and 120% (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. Information regarding the ICV shall be reported on Form IIA-IN.

12.2 Continuing Calibration Verification (CCV)

The CCV Standard shall be prepared by the analyst at a concentration equivalent to the mid-point of the calibration curve and carried through the entire preparation and analysis procedure. If the deviation of the CCV is greater than the control limits of 80% (low) and 120% (high), the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration verification shall be performed. Information regarding the CCV shall be reported on Form IIA-IN.

12.3 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

12.3.1 To verify linearity near the CRQL, the Contractor shall analyze a CRI at the beginning and end of each sample analysis run, but not before the ICV. In addition, the Contractor shall analyze and report the results for the CRI at a frequency of not less than once per 20 analytical samples[1] per analysis run. The CRI analysis shall be run immediately followed by the CCV and Continuing Calibration Blank (CCB) analyses. The CRI shall be prepared by spiking an aliquot of reagent water with mercury at the CRQL. The CRI shall be taken through the same process used to digest and analyze the associated samples.

12.3.2 CRI and percent recovery results shall be reported on Form IIB-IN. If the percent recovery falls outside the control limits of 70-130%, the CRI shall be re-analyzed immediately. If the result of the re-analysis falls within the control limits, no further corrective action is required. If the result of the re-analysis does not fall within the control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, or the CRI and associated samples redigested and analyzed.

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 while the preparation blank is used to monitor for possible contamination.

12.4.1 Initial and Continuing Calibration Blank (ICB/CCB)

The ICB and CCB are prepared with acids and reagent water and carried through the entire preparation and analysis procedure. If the absolute value of the calibration blank (ICB/CCB) result exceeds the CRQL (see Exhibit C), the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of the preceding 10 analytical samples or all analytical

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[1]As defined in Exhibit G, CRI is an analytical sample.

samples analyzed since the last compliant calibration blank shall be performed.

12.4.2 Preparation Blank (PB)

12.4.2.1 The PB shall contain all the reagents and in the same volumes as used in processing the samples. The PB 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 PB, 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 [2] 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. (see Form III-IN). Each Sample Data Package shall contain the results of all PB analyses associated with the samples in that SDG.

12.4.2.4 The PB is 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 the analyte in the associated samples shall be greater than or equal to 10 times the blank concentration. Otherwise, all samples associated with that blank, with the analyte concentration less than 10 times the blank concentration and above the CRQL, shall be redigested and re-analyzed with appropriate new Quality Control (QC). The only exception to this shall be an identified field blank. 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 and associated with the blank shall be redigested and re-analyzed with appropriate new QC.

12.4.2.4.4 The values for the PB shall be reported on Form III-IN.

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., water, soil) or for each SDG. [3] The sample

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[2]A group of samples prepared at the same time.

[3]USEPA may require additional spike sample analyses, upon USEPA Regional CLP Project Officer (CLP PO) request.

and its associated spike sample shall initially be run at the same dilution.

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 at 1 µg/L (water) or 0.5 mg/kg (soil). Adjustment shall be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values.

12.5.4 If the spike recovery is not at or within the limits of 75-125%, the data of all samples received and associated with that spike sample and determined by the same analytical method shall be flagged with the letter "N" on Forms IA-IN and VA-IN. An exception to this rule is granted when the sample concentration exceeds the spike added 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 In the instance where there is more than one spike sample per matrix, per method, per SDG, and 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 (%R) are calculated as follows:

EQ. 4 Spike Percent Recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

WHERE, SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

12.5.6 When sample concentration is less than the Method Detection Limit (MDL), use SR = 0 only for purposes of calculating percent recovery. The Spike Sample Results (SSRs), Sample Results (SRs), Spike Added (SA), and percent recovery (positive or negative) shall be reported on Form VA-IN.

12.5.7 The units used for reporting SSRs will be identical to those used for reporting sample results on Form IA-IN.

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., water, soil) or for each SDG. [4] Duplicates cannot be averaged for reporting on Form IA-IN. The

[4]USEPA may require additional duplicate sample analyses, upon USEPA Regional CLP PO request.

sample and its associated duplicate sample shall initially be run at the same dilution.

12.6.2 Duplicate sample analyses are 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)

D = Duplicate Result

12.6.3 The results of the duplicate sample analyses shall be reported on Form VI-IN. 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 of the CRQL value shall be entered in the "Control Limit" column on Form VI-IN 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 MDL, the RPD is not calculated on Form VI-IN. For solid sample or solid 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 and percent solids), 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 "*" on Forms IA-IN and VI-IN. 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 each element will be added to Form VI-IN at a later date based on these precision results.

12.7 Laboratory Control Sample (LCS) Analysis

12.7.1 A solid LCS (LCSS) shall be analyzed using the same sample preparations, analytical methods, and Quality Assurance (QA)/QC procedures employed for the EPA samples received.

12.7.2 The USEPA provided LCSS shall be prepared and analyzed using the procedures applied to the solid samples received (exception: percent solids determination not required). If the USEPA LCSS is unavailable, other USEPA QC Check samples or other certified materials may be used. In such a case, control limits for the LCSS must be documented and provided. One LCSS shall be prepared and

analyzed for every group of solid samples in a SDG, or for each batch of samples digested, whichever is more frequent.

12.7.3 All LCSS and percent recovery results will be reported on Form VII-IN. If the results for the LCSS fall outside the control limits established by USEPA, the analyses shall be terminated, the problem corrected, and the samples associated with that LCSS redigested and re-analyzed with appropriate new QC.

12.8 Method Detection Limit (MDL) Determination

12.8.1 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, and 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.

12.8.2 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.

12.8.3 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C.

12.8.4 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).

12.8.5 The MDL results shall be reported on Form IX-IN.

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 Government Printing Office. 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Not applicable.

EXHIBIT D - PART D

ANALYTICAL METHODS
FOR
TOTAL CYANIDE ANALYSIS

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Exhibit D - Analytical Methods for Total Cyanide Analysis

Table of Contents

Exhibit D - Analytical Methods for Total Cyanide Analysis

Table of Contents (Con't)

1.0 SCOPE AND APPLICATION

The analytical method that follows is designed to analyze various water types, sediment, sludge, and soil samples taken from hazardous waste sites, for total cyanide.

This analytical method includes the use of acid and heat to remove cyanide from the sample.

2.0 SUMMARY OF METHOD

2.1 Waters and Soils

2.1.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined colorimetrically.

2.1.2 In the colorimetric 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 for a complete list of definitions.

4.0 INTERFERENCES

Interferences are eliminated or reduced by using the distillation procedure.

4.1 Sulfides

Sulfides adversely affect the colorimetric 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 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.

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 Conventional Distillation of Water and Soils

6.1.1 Reflux distillation apparatus. The boiling flask should be of 1 Liter (L) size with an inlet tube and provision for condenser. The gas absorber may be a Fisher-Milligan scrubber.

6.1.2 Spectrophotometer suitable for measurements between 570 and 580 nanometers (nm) with a 1.0 centimeter (cm) cell or larger (for manual spectrophotometric method).

6.1.3 Automated analyzer instrumentation (for automated spectrophotometric method) including:

6.1.3.1 Sampler

6.1.3.2 Pump

6.1.3.3 Cyanide manifold

6.1.3.4 Colorimeter with 15 millimeters (mm) flowcells and 580 nm filters

6.1.3.5 Recorder

6.1.3.6 Data system (optional)

6.1.3.7 Glass or plastic tubes for the sampler

6.2 Midi Distillation of Water and Soils

6.2.1 Midi reflux distillation apparatus

6.2.2 Heating block - Capable of maintaining 125°C (±5°C).

6.2.3 Auto analyzer system with accessories:

6.2.3.1 Sampler

6.2.3.2 Pump

6.2.3.3 Cyanide cartridge

6.2.3.4 Colorimeter with 50 mm flowcells and 580 nm filter

6.2.3.5 Chart recorder or data system

6.2.4 Assorted volumetric glassware, pipets, and micropipets

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 D1193-77). Use this preparation for all reagents, standards, and dilutions of solutions.

7.1.2 Conventional Distillation and Preparation Reagents of Water and Soils

7.1.2.1 Sodium hydroxide solution, 1.25N - Dissolve 50 grams (g) of NaOH in reagent water, and dilute to 1 Liter (L) with reagent water. (Same Distillation and Preparation Reagent for Midi Distillation of Water and Soils.)

7.1.2.2 Cadmium carbonate - Powdered

7.1.2.3 Ascorbic acid - Crystals

7.1.2.4 Sulfuric acid - Concentrated

7.1.2.5 Hydrochloric acid (HCl) - Concentrated (specific gravity 1.19).

7.1.2.6 Magnesium chloride solution - Weigh 510 g of $MgCl_2 \cdot 6H_2O$ into a 1000 milliliter (mL) flask, dissolve, and dilute to 1 L with reagent water. (Same Distillation and Preparation Reagent for Midi Distillation of Water and Soils.)

7.1.3 Midi Distillation and Preparation Reagents of Water and Soils

7.1.3.1 Sodium hydroxide absorbing solution and sample wash solution, 0.25N - Dissolve 10.0 g NaOH in reagent water and dilute to 1 L.

7.1.3.2 Sulfuric acid, 50% (v/v) - Carefully add a portion of concentrated H_2SO_4 to an equal portion of reagent water.

7.1.4 Manual Spectrophotometric Reagents for Water and Soils

7.1.4.1 Acetate Buffer - Dissolve 410 g of $NaC_2H_3O_2 \cdot 3H_2O$ in 500 mL of reagent water. Add sufficient glacial acetic acid to adjust pH to 4.5 (approximately 500 mL).

7.1.4.2 Chloramine-T solution - Dissolve 1.0 g of white, water soluble chloramine-T in 100 mL of reagent water and refrigerate until ready to use. Prepare fresh weekly.

7.1.4.3 Color Reagent

7.1.4.3.1 Pyridine-barbituric acid reagent - Place 15 g of barbituric acid in a 250 mL volumetric flask and add just enough reagent water to wash the sides of the flask and wet the barbituric acid. Add 75 mL of pyridine and mix. Add 15 mL of HCl (specific gravity 1.19), mix, and cool to room temperature. Dilute to 250 mL with reagent water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.

7.1.5 Semi-Automated Spectrophotometric Reagents for Conventional and Midi Distillation of Water and Soils

7.1.5.1 Chloramine-T solution - Dissolve 0.40 g of chloramine-T in reagent water and dilute to 100 mL. Prepare fresh daily.

7.1.5.2 Acetate Buffer - Dissolve 410 g of $NaC_2H_3O_2 \cdot 3H_2O$ in 500 mL of reagent water. Add sufficient glacial acetic acid to adjust pH to 4.5 (approximately 500 mL).

7.1.5.3 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 HCl 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).

7.1.5.4 Sampler wash - Dissolve 10 g of NaOH in reagent water and dilute to 1 L. (For conventional distillation of water and soils only.)

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards 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 Reagents for Water and Soils

7.2.2.1.1 Stock cyanide solution - Dissolve 2.51 g of KCN and 2 g KOH in 1 L of reagent water. Standardize with 0.0192N $AgNO_3$.

7.2.2.1.2 Standard cyanide solution, intermediate - Dilute 50.0 mL of stock (1 mL = 1 milligram (mg) CN) to 1000 mL with reagent water.

7.2.2.1.3 Standard cyanide solution - Prepare fresh daily by diluting 100 mL of intermediate cyanide solution to 1000 mL with reagent water and store in a glass stoppered bottle. 1 mL = 5.0 micrograms (µg) CN [5.0 milligrams per Liter (mg/L)].

7.2.2.1.4 Sodium hydroxide solution, 0.25N - Dissolve 10 g of NaOH in reagent water and dilute to 1 L.

7.2.2.2 Stock Standard Reagents for Midi Distillation of Water and Soils

7.2.2.2.1 Stock cyanide solution, 1000 mg/L CN - Dissolve 2.51 g of KCN and 2.0 g KOH in reagent water and dilute 1 L. Standardize with 0.0192N $AgNO_3$.

7.2.2.2.2 Intermediate cyanide standard solution, 10 mg/L CN - Dilute 1.0 mL of stock cyanide solution (see Section 7.2.2.2.1) plus 20 mL of 1.25N NaOH solution (see Section 7.1.2.1) to 100 mL with reagent water. Prepare this solution at time of analysis.

7.2.2.2.3 Sodium hydroxide solution, 0.1N - Dissolve 4 g of NaOH in reagent water and dilute to 1 L.

7.2.3 Secondary Dilution Standards

7.2.3.1 Secondary Dilution Standards

Prepare secondary dilution standard solutions by diluting the appropriate volumes of stock standards with 0.25N NaOH. The final concentration of NaOH in all standards should be 0.25N.

7.2.4 Working Standards

7.2.4.1 Method Detection Limit (MDL) Solution

7.2.4.1.1 The MDL solution shall be at a concentration of 3 to 5 times the expected MDL.

7.2.4.2 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

7.2.4.2.1 The concentration of the CRI for cyanide shall be at the CRQL. Information regarding the CRI shall be reported on Form IIB-IN.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

8.1.1 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 (which has been pre-moistened with pH 4 acetate buffer solution) 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 or lead 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 a 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 gram (g) of ascorbic acid for each liter of sample volume. Preserve the sample with NaOH to pH greater than 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

8.2.1 The 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 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. The analyst should follow the instructions provided by the manufacturer of the particular instrument. It is the responsibility of the analyst to verify that 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 colorimeters. Set up the manifold and complete system per manufacturer's instructions. Allow the colorimeter 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 7.1.5) through reagent lines. Adjust the baseline using the appropriate control on the colorimeter. Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations [per 250 milliliter (mL)].

9.3 Spectrophotometric Instrument Calibration Procedure

9.3.1 Instruments shall be calibrated daily or once every 24 hours, and each time the instrument is set up. The instrument standardization date and time shall be included in the raw data.

9.3.2 The date and time of preparation and analysis shall be given in the raw data.

9.3.3 Calibration standards shall be prepared fresh daily or each time an analysis is to be made and discarded after use. Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range. One of the calibration standards shall be at the Contract Required Quantitation Limit (CRQL). The acceptance criteria for the initial calibration curve is a correlation coefficient greater than or equal to 0.995.

9.3.4 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 Only if the ICV Solution is not available from USEPA, or where a certified solution of the analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

9.4.3 The ICV shall be distilled. This means that an ICV must be distilled with each batch of samples analyzed and that the samples distilled with an ICV must be analyzed with that particular ICV.

9.4.4 The value for the ICV shall be reported on Form IIA-IN.

9.5 Continuing Calibration Verification (CCV)

9.5.1 To ensure calibration accuracy during each analysis run, one of the following standards is to be used for the CCV and shall be analyzed and reported at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The standard shall also be analyzed and reported at the beginning of the run and after the last analytical sample. The analyte concentration in the CCV standard shall be different than the concentration used for the ICV and shall be one of the following solutions at or near the mid-range level of the calibration curve:

- USEPA Solutions
- NIST Standards
- A Contractor-prepared standard solution

The same CCV standard shall be used throughout the analysis runs for a Sample Delivery Group (SDG) of samples received.

9.5.2 Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses, and other related operations that may affect the CCV measured result may not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it, as well as the difference in time between the CCV and the analytical sample immediately preceding it, may not exceed the lowest difference in time between any two consecutive analytical samples associated with the CCV.

9.5.3 Information regarding the CCV shall be reported on Form IIA-IN.

9.6 Initial and Continuing Calibration Blank (ICB/CCB)

A calibration blank shall be analyzed at the wavelength used for analysis immediately after every ICV and CCV, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. The blank shall be analyzed at the beginning of the run and after the last analytical sample.

NOTE: A CCB shall be analyzed immediately after the last CCV, and the last CCV shall be analyzed immediately after the last analytical sample of the run. The results for the calibration blanks shall be reported on Form III-IN.

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 Sample Management Office (SMO) to inform them of the problem. SMO will contact the Region for instructions. The 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 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 sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the Region. If all phases of the sample are amenable to analysis, the 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 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 Region's decision in the SDG Narrative.

10.1.3 Soil samples are not dried prior to analysis. A separate percent solids determination must be made in accordance with the procedure in Exhibit D - Introduction to Analytical Methods, Section 1.6.

10.1.4 Before preparation is initiated for an aqueous 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 (which has been pre-moistened with pH 4 acetate buffer solution). If the test strip turns black, the Contractor shall treat the total volume of sample with powdered cadmium carbonate or lead 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.

10.2 Water and Soil Preparation of Standards and Samples

10.2.1 Standards Preparation

10.2.1.1 It is not imperative that all standards be distilled in the same manner as the samples. At least one standard (mid-range) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within ±15% of the undistilled standards, the operator shall find and correct the cause of the apparent error before proceeding.

10.2.1.2 Standards for Manual Spectrophotometric Analysis of Water and Soil Samples

Prepare a minimum of three standards and a blank by pipetting suitable volumes of standard solution into 250 milliliter (mL) volumetric flasks.

NOTE: The concentration of one of the calibration standards shall be at the Contract Required Quantitation Limit (CRQL).

To each standard, add 50 mL of 1.25N NaOH and dilute to 250 mL with reagent water. The same method for color development (i.e., pyridine-barbituric acid or pyridine-pyrazolone) must be used for both the samples and standards. Standards must bracket the concentration of the samples. If dilution is required, use the blank solution.

10.2.1.3 Standards for Semi-Automated Spectrophotometric Analysis of Water and Soil Samples

Calibration standards - Prepare a blank and at least three calibration standards over the range of the analysis by pipetting suitable volumes of standard solution into volumetric flasks. One calibration standard must be at the CRQL. Add NaOH to each standard to bring the concentration of NaOH to 10 grams per Liter (g/L). Store at 4°C (±2°C).

10.2.1.4 Standards for Midi Distillation Preparation and Semi-Automated Spectrophotometric Analysis of Water and Soil Samples

Prepare a minimum of three standards and a blank by pipetting suitable volumes of standard solution into 50 mL volumetric flasks. Dilute standards to 50 mL with 0.25N NaOH.

NOTE: One calibration standard must be at the CRQL.

10.2.2 Water Samples Preparation (Distillation)

10.2.2.1 Preparation Method/Code (DW1)

10.2.2.1.1 Place 500 mL of sample in the 1 liter boiling flask. Add 50 mL of NaOH solution (see Section 7.1.2.1) to the absorbing tube

and dilute if necessary with reagent water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train.

10.2.2.1.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

NOTE: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to re-adjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

10.2.2.1.3 Slowly add 25 mL concentrated sulfuric acid (H_2 SO_4) (see Section 7.1.2.4) through the air inlet tube. Rinse the tube with reagent water and allow the airflow to mix the flask contents for three minutes. Pour 20 mL of magnesium chloride solution (see Section 7.1.2.6) into the air inlet and wash down with a stream of water.

10.2.2.1.4 Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

10.2.2.1.5 Drain the solution from the absorber into a 250 mL volumetric flask and bring up to volume with reagent water washings from the absorber tube.

NOTE: The distillation procedure results in a two-fold concentration of the sample.

10.2.3 Water Samples Preparation (Midi-Distillation)

10.2.3.1 Preparation Method/Code (DW2)

10.2.3.1.1 The procedure described here utilizes a midi distillation apparatus and requires a sample aliquot of 50 mL or less for aqueous samples.

10.2.3.1.2 Pipet 50 mL of sample, or an aliquot diluted to 50 mL, into the distillation flask along with 2 or 3 boiling chips.

10.2.3.1.3 Add 50 mL of 0.25N NaOH (see Section 7.1.3.1) to the gas absorbing impinger.

10.2.3.1.4 Connect the boiling flask, condenser, and absorber in the train. The excess cyanide trap contains 0.5N NaOH.

10.2.3.1.5 Turn on the vacuum and adjust the gang (Whitney) valves to give a flow of three bubbles per second from the impingers in each reaction vessel.

10.2.3.1.6 After five minutes of vacuum flow, inject 5 mL of 50% (v/v) H_2SO_4 (see Section 7.1.3.2) 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 magnesium chloride solution (see Section 7.1.2.6) through the top air inlet tube of the distillation head into the reaction flask. Excessive foaming from samples containing surfactants may be quelled by the addition of either another 2 mL of 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.

10.2.3.1.8 Turn on the heating block and set for 123-125°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 one and a half 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. The solutions must be analyzed for cyanide within the 12 day holding time specified in Section 8.3.

10.2.4 Soil Samples Preparation

10.2.4.1 Preparation Method/Code (DS1) (Distillation)

10.2.4.1.1 Accurately weigh a representative 1-5 gram (g) portion of wet sample and transfer it to a boiling flask. Add 500 mL of reagent water. Shake or stir the sample so that it is dispersed.

10.2.4.1.2 Add 50 mL of NaOH solution (see Section 7.1.2.1) to the absorbing tube and dilute if necessary with reagent water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber, and trap in the train.

10.2.4.1.3 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

NOTE: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to re-adjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

10.2.4.1.4 Slowly add 25 mL of concentrated H_2SO_4 (see Section 7.1.2.4) through the air inlet tube. Rinse the tube with reagent water and allow the airflow to mix the flask contents for 3 minutes. Pour 20 mL of magnesium chloride solution (see Section 7.1.2.6) into the air inlet and wash down with a stream of water.

10.2.4.1.5 Heat the solution to boiling, taking care to prevent the solution from backing up and overflowing into the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

10.2.4.1.6 Drain the solution from the absorber into a 250 mL volumetric flask and bring up to volume with reagent water washings from the absorber tube.

10.2.4.2 Preparation Method/Code (DS2) (Midi-Distillation)

10.2.4.2.1 The procedure described here utilizes a midi distillation apparatus and requires a sample aliquot of 1 gram for solid materials.

10.2.4.2.2 Weigh 1.0 g of sample (to the nearest 0.01 g) into the distillation flask and dilute to 50 mL with reagent water. Add 2 or 3 boiling chips.

10.2.4.2.3 Add 50 mL of 0.25N NaOH (see Section 7.1.3.1) to the gas absorbing impinger.

10.2.4.2.4 Connect the boiling flask, condenser, and absorber in the train. The excess cyanide trap contains 0.5N NaOH.

10.2.4.2.5 Turn on the vacuum and adjust the gang (Whitney) valves to give a flow of three bubbles per second from the impingers in each reaction vessel.

10.2.4.2.6 After five minutes of vacuum flow, inject 5 mL of 50% (v/v) H_2SO_4 (see Section 7.1.3.2) 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.

10.2.4.2.7 Add 2 mL of magnesium chloride solution (see Section 7.1.2.6) through the top air inlet tube of the distillation head into the reaction flask. Excessive foaming from samples containing surfactants may be quelled by the addition of either another 2 mL of 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.

10.2.4.2.8 Turn on the heating block and set for 123-125°C. Heat the solution to boiling, taking care to prevent solution backup by periodic adjustment of the vacuum flow.

10.2.4.2.9 After one and a half hours of refluxing, turn off the heat and continue the vacuum for an additional 15 minutes. The flasks should be cool at this time.

10.2.4.2.10 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. The solutions must be analyzed for cyanide within the 12 day holding time specified in Section 8.3.

10.2.5 Non-Distilled Analyses

10.2.5.1 Preparation Method/Code (NP1)

10.2.5.1.1 This code shall be used to report samples that are not distilled prior to analysis.

10.2.5.1.2 This Preparation Method/Code shall also be used to report the non-distilled Method Detection Limit (MDL). The concentration of this MDL shall be used to determine the appropriate concentration qualifier for the results of instrument QC analyses [except the distilled Initial Calibration Verification (ICV)].

10.3 Sample Analysis

10.3.1 Manual Spectrophotometric Determination

10.3.1.1 Allow all standards and samples to come to ambient room temperature prior to analysis. Withdraw 50 mL or less of the solution from the flask and transfer to a 100 mL volumetric flask. If less than 50 mL is taken, dilute to 50 mL with 0.25N sodium hydroxide solution (see Section 7.1.3.1). Add 1.0 mL of acetate buffer (see Section 7.1.4.1) and mix. The dilution factor must be reported on Form XIII-IN.

10.3.1.2 Add 2 mL of chloramine-T (see Section 7.1.4.2) and mix. After 1 to 2 minutes, add 5 mL of pyridine-barbituric acid solution (see Section 7.1.4.3.1) and mix. Dilute to mark with reagent water and mix again. Allow 8 minutes for color development then read absorbance between 570 and 580 nanometers (nm) in a 1 centimeter (cm) cell within 15 minutes.

10.3.2 Semi-Automated Spectrophotometric Determination of Distillates

10.3.2.1 Set up the manifold. Pump the reagents through the system until a steady baseline is obtained.

10.3.2.2 Place calibration standards, blanks, and control standards in the sampler tray, followed by distilled samples, distilled duplicates, distilled standards, distilled spikes, and distilled blanks. Allow all standards and samples to come to ambient room temperature prior to analysis.

10.3.2.3 When a steady reagent baseline is obtained and before starting the sampler, adjust the baseline using the appropriate knob on the colorimeter. Aspirate a calibration standard and adjust the colorimeter until the desired signal is obtained. Establish the baseline and proceed to analyze calibration standards, blanks, control standards, distilled samples, and distilled Quality Control (QC) samples.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Water/Aqueous Sample Calculation

11.1.1 For semi-automated colorimetric determination (Non-Midi-Distillation), measure the instrument response of the calibration standards and calculate a linear regression equation. Apply the equation to the samples and Quality Control (QC) samples to determine the cyanide concentration in the distillates. To determine the concentration of cyanide in the original sample, MULTIPLY THE RESULTS BY ONE-HALF (since the original volume was 500 milliliter (mL) and the distillate volume was 250 mL). Also correct for, and report on Form XIII-IN, any dilutions which were made before or after distillation.

11.1.2 For manual colorimetric determination, calculate the cyanide, in micrograms per Liter (µg/L), in the original sample as follows:

EQ. 1 Aqueous Sample Concentration (Manual)

$$\text{CN Concentration } (\mu g/L) = \frac{A \times 1000\ mL/L}{B} \times \frac{50\ mL}{C}$$

WHERE,

A	=	µg CN read from standard curve (per 250 mL)
B	=	mL of original sample for distillation (see Section 10.2.2.1.1)
C	=	mL taken for colorimetric analysis (see Section 10.3.1.1)
50 mL	=	volume of original sample aliquot (see Section 10.3.1.1)
1000 mL/L	=	conversion mL to L

The minimum value that can be substituted for A is the Method Detection Limit (MDL) value adjusted for volume.

11.2 Soil Sample Calculation

11.2.1 A separate determination of percent solids must be performed (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

11.2.2 The concentration of cyanide in the sample is determined as follows:

11.2.2.1 Manual Spectrophotometric

EQ. 2 Soil Sample Concentration (Manual)

$$\text{CN Concentration } (mg/kg) = \frac{A \times \frac{50\ mL}{B}}{C \times \frac{\%\ solids}{100}}$$

WHERE,

A	=	µg CN read from standard curve (per 250 mL).
B	=	mL of distillate taken for colorimetric determination (see Section 10.3.1.1).
C	=	wet weight of original sample in g (see Section 10.2.4.1.1).
50 mL	=	standard volume taken for colorimetric determination (see Section 10.3.1.1)
% solids	=	percent solids (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

11.2.2.2 Semi-Automated Spectrophotometric for Non-Midi-Distillates

If the semi-automated method is used, measure the peak heights of the calibration standards (visually or using a data system) and calculate a linear regression equation. Apply the equation to the samples and QC audits to determine the cyanide concentration in the distillates.

EQ. 3 Soil Sample Concentration (Semi-automated)

$$\text{CN Concentration (mg/kg)} = \frac{A \times .25}{C \times \frac{\% \text{ solids}}{100}}$$

WHERE,

A	=	µg/L determined from standard curve.
C	=	wet weight of original sample in g (see Section 10.2.4.1.1).
.25	=	conversion factor for distillate final volume (see Section 10.2.4.1.6).
% solids	=	percent solids (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

The minimum value that can be substituted for A is the MDL value.

11.3 Calculations for Midi Distillation of Waters and Soils

11.3.1 Calculations for Semi-automated Colorimetric Determination

11.3.1.1 Prepare a standard curve by plotting absorbance (peak heights, determined visually or using a data system) of standards (y) versus cyanide concentration values (total µg CN/L) (x). Perform a linear regression analysis.

11.3.1.2 Multiply all distilled values by the standardization value to correct for the stock cyanide solution not being exactly 1000 milligrams per Liter (mg/L) (see Section 7.2.2.2.1).

11.3.1.3 Using the regression analysis equation, calculate sample receiving solution concentrations from the calibration curve.

11.3.1.4 Calculate the cyanide of aqueous samples in µg/L of original sample, as follows:

EQ. 4 Aqueous Sample Concentration (Midi)

$$\text{CN Concentration } (\mu g/L) = \frac{A \times D \times F}{B}$$

WHERE,

A = µg/L CN of sample from regression analysis

B = volume of original sample for distillation (0.050 L) (see Section 10.2.3.1.2)

D = any dilution factor necessary to bracket sample value within standard values

F = sample receiving solution volume (0.050 L)

The minimum value that can be substituted for A is the MDL value.

11.3.1.5 Calculate the cyanide of solid samples in mg/kg of original sample, as follows:

11.3.1.5.1 A separate determination of percent solids must be performed (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

11.3.1.5.2 The concentration of cyanide in the sample is determined as follows:

EQ. 5 Soil Sample Concentration (Midi)

$$\text{CN Concentration } (mg/kg) = \frac{A \times D \times F}{B \times E}$$

WHERE,

A = µg/L CN of sample from regression analysis curve

B = wet weight of original sample (see Section 10.2.4.2.2)

D = any dilution factor necessary to bracket sample value within standard values

E = % solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)

F = sample receiving solution volume (0.050 L)

The minimum value that can be substituted for A is the MDL value.

11.4 Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation

To calculate the adjusted aqueous MDL or adjusted aqueous CRQL for the manual colorimetric method, multiply the MDL (µg/L) or CRQL (µg/L) by 0.25 and substitute the result for the "A" term in Equation 1. To calculate the adjusted aqueous MDL or adjusted aqueous CRQL for all other methods, follow the instructions in Section 11.1.1 or substitute

the MDL (µg/L) or CRQL (µg/L) for the "A" term in Equation 4, as appropriate.

The adjusted soil MDL or adjusted soil CRQL for all methods shall be calculated as follows:

EQ. 6 Adjusted Soil MDL/Adjusted Soil CRQL Concentration

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_M}{W_R} \times \frac{1}{S}$$

WHERE,

C	=	MDL or CRQL concentration (mg/kg)
W_M	=	minimum method required wet sample weight (g)
W_R	=	reported wet sample weight (g)
S	=	% Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

For the midi-distillation, multiply the adjusted concentration value (mg/kg) obtained in Equation 6 by any applicable 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 distilled. If measurements exceed the control limits of 85% (low) and 115% (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. Information regarding the ICV shall be reported on Form IIA-IN.

12.2 Continuing Calibration Verification (CCV)

The CCV standard shall be prepared by the analyst at a concentration equivalent to the mid-point of the calibration curve. If the deviation of the CCV is greater than the control limits of 85% (low) and 115% (high), the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration verification shall be performed. Information regarding the CCV shall be reported on Form IIA-IN.

12.3 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

12.3.1 To verify linearity near the CRQL, a standard at the CRQL (CRI) shall be prepared, in the same matrix as the calibration standards, and analyzed at the beginning and at the end of each sample analysis run, but not before the ICV. In addition, the Contractor shall analyze the CRI at a frequency of not less than once per 20 analytical samples[1] per analysis run. The CRI analysis shall be run immediately followed by the CCV and Continuing Calibration Blank (CCB) analyses. The CRI shall be prepared by spiking an aliquot of reagent water with cyanide to yield a concentration in the final solution equal to the CRQL.

12.3.2 CRI and percent recovery results shall be reported on Form IIB-IN. If the percent recovery falls outside the control limits of 70-130%, the CRI shall be re-analyzed immediately. If the result of the re-analysis falls within the control limits, no further corrective action is required. If the result of the re-analysis does not fall within the control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the CRI analyzed, and the samples associated with the CRI re-analyzed.

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 while the preparation blank is used to monitor for possible contamination.

12.4.1 Initial and Continuing Calibration Blank (ICB/CCB)

The ICB and CCB are prepared with reagents and reagent water. If the absolute value of the calibration blank (ICB/CCB) result exceeds the CRQL (see Exhibit C), the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of the preceding 10 analytical samples or all analytical

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[1]As defined in Exhibit G, CRI is an analytical sample.

samples analyzed since the last compliant calibration blank shall be performed.

12.4.2 Preparation Blank (PB)

12.4.2.1 The PB shall contain all the reagents and in the same volumes as used in processing the samples. The PB shall be carried through the complete procedure and contain the same concentration in the final solution as the sample solution used for analysis.

12.4.2.2 At least one PB, 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 [2] of samples distilled, 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 of samples to Preparation Blank two, etc. (see Form III-IN). Each Sample Data Package shall contain the results of all the PB analyses associated with the samples in that SDG.

12.4.2.4 The PB is 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 the analyte in the associated samples shall be greater than or equal to 10 times the blank concentration. Otherwise, all samples associated with the blank, with the analyte concentration less than 10 times the blank concentration and above the CRQL, shall be redistilled and re-analyzed with appropriate new QC. The only exception to this shall be an identified field blank. 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 associated with the blank and reported below 10 times CRQL shall be reprepared and re-analyzed with appropriate new QC.

The values for the preparation blank shall be reported on Form III-IN.

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 distillation and/or measurement methodology. The spike is added prior to any distillation steps. At least one spike sample analysis (matrix spike) shall be performed on each group of samples of a similar

[2] A group of samples prepared at the same time.

matrix type (i.e., water, soil) or for each SDG. [3] The sample and its associated spike sample shall initially be run at the same dilution.

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 spiking solution shall be added to yield a final concentration of 100 µg/L in the final sample solution prepared for analysis (i.e., post-distillation). The final volume of the sample after distillation shall be the basis for the amount of cyanide to be added as the spike. For instance, the full volume distillation procedure will require addition of 25 µg cyanide to the sample prior to distillation [based on the final distillate volume of 250 milliliter (mL)] to meet the specified spiking level; and the midi distillation procedure requires the addition of 5 µg of cyanide to the sample prior to distillation (based on the final distillate volume of 50 mL).

12.5.3.1 For soil samples, the final sample solution prepared for analysis (i.e., the distillate) shall contain cyanide spiked at a concentration of 100 µg/L regardless of the distillation procedure employed, or the amount of sample used for distillation. The final sample volume after distillation shall be used as the basis for the amount of cyanide to add as the spike. The units for reporting soil sample cyanide results shall be mg/kg. To convert from µg/L to mg/kg, the equation below shall be used:

EQ. 7 Conversion to mg/kg

$$\text{mg/kg} = \text{µg/L} \times \frac{\text{final distillate volume (L)}}{\text{sample weight (g)}}$$

12.5.4 If the spike recovery is not at or within the limits of 75-125%, the data of all samples received and associated with that spike sample and determined by the same analytical method shall be flagged with the letter "N" on Forms IA-IN and VA-IN. An exception to this rule is granted when the sample concentration exceeds the spike added 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 4 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 sample at 2 times the indigenous level or 2 times CRQL, whichever is greater. Results of the post-distillation spike shall be reported on Form VB-IN.

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[3]USEPA may require additional spike sample analyses, upon USEPA Regional CLP Project Officer (CLP PO) request.

12.5.6 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. 8 Spike Percent Recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

WHERE,

SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

12.5.7 When the sample concentration is less than the Method Detection Limit (MDL), use SR = 0 only for purposes of calculating percent recovery. The Spike Sample Results (SSRs), Sample Results (SRs), Spike Added (SA), and percent recovery (positive or negative) shall be reported on Form VA-IN.

12.5.8 The units used for reporting spike sample results will be identical to those used for reporting sample results on Form IA-IN.

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., water, soil) or for each SDG. [4] Duplicates cannot be averaged for reporting on Form IA-IN. The sample and its associated duplicate sample shall initially be run at the same dilution.

12.6.2 Duplicate sample analyses are 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. 9 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

WHERE,

RPD = Relative Percent Difference

S = Sample Result (original)

D = Duplicate Result

12.6.3 The results of the duplicate sample analyses shall be reported on Form VI-IN. A control limit of 20% for RPD shall be used for

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[4]USEPA may require additional duplicate sample analyses, upon USEPA Regional CLP PO request.

original and duplicate sample values greater than or equal to five times the CRQL (see Exhibit C). A control limit of the CRQL value shall be entered in the "Control Limit" column on Form VI-IN 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 MDL, the RPD is not calculated on Form VI-IN. For solid sample or solid 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 and percent solids), 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 "*" on Forms IA-IN and VI-IN. 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 each element will be added to Form VI-IN at a later date based on the precision results.

12.7 Laboratory Control Sample (LCS) Analysis

12.7.1 A solid LCS (LCSS) shall be analyzed using the same sample preparations, analytical methods, and Quality Assurance (QA)/QC procedures employed for the EPA samples received. For cyanide, a distilled ICV shall be used as the aqueous LCS (LCSW).

12.7.2 The USEPA provided LCSS shall be prepared and analyzed using each of the procedures applied to the solid samples received (exception: percent solids determination not required). If the USEPA LCSS is unavailable, other USEPA QC Check samples or other certified materials may be used. In such a case, the control limits for LCSS must be documented and provided. One LCSS shall be prepared and analyzed for every group of solid samples in a SDG, or for each batch of samples distilled, whichever is more frequent.

12.7.3 All LCSS and percent recovery results will be reported on Form VII-IN. If the results for the LCSS fall outside the control limits established by USEPA, the analyses shall be terminated, the problem corrected, and the samples associated with that LCSS reprepared and re-analyzed with appropriate new QC.

12.8 Method Detection Limit (MDL) Determination

12.8.1 Before any field samples are analyzed under this contract, the MDLs shall be determined for non-distilled analyses (Preparation Method/Code "NP1") and for each distillation procedure and instrument used, prior to the start of the contract analyses, and annually thereafter, and 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.

12.8.2 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 distillation procedure used and shall analyze these samples on each instrument used. The Contractor shall also analyze the non-distilled MDL samples on each instrument used.

12.8.3 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C.

12.8.4 The non-distilled MDL (Preparation Method/Code "NP1") shall be used to determine the appropriate concentration qualifier for the results of instrument QC analyses (except the distilled ICV).

12.8.5 The results of the MDL determination study shall be forwarded to the USEPA Regional CLP PO, Sample Management Office (SMO), and Quality Assurance Technical Support (QATS).

12.8.6 The MDL results shall be reported on Form IX-IN.

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 335.2. 1980.

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.

EXHIBIT E

CONTRACT LABORATORY PROGRAM QUALITY ASSURANCE MONITORING PLAN

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Exhibit E - Contract Laboratory Program Quality Assurance Monitoring Plan

Table of Contents

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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 Incentives/Sanctions

The Contractor may anticipate incentives by consistently providing the following: (1) high quality, technically sound data as stipulated by the ILM05.2 contract; (2) on-time or early delivery of the Sample Delivery Group (SDG) Cover Sheet; (3) above average Quarterly Blind (QB) Performance Evaluation (PE) sample scores; (4) diskettes that pass the initial Contract Compliance Screening (CCS) acceptance criteria; and (5) SDGs delivered on-time. 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 above, will earn a higher PSA score, thereby increasing the likelihood of receiving samples for analyses. 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); 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 ILM05.2 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 OERR Analytical Operations/Data Quality Center (AOC), Regional Data Users, Sample Management Office (SMO), and the Quality Assurance Technical Support (QATS) Laboratory. 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 OERR AOC Inorganic Program Manager (AOC 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 Management 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 raw data and required documentation for Regional review;
- Submit results of all analyzed laboratory evaluation samples, and adhere to corrective action procedures;
- Submit, upon request, instrument data tapes and applicable documentation for tape audits, including a copy of the Sample Data Package;
- 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 Verification of Method Detection Limits (MDLs)

The Contractor shall perform and report annual verification of MDLs by the method specified in Exhibit D, by type, matrix, and model for each instrument used on this contract, to 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 the MDLs shall meet the requirements specified in Exhibit C.

4.4 Quarterly Verification of Linear Ranges/Interelement Correction Factors

The Contractor shall perform and report quarterly verification of linear ranges by the method specified in Exhibit D, by type and model for each instrument used on this contract, to SMO, QATS, and the USEPA Regional CLP PO as specified in Exhibit B. The Contractor shall also report, as specified in Exhibit B, integration times. For Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) methods, the Contractor shall also report, as specified in Exhibit B, wavelengths used and 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. The exception is the CRI analysis, which may be re-analyzed once before corrective action is necessary.

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, linear dynamic range, and interference effects shall be established for each analyte on a particular instrument. All reported measurements shall be within the instrumental linear ranges. The Contractor shall maintain QC data confirming instrument performance and analytical results.

5.0 QUALITY ASSURANCE MANAGEMENT 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/quality1/qa_docs.html.

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 OERR Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (AOC 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 Management 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 Management 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 OERR AOC 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 Incentives/Sanctions

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 ILM05.2. Failure to comply with the requirements of this section may result in sanctions as described in the contract.

6.0 STANDARD OPERATING PERFORMANCE STANDARDS

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/qa_docs.html.

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) ILM05.2 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 OERR Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (AOC 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 explain 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; and
- Instrument error and editing flag descriptions and resulting corrective actions.

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;
- 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 OERR AOC 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 Exnibit E, Sectıon 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 maıntain 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 Incentives/Sanctions

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 ILM05.2. Failure to comply with the requirements specified herein may result in sanctions 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 Sample Data Package 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 Sample Data Package 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 4 business days to correct deficiencies and shall send all corrections to the 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 Incentives/Sanctions

7.4.1 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.

7.4.2 The Contractor shall correct deficiencies and resubmit the data within 4 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 ILM05.2. Correct resubmission of the data may also result in a reduction in overall sanctions. Specific details on incentives 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 sanctions as described in the contract.

8.0 ANALYTICAL PERFORMANCE STANDARDS REQUIREMENTS

8.1 Overview

USEPA will not supply analytical reference 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.

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. 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. One way this can be demonstrated is to prepare and analyze three solutions: a high standard, a low standard, and a standard at the target concentration (see Sections 8.3.2.1 and 8.3.2.2). The supplier must then demonstrate that the analytical results for the high standard and low standard are consistent with the difference in theoretical concentrations. This is done by the Student's t-test in Section 8.3.2.4. If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test in Section 8.3.2.5. Thus, the standard is certified to be within 10% of the target concentration using the equations in Section 8.3.2.6. If the procedure above is used, the supplier must document that the following have been achieved.

8.3.2.1 Two solutions of identical concentration shall be prepared independently from neat materials. An aliquot of the first solution shall be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration 10% greater than the target standard. This is called the "high standard". One further aliquot is taken from the second solution and diluted to a concentration 10% less than the target standard. This is called the "low standard".

8.3.2.2 Six replicate analyses of each standard (a total of 18 analyses) shall be performed in the following sequence: low standard; target standard; high standard; low standard; target standard; high standard; etc.

8.3.2.3 The mean and variance of the six results for each solution shall be calculated:

EQ. 2 Mean

$$\text{MEAN} = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6}$$

EQ. 3 Variance

$$\text{VARIANCE} = \frac{Y_1^2 + Y_2^2 + Y_3^2 + Y_4^2 + Y_5^2 + Y_6^2 - 6(\text{MEAN})^2}{5}$$

The values Y_1, Y_2, Y_3, ..., represent the results of the six analyses of each standard. The means of the low, target, and high standards are designated M_1, M_2, and M_3, respectively. The variances of the low, target, and high standards are designated V_1, V_2, and V_3, respectively. Additionally, a pooled variance, V_p, is calculated.

EQ. 4 Pooled Variance

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + \frac{V_3}{1.21}}{3}$$

If the square root of V_p is less than one percent of M_2, then $M_2^2/10,000$ is to be used as the value of V_p in all subsequent calculations.

8.3.2.4 The test statistic shall be calculated:

EQ. 5 Low and High Standard Test Statistic

$$\text{TEST STATISTIC} = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3} \right)^{0.5}}$$

If the test statistic exceeds 2.13, then the supplier has failed to demonstrate a 20% difference between the high and low standards. In such a case, the standards are not acceptable.

8.3.2.5 The test statistic shall be calculated:

EQ. 6 Target Standard Test Statistic

$$\text{TEST STATISTIC} = \frac{\left| M_2 - \left(\frac{M_1}{1.8} \right) - \left(\frac{M_3}{2.2} \right) \right|}{\left(\frac{V_p}{4} \right)^{0.5}}$$

If the test statistic exceeds 2.13, the supplier has failed to demonstrate that the target standard concentration is midway between the high and low standards. In such a case, the standards are not acceptable.

8.3.2.6 The 95% confidence intervals for the mean result of each standard shall be calculated:

EQ. 7 Low Standard Interval

$$\text{Interval for Low Standard} = M_1 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

EQ. 8 Target Standard Interval

$$\text{Interval for Target Standard} = M_2 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

EQ. 9 High Standard Interval

$$\text{Interval for High Standard} = M_3 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

8.3.2.6.1 These intervals shall not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate the 10% difference in concentrations. In such a case, the standards are not acceptable.

8.3.2.6.2 In any event, the Contractor is responsible for the quality of the standards employed for analyses under this contract.

8.4 Requesting Standards from the USEPA Standards Repository

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.

8.5 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.

8.5.1 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.

8.5.2 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.

8.5.3 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.

8.5.4 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.

8.6 Incentives/Sanctions

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 ILM05.2 Statement of Work (SOW). Failure to meet the requirements set forth in this section may result in sanctions 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 Incentives/Sanctions

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. 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 sanctions 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 OERR Analytical Operations/Data Quality Center (AOC). 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 OERR AOC, 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 The PE sample(s) may be scheduled with the Contractor as frequently as on a Sample Delivery Group (SDG)-by-SDG basis. The PE samples may be sent either by the Regional client or the USEPA OERR Analytical Operations/Data Quality Center (AOC). 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 OERR AOC 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 and quantitation. 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), and 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 The Contractor shall be notified by the USEPA Contracting Officer concerning agreement or disagreement with the proposed remedy for unacceptable performance.

11.2.8 A Remedial QB Audit is a unique analytical Case containing only QB samples. A Remedial QB Audit may be scheduled by the USEPA OERR AOC 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 turn-over of key personnel). Sections 11.2.2 through 11.2.7 apply to the Remedial QB Audit process.

11.3 Incentives/Sanctions

The Contractor shall analyze PE and QB samples with acceptable analytical results in accordance with the contractual requirements as described in this section. 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 sanctions 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) 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 Management 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 and raw data;
- Bench sheets and analytical logbook maintenance and review; and
- Review of the Contractor's sample analysis/data package inspection/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; 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 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 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 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 Incentives/Sanctions

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. 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 sanctions 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 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 all 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), Contract Required Quantitation Limits (CRQL) Check Standards (CRIs), 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 and Linear Range Analyses (LRS) which are used to set the MDL and LRV values for the year/quarter 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 three years after submission of the reconciled Complete SDG File. Electronic media shipped to the USEPA designated recipient must be fully usable by the recipient. Diskettes must be 3.5 inch, high density, 1.44 MB MS/DOS formatted and tapes must be either 4 mm or 8 mm. 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 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 Spectrophotometer (ICP-AES)/ICP - Mass Spectrophotometer (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 Sample Data Package.

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 Incentives/Sanctions

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 ILM05.2 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 sanctions 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 diskettes 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 OERR Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (AOC 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

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 CADRE 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.
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.

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)

Exhibit E: Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)	Quality Assurance Management Plan	As outlined in Exhibit E, Sections 5.1.1 and 5.1.2, a written QA Management Plan 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 Section E.2 of the ILM05.2 IFB and must be performed as stated.	The sample data package 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.	Regional validation and/or CADRE reports are generated for all data packages.

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)

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

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan (Con't)

Exhibit H: Data Dictionary and Format for Data Deliverables in Computer-Readable Format	Data Dictionary and Format	Performance standards are outlined in Exhibit H and Appendix A.	CCS in Exhibit E, Section 7.0, will be used to monitor electronic deliverables.
Appendix B: Modified Analysis	GFAA requirements are outlined in Appendix B, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Appendix B, Sections 9.0 through 11.0.	QA monitoring plan is outlined in Appendix B, Section 12.0, and Exhibit E.

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EXHIBIT F

CHAIN-OF-CUSTODY, DOCUMENT CONTROL
AND WRITTEN STANDARD OPERATING PROCEDURES

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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 include:

- 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 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 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.

2.4 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., ICP-AES (metals)] shall be included in the title.

2.5.3 When columns are used to organize information recorded on laboratory documents, the information recorded in the columns shall be identified in a column heading.

2.5.4 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.

2.5.5 The laboratory name shall be identified on preprinted laboratory documents.

2.5.6 Each laboratory document entry shall be dated with the month/day/year (e.g., 01/01/1999) and signed by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.

2.5.7 Notations on laboratory documents shall be recorded in ink.

2.5.8 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.

2.5.9 Unused portions of laboratory documents shall be lined-out.

2.5.10 Pages in bound and unbound logbooks shall be sequentially numbered.

2.5.11 Instrument-specific run logs shall be maintained to enable the reconstruction of run sequences.

2.5.12 Logbook entries shall be in chronological order.

2.5.13 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).

2.5.14 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).

2.5.15 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.

2.5.16 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:

- logbook pages;
- bench sheets;
- screening records;
- preparation records;
- repreparation records;
- analytical records;
- re-analysis 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 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 SMO 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 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., 01/01/1999);
- 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;
- 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.

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EXHIBIT G

GLOSSARY OF TERMS

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ABSORBANCE - A measure of the decrease in incident light passing through a sample into a detector. It is defined mathematically as:

Absorbance

$$A = -\log \frac{I}{Io}$$

WHERE,

I = Radiation intensity of a sample.
I_B = 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 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 (USEPA and non-USEPA), matrix spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, Interference Check Samples (ICSs), Contract Required Quantitation Limit (CRQL) Check Standards (CRIs), Laboratory Control Samples (LCSs), Performance Evaluation (PE) samples, Preparation Blanks (PBs), and Linear Range Samples (LRSs).

ANALYTICAL SEQUENCE - The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB. All sample analyses during the analytical sequence are subject to the QC protocols set forth in Exhibits D and E of this contract unless otherwise specified in the individual methods.

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.

ASTM - American Society for Testing and Materials. 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 individual definitions for types of blanks.

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.

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 every 10 analytical samples or every 2 hours, whichever is more frequent.

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 SMO Contractor.

CONTRACT LABORATORY PROGRAM (CLP) - Supports the 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 Operations/Data Quality Center (AOC) of the Office of Emergency and Remedial Response (OERR) of USEPA.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) CHECK STANDARD (CRI) - A single parameter or multi-parameter standard solution prepared at the CRQL and used to verify the instrument calibration at low levels.

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 - MM/DD/YYYY - Where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YYYY = 1998, 1999, 2000, 2001, etc.

DAY - Unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - An official record of the sample preparation (digestion).

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 a water/aqueous 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 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.

GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA) - A technique for the determination of analytes in which a sample aliquot is injected into a hollow graphite tube, which is then heated to atomize the analyte. The vapor absorbs light at wavelengths characteristic of the element(s) atoms present.

HOLDING TIME - 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 SPECTROSCOPY (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/charge 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 for the purpose of measuring absorbance, emission or concentration of an analyte. May also be referred to as exposure.

INSUFFICIENT QUANTITY - When there is not enough volume (water/aqueous 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 - 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 control sample of known composition. Laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the 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).

LINEAR RANGE, LINEAR DYNAMIC RANGE - The concentration range over which the instrument response remains linear.

MATRIX - The predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is either water/ aqueous or soil/sediment. 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 (water/aqueous or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

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.

NARRATIVE (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.

PERCENT DIFFERENCE (%D) - As used in this 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 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 D1193-77, "Standard Specification for Reagent Water".

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this 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 (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 SOW. A run begins with the instrument calibration and is to be completed within a 24-hour period.

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 Case of field samples received, or
- 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).

- 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 samples in one SDG, all water samples in another) at the discretion of the laboratory.

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.

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 or 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 - 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) - An 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 ICP-MS accuracy, resolution, and precision prior to calibration.

USEPA OERR AOC INORGANIC PROGRAM MANAGER (AOC PM) - The USEPA, OERR AOC Official who manages the CLP Inorganic Program.

USEPA REGIONAL CLP PROJECT OFFICER (CLP PO) - The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a 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 Sample Traffic Report/Chain of Custody Record.

WET WEIGHT - The weight of a sample aliquot including moisture (undried).

10% FREQUENCY - A frequency specification during an analytical sequence allowing for no more than 10 analytical samples between required calibration verification measurements, as specified by the contract SOW.

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EXHIBIT H

DATA DICTIONARY AND FORMAT
FOR DATA DELIVERABLES IN
COMPUTER-READABLE FORMAT

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Exhibit H - Data Dictionary and Format for Data Deliverables
in Computer-Readable Format

Table of Contents

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1.0 USEPA AGENCY STANDARD IMPLEMENTATION

1.1 Format Characteristics

The following constitutes an implementation of the USEPA Agency Standard for Electronic Data Transmission based upon analytical results and ancillary information required by the contract. All data generated by a single analysis are grouped together, and the groups are aggregated to produce files that report data from a Sample Delivery Group (SDG). Because this implementation is only a subset of the USEPA Agency Standard, some fields have been replaced by delimiters as place holders for non-Contract Laboratory Program (CLP) data elements.

1.1.1 This implementation includes detailed specifications for the required format of each record. The position in the record where each field is to be contained relevant to other fields is specified, as well as the maximum length of the field. Each field's required contents are specified as literal (contained in quotes), which must appear exactly as shown (without quotes), or as a variable for which format and/or descriptions are listed in the format/contents column. Options and examples are listed for most fields. For fields where more than three options are available, a list and description of options are supplied following the record descriptions. Fields are separated from each other by the delimiter "|" (ASCII 124). Fields that do not contain data should be zero length or a blank field (empty with no space or additional delimiters between the delimiters before and after the field) with the delimiter as a place holder. For the purposes of Section 9 of this Exhibit, wherever "blank" is given as an option under the "Format/Contents" column, it refers to a blank field as explained above.

1.1.2 Numeric fields may contain numeric digits, a decimal place, and a leading minus sign. A positive sign is assumed if no negative sign is entered in a numeric field and must not be entered into any numeric field. Values that exceed the maximum length allowed shall be reported to the maximum possible, maintaining the specified decimal place and maximum field length restrictions.

1.1.3 Requirements for significant figures and number of decimal places are specified in Exhibit B. The numeric field lengths are specified such that all possible numeric values can be written to the file. The size of the numeric field indicates the maximum number of digits, including a decimal place and negative sign, if appropriate, that can appear in the field at the same time. Therefore, the number reported may need to be rounded (using rounding rules described in Exhibit B) to fit into the field. The rounding must maintain the greatest significance possible providing the field length limitation. In addition, the rounded number that appears on the form, and therefore the field in the diskette file, must be used in any calculation that may result in other numbers reported on the same form or other forms in the SDG. Field lengths should only be as long as necessary to contain the data; packing with blanks is not allowed.

1.1.4 USEPA is currently developing a data delivery strategy that may be used as an alternative to the requirements stated in Exhibit H. This strategy's intent is to provide a neutral data delivery structure to the Contractor that will further facilitate the exchange of analytical information generated under this analytical protocol. The proposed strategy is intended to accommodate laboratories that generate data transmission files under multiple data formats. Upon implementation of this alternate electronic data delivery strategy by the USEPA and prior to submission of data in alternate format(s), the

Contractor must first demonstrate its ability to provide electronic data as stated in this Exhibit H and obtain written permission from the USEPA for the submission of data in alternate format(s). The Contractor will receive a written response to its request within 90 calendar days. However, until the implementation of this alternate electronic data delivery strategy by the USEPA, all electronic data deliverables must be provided as specified in this Exhibit H.

2.0 RECORD TYPES

2.1 Specifications

The USEPA Agency Standard consists of variable length ASCII records. Maximum field length specifications match the reporting requirements in Exhibit B. The last two bytes of each record must contain "carriage return" and "line feed", respectively.

2.1.1 This implementation consists of twelve record types that can be summarized in four groups, designated by the first record type in each group:

Type	Type ID	Contents
Run Header	10	Information pertinent to a group of samples processed in a continuous sequence; usually several per SDG
Sample Header	20	Sample identifying, qualifying, and linking information
Results Record	30	Analyte results and qualifications
Comments Record	90	Free form comments

2.1.2 All record types given are mandatory. Type 10, representing the analytical run, contains the instrument and run IDs which act as an identifying label for the run. All 10, 20, 30, and 90 series records following that record pertain to the same analytical run. Type 20, representing the sample, contains the EPA Sample ID which acts as an identifying label for the sample. The QC code indicates whether the data is from an environmental sample, calibration, or QC sample. All 20, 30, and 90 series records following that record pertain to the same sample. Type 30, representing an individual analyte, contains an identifier to identify the analyte. All 30 series records following that record pertain to the same analyte.

3.0 PRODUCTION RUNS

3.1 Specifications

A production run represents a "group" or "batch" of samples that are processed in a continuous sequence under relatively stable conditions. Specifically:

3.1.1 Calibration - All samples in a run use the same initial calibration data. For mercury analyses, samples prepared by a certain method must be analyzed with calibration and QC standards prepared by the same method. Therefore, all samples, calibration standards, and QC standards in a run must be associated with the same Preparation Code (Type 21 record).

3.1.2 Method number - Constant throughout a run.

3.1.3 Instrument conditions - Constant throughout a run. Results obtained on different instruments cannot be combined in one run.

3.1.4 Thus, each separate group of analyses on each instrument will consist of a separate production run, and must be reported in a separate file.

3.1.5 The run numbers in a Sample Delivery Group (SDG) must be unique; that is, there shall only be one Run Number "1", only one Run Number "2", etc. in an SDG.

3.1.6 In addition, later runs within a method for an analyte shall have a higher run number than earlier ones. For example, if arsenic is quantitated by the Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) method on 01/01/1999 beginning at 12:02 and arsenic is later quantitated by the ICP-AES method on 01/01/1999 beginning at 18:06, then the run beginning at 12:02 shall have a lower run number than the run beginning at 18:06.

3.2 Example

The following is an example of the sequence of record types in a production run.

10 Contains run header information. Occurs once per run.

16 Contains additional run header information. Occurs once per run.

20 Acts as a header for the following method and instrument parameters information. Occurs at least once per run with EPA sample number equal to "MDL". Analysis year, analysis month, analysis day equal the year, month and day the Method Detection Limit (MDL) was computed. Analyte count equals the number of the Type 30 records that follow.

21 Contains the Preparation Code (field #5) and the Preparation Date (fields #8, 9, 10) for the MDL. Occurs at least once per run with each Type 21 record preceded by the relevant Type 20 record and immediately followed by its related Type 30 record(s).

30 Contains the Analyte Identifier "C" (field #2), the Analyte CAS Number (field #3), the MDL Label "U" (field #20), and the MDL (field #21). Occurs once for each analyte used in the run.

20

21

30

20 Acts as a header for the following instrument parameter information. Occurs once per run with EPA sample number equal to "LRV". Analysis year, analysis month, analysis day equal the year, month and day the linear ranges were computed. Analyte count equals the number of Type 30, 32 and 34 groups that follow.

30 Contains only the Analyte CAS Number and the Analyte Identifier. Occurs once for each analyte used in the run.

32 Contains integration time information for the preceding analyte on the Type 30 record.

34 Contains the Contract Required Quantitation Limit (CRQL) and Linear Range information for the preceding analyte on the Type 30 record. There are as many consecutive Type 34 records as there are different wavelengths or masses used for the analyte identified on preceding Type 30.

30

32

34

20 Acts as a header for the following instrument parameter information. Occurs once per run with EPA sample number equal to "BCD". Analysis year, analysis month, analysis day equal the year, month and day the background correction factors were computed. Analyte count equals the number of the Type 30 and 35 groups that follow.

30 Contains only the Analyte CAS Number and the Analyte Identifier. Occurs once for each analyte used in the run.

35 Contains the background and interelement correction information for the preceding analyte on the Type 30 record. There are as many consecutive Type 35 records as there are interelement correction factors for the analyte identified on preceding Type 30.

30

35

20 Contains header information for sample and QC data.

21 Contains additional information for analytical and instrument QC samples. Will always be preceded by a Type 20 record.

22 Contains additional information for analytical samples. Will usually follow a Type 21 record.

30 Contains the sample level concentration, true or added value and QC value for each analyte. Occurs once for

each analytical result for the EPA sample number of the previous Type 20 record.

31 Reports any instrumental data necessary to obtain the result reported on the previous Type 30 record. Will always be preceded by a Type 30 or 31 record. For Inductively Couple Plasma - Mass Spectrometry (ICP-MS), there are as many Type 31 records as there are isotopes for the analyte identified on the preceding Type 30 record.

30 Values for the next analyte being measured.

31 Values for the next analyte being measured.

30

31

Type 30-31 record sequence continues as many times as the value of the ANALYTE COUNT on the previous Type 20 record.

20 Next Sample Header record - The following applies to the next sample data.

21

22

30

31

30

31 etc.

20

21

22

30

31 etc.

4.0 RECORD SEQUENCE

4.1 Specifications

A Run Header (Type 10) record must be present as the first record in the file (run). Further occurrences of the Type 10 record in the file are not allowed.

4.1.1 A Type 16 record must immediately follow the Type 10 record. Further occurrences of the Type 16 record in the file are not allowed.

4.1.2 The first Type 20 records with EPA sample numbers MDL, LRV, and BCD are headers for the run-wide method and instrument parameters.

4.1.3 The first Type 20 record of the Type 21, 30 group is a header for the annually determined Method Detection Limits (MDLs) and must immediately follow the Type 16 record. A Type 20 record of the Type 21, 30 group must be present for each MDL reported in the run. For ICP-AES, ICP-MS, and cyanide analyses, an MDL associated with Preparation Code "NP1" must be present in each run. This MDL shall be used in the qualification of the data reported for non-prepared samples and instrument QC analyses (except the distilled Initial Calibration Verification (ICV) standard for cyanide).

4.1.4 The next Type 20 record of the Type 30, 32, 34 group is a header for the Linear Range Values (LRVs) and must immediately follow the last Type 30 record of the Type 21, 30 group that pertains to the MDL. The linear range values for all methods except the Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) methods are the analytically determined concentrations of the highest instrument calibration standards that are used in the generation of the calibration curve at the beginning of every run. The linear range values for the ICP-AES and ICP-MS methods are the quarterly determined values that are reported on Form XI-IN of the hardcopy.

4.1.5 The next Type 20 record of the Type 30, 35 group is a header for the ICP-AES Background Correction Data (BCD) and must immediately follow the last Type 34 record of the Type 30, 32, 34 group that pertains to the linear range values. This Type 20 record is not required for methods MS, AV, CV, CA, AS and C (i.e., ICP-MS, mercury, and cyanide analyses).

4.1.6 These are the only occurrences of the Type 20 records that do not relate to actual analyses in the run. Therefore, the only fields that are not blank in these occurrences of the Type 20 record are the RECORD TYPE ("20"); EPA SAMPLE NUMBER ("MDL", "LRV" and "BCD"); Analysis Year/Year Computed, Analysis Month/Month Computed, Analysis Day/Day Computed ("YYYY", "MM", "DD"); and ANALYTE COUNT.

4.1.7 A minimum of one Type 30 record must immediately follow the Type 21 record of the Type 21, 30 group with EPA sample number MDL, and the total number of Type 30 records must be equivalent to the ANALYTE COUNT on the Type 20 record.

4.1.8 A minimum of one Type 30, 32, 34 group with EPA sample number LRV must immediately follow the Type 20 record which is preceded by the last Type 30 record of the final Type 21, 30 group. The information in each Type 30, 32, 34 group must pertain to one and only one analyte. The number of Type 30, 32, 34 groups must be equivalent to the ANALYTE COUNT on the Type 20 record.

4.1.9 A minimum of one Type 30, 35 group with EPA sample number BCD must immediately follow the Type 20 record for background correction data (if required). This Type 20 is preceded by the last Type 34 record of the final Type 30, 32, 34 group. The information in each Type 30, 35 group must pertain to one and only one analyte. The number of Type 30, 35 groups must be equivalent to the ANALYTE COUNT on the Type 20 record.

4.1.10 The Type 20 record that relates to the analysis of the first instrument calibration standard must immediately follow the last Type 30, 35 group for ICP-AES, or the last Type 30, 32, 34 group for mercury and cyanide analyses. For ICP-MS, the Type 20 record for the first instrument tune standard analysis must immediately follow the last Type 30, 32, 34 group and the Type 20 record for the first instrument calibration standard must immediately follow the last 30, 31 group from the last tune standard analyzed. After the appearance of these Type 20 records in the file, further occurrences of the Type 32, 34 and 35 records in that file are not allowed.

4.1.11 Each environmental sample, calibration, or Quality Control (QC) sample is represented by a group composed of Type 20, 21, and 22 records, which hold sample level identifying information, followed by a minimum of one group composed of Type 30 and 31 records for each analyte. The Type 20 record holds a count for the number of analytes being used to determine results. The ANALYTE COUNTER must have a value equivalent to the number of Type 30 groups associated with each Type 20 record.

4.1.12 Except for the first Type 20 records (EPA sample numbers MDL, LRV, BCD) for method ICP-AES and the first two Type 20 records (EPA sample numbers MDL, LRV) for the methods for ICP-MS, mercury and cyanide analyses, all Type 20 records should occur in the order of sample analysis.

4.1.13 Type 90 comment records may be defined to occupy any position except before the Type 10 (header) record. Comments pertaining to the whole run such as ones on Cover Page must appear before the first Type 20 record. Comments pertaining to a particular sample such as ones on Forms IA-IN and IB-IN must appear after the Type 20 record for that sample, but before the first Type 30 record associated with that sample. Comments pertaining to a particular analyte must appear after the Type 30 record of that analyte, but before the Type 30 record of the following analyte.

4.1.14 The Type 92 record which contains the sample associated data that is reported at the bottom of Forms IA-IN and IB-IN must appear anywhere after the Type 22 record for that EPA Field Sample, but before the Type 20 record of the next sample.

5.0 FILE/RECORD INTEGRITY

All record types must contain the following check fields to ensure file and record integrity:

Record Position	Field Length	Field Contents	Remarks
First Field	2	Record type or identifier	"10" or as appropriate
Last Field	5	Record sequence number	00000-99999, repeated as necessary
	4	Record checksum[1]	Four hexadecimal digits
	2	Must contain CR and LF	

6.0 DATES AND TIMES

Date or time-of-day information consists of successive groups of digits, each separated by delimiters. Dates are given in the order YYYY MM DD, and times as HH MM. All hours must be given as 00 to 23 using a 24 hour clock and must be local time. All days shall be given as 01 to 31. All months shall be given as 01 to 12 (e.g., 01 is January, 02 is February). -

7.0 MULTIPLE VOLUME DATA

There is no requirement under this format that all the data from an entire Sample Delivery Group (SDG) fit onto a single diskette. However, each single production run must fit onto a single diskette if possible. If that is not possible, then it is necessary that all files start with a Type 10 record, and that the multiple Type 10 records for each file of the same production run be identical. Information for a single sample may not be split between files.

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[1]The checksum is the sum of the ASCII representation of the data on the record up to the Record Sequence Number (not including the Record Sequence Number), plus the checksum of the previous record. The sum is taken modulo 65536 (2^{16}) and is represented as four hexadecimal digits (i.e., the remainder of the sum divided by 65536 represented as four hexadecimal digits).

8.0 DELIVERABLE

8.1 Requirements

The file shall be submitted on IBM-compatible, 3.5 inch, high density 1.44 MB diskettes. The diskettes shall be formatted and recorded using DOS/Windows Operating Systems. The diskettes shall contain all information relevant to one and only one Sample Delivery Group (SDG). An alternative means of electronic transmission may be utilized if approved in advance by USEPA.

8.1.1 USEPA Agency Standard data from an entire SDG may not fit onto a single diskette. If a single production run is being split onto multiple diskettes, then all files shall start with a Type 10 record, and the multiple Type 10 records for each file of the same production run shall be identical. Do not split the data from a single sample onto multiple diskettes.

8.1.2 Information on the diskette **must correspond** to information submitted in the hardcopy raw data package and on the hardcopy raw data package forms. Unused records shall not be included on the diskettes. If the information submitted in the hardcopy data package forms is changed, the information in the electronic file (e.g., diskette) shall be changed accordingly, and a complete electronic deliverable containing all the information for the SDG shall be resubmitted along with the hardcopy at no additional cost to USEPA.

8.1.3 Each diskette shall be identified with an external label containing (in this order) the following information:

Disk Density
File Name(s)
Laboratory Name (optional)
Laboratory Code
Contract Number
Case Number/SDG
NRAS Number (where applicable)
Initial Submission or Resubmission (as applicable) and Date

8.1.4 The format for File Name shall be XXXXXX.I01 to XXXXXX.I99, where XXXXXX is the SDG identifier, I designates inorganics, and 01 through 99 is the file number.

8.1.5 Dimensions of the label must be in the range of 2-1/2" to 2-3/4" long by 2" to 2-1/8" wide for a 3-1/2" diskette.

9.0 RECORD LISTING

The following section provides information for the usage of each of the record types. Where specified, labels indicate the nature of the value(s) that follow on that record. If the value(s) will not be reported, the label shall be omitted. Listed below is every record type required to report data from a single Sample Delivery Group (SDG).

9.1 Production Run First Header Record (Type 10)

Use: Each production run will start with a Record Type 10.

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"10"
1	Delimiter	|
4	ANALYSIS START YEAR	YYYY
1	Delimiter	|
2	ANALYSIS START MONTH	MM
1	Delimiter	|
2	ANALYSIS START DAY	DD
1	Delimiter	|
2	ANALYSIS START HOUR	HH
1	Delimiter	|
2	ANALYSIS START MINUTE	MM
1	Delimiter	|
5	METHOD TYPE	CHARACTER [2]
1	Delimiter	|
8	METHOD NUMBER	"ILM05.2" (SOW)
1	Delimiter	|
3	MANAGER'S INITIALS	CHARACTER
1	Delimiter	|
6	LAB CODE	CHARACTER
4	Delimiter	||||
11	CONTRACT NUMBER	CHARACTER
1	Delimiter	|
10	INSTRUMENT ID	CHARACTER
2	Delimiter	||
25	LABORATORY NAME	CHARACTER
1	Delimiter	|
2	RUN NUMBER	NUMERIC [3]
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

---

[2]Analysis Method Types are:
"P" for ICP-AES
"MS" for ICP-MS
"CV" for Manual Cold Vapor AA
"AV" for Automated Cold Vapor AA
"AS" for Semi-Automated Spectrophotometric
"C" for Manual Spectrophotometric

[3]Run number values are 01 through 99. Each production run will be assigned a unique Run Number. Run Numbers are to be assigned sequentially beginning with 01 and will equal the number of production runs.

9.2 Production Run Second Header Record (Type 16)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"16"
1	Delimiter	|
4	ANALYSIS END YEAR	YYYY
1	Delimiter	|
2	ANALYSIS END MONTH	MM
1	Delimiter	|
2	ANALYSIS END DAY	DD
1	Delimiter	|
2	ANALYSIS END HOUR	HH
1	Delimiter	|
2	ANALYSIS END MINUTE	MM
1	Delimiter	|
1	AUTO-SAMPLER USED	"Y" or "N" [4]
1	Delimiter	|
1	INTERELEMENT CORRECTIONS APPLIED	"Y" or "N" [5]
1	Delimiter	|
1	BACKGROUND CORRECTIONS APPLIED	"Y" or "N" [5]
1	Delimiter	|
1	RAW DATA GENERATED	"Y" or "N" or "B" [6]
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[4]Enter "Y" if an auto-sampler is used with equal time and intervals between analysis.

[5]These are the answers to the first two questions on the Cover Page of the hardcopy deliverable. "Y" equals "YES", and "N" equals "NO".

[6]This is the answer to the third question on the Cover Page of the hardcopy deliverable. "Y" equals "YES", "B" equals BLANK, and "N" equals "NO".

9.3 Mandatory Sample Header Data Record (Type 20)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD	"20"
1	Delimiter	|
2	REGION	NUMERIC
1	Delimiter	|
12	EPA SAMPLE NUMBER	CHARACTER [7]
1	Delimiter	|
5	MATRIX	CHARACTER[8]
1	Delimiter	|
3	QC CODE	CHARACTER
1	Delimiter	|
3	SAMPLE QUALIFIER	CHARACTER
1	Delimiter	|
5	CASE NUMBER	CHARACTER
1	Delimiter	|
6	SDG NUMBER	CHARACTER
1	Delimiter	|
4	ANALYSIS YEAR/YEAR COMPUTED	YYYY
1	Delimiter	|
2	ANALYSIS MONTH/MONTH COMPUTED	MM
1	Delimiter	|
2	ANALYSIS DAY/DAY COMPUTED	DD
1	Delimiter	|
2	ANALYSIS HOUR	HH
1	Delimiter	|
2	ANALYSIS MINUTE	MM
2	Delimiter	||
2	SAMPLE WT/VOL UNITS	"G"/"ML"[9]
1	Delimiter	|
5	SAMPLE WT/VOL	NUMERIC[10]
1	Delimiter	|
3	ANALYTE COUNT	NUMERIC
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[7]EPA Sample Number as it appears on Form XIII-IN of the hardcopy deliverable except for the first Type 20 records. The first Type 20 record must have an EPA sample number of "MDL"; after all Type 20 records with an EPA sample number of "MDL", the next Type 20 record must have an EPA sample number of "LRV"; for ICP-AES, the Type 20 record following the "LRV" must have an EPA sample number of "BCD".

[8]For matrix, "1" equals "WATER" and "F" equals "SOIL". A matrix identifier ("1" or "F") is required for all EPA sample numbers except "BCD".

[9]"G" equals grams and "ML" equals milliliters.

[10]This is the size of the sample at the beginning of the digestion procedure.

9.3.1 SAMPLE QC CODES LISTING FOR TYPE 20

NOTE: These QC codes appear in the QC code field on the Type 20 record (R20F5). They are used to indicate the type of data that is being reported.

QCC	Name	Definition
LRB	LABORATORY (REAGENT) BLANK	The Preparation Blank (see Exhibit G).
LCB	LABORATORY CALIBRATION BLANK	The Continuing Calibration Blank (CCB) (see Exhibit G).
LIB	LABORATORY INITIAL BLANK	The Initial Calibration Blank (ICB) (see Exhibit G).
LCM	LABORATORY CONTROL SOLUTION	The Laboratory Control Sample (LCS) (see Exhibit G).
LD2	LABORATORY DUPLICATE SECOND MEMBER	This is the second aliquot and is identified as "D" on Form VI-IN of the hardcopy.
LVM	LABORATORY CALIBRATION VERIFICATION SOLUTION	These values are identified as "Initial Calibration Verification" (ICV) on Form IIA-IN of hardcopy.
LVC	LABORATORY CONTINUING CALIBRATION VERIFICATION	These values are identified as "Continuing Calibration Verification" (CCV) on Form IIA-IN of hardcopy.
LVD	LABORATORY DISTILLED VERIFICATION SOLUTION	These values are the "distilled ICV" results for cyanide. Refer to Exhibit D, Section 12.7.1 for cyanide.
LSF	LABORATORY SPIKED SAMPLE - FINAL VALUES	These are the "Spiked Sample Result (SSR)" values of Form VA-IN of hardcopy.
LDO	LABORATORY DILUTED SAMPLE BACKGROUND (ORIGINAL) VALUES	These values are the "Initial Sample Result (I)" values on Form VIII-IN of hardcopy.
LDF	LABORATORY DILUTED SAMPLE - FINAL VALUES	These are the "Serial Dilution Result(S)" values Form VIII-IN of hardcopy.

PDO	POST-DIGESTION SPIKE BACKGROUND (ORIGINAL) VALUES	This value is identified as "Sample Result" (SR) on Form VB-IN of hardcopy.
PDF	POST-DIGESTION SPIKE BACKGROUND (FINAL) VALUES	This value is identified as "Spiked Sample Result" (SSR) on Form VB-IN of hardcopy.
LPC	CRQL CHECK STANDARD	Laboratory Performance Check Solution for analysis methods P, MS, CV, AV, AS, and C (EPA sample number is CRI##). These results are reported on Form IIB-IN of hardcopy.
LSA	LABORATORY INTERFERENCE CHECK SOLUTION A	The results of this solution analysis (EPA sample number is ICSA##) are reported on Forms IVA and IVB-IN of hardcopy.
LSB	LABORATORY INTERFERENCE CHECK SOLUTION AB	The results of this solution analysis (EPA sample number is ICSAB##) are reported on Forms IVA and IVB-IN of hardcopy.
LTS	LABORATORY TUNE SAMPLE	The results of these solution analyses are reported on Form XIV-IN of hardcopy.
FRB	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.
FRM	PERFORMANCE EVALUATION (PE) SAMPLE	This is a sample of known composition provided by USEPA for Contractor analysis and is used to evaluate Contractor performance.
FLD	FIELD SAMPLE	This is the sample that is identified by a unique EPA sample number on the Traffic Report/Chain of Custody Record.
ZZQ	NON-SDG SAMPLE	This is any sample that is analyzed and is not part of the SDG (EPA sample number is ZZZZZZ).
STB	CALIBRATION STANDARD	This is the instrument calibration Blank Standard (EPA sample number is S0).

STC	CALIBRATION STANDARD	This is the instrument calibration CRQL Standard (EPA sample number is Sx where x is the CRQL value of the analyte).
STD	CALIBRATION STANDARD	This is the instrument calibration standard other than the Blank Standard or the CRQL Standard (EPA sample number is S).
STM	MIDRANGE STANDARD	This is the distilled cyanide Mid-range Standard (EPA sample number is MIDRANGE##). Refer to Exhibit D, Section 10.2.1.1, for cyanide.
STR	RESLOPE SAMPLE	This is the resloping that is permitted for mercury analysis (EPA sample number is RESLOPE##). Refer to Exhibit D, Section 9.1.5, for mercury.
STL	BASELINE SAMPLE	This is the baseline correction that is permitted for mercury analysis (EPA sample number is BASELINE##). Refer to Exhibit D, Section 9.1.5, for mercury.
MDQ	METHOD DETECTION LIMIT	These are the annually determined analyte detection limits that are reported on Form IX-IN of hardcopy. (EPA sample number is MDL).
LRQ	LINEAR RANGE VALUE	These are the quarterly determined values for ICP-AES and ICP-MS methods that are reported on Form XI-IN of hardcopy. For all other methods, these are the analytically determined concentrations of the highest instrument calibration standards that are used in the generation of the calibration curve at the beginning of every run. (EPA sample number is LRV).
BCQ	BACKGROUND CORRECTION	These are the ICP-AES annually determined interelement correction factors that are reported on Forms XA and XB-IN of hardcopy. (EPA sample number is BCD).

NOTE: All field samples that are reported on the Traffic Report/Chain of Custody Record shall contain the QC code "FLD" in Record Type 20

Field Number 5 (R20F5) except when "FLD" is superseded by "FRB" (Field Blank Sample), "FRM" (PE Sample).

For Matrix Spike and Duplicate sample analysis (Forms VA-IN and VI-IN of hardcopy), the "Sample" result shall contain the QC code "FLD" in R20F5, the "Spiked Sample Result" shall contain the QC Code "LSF" in R20F5, and the "Duplicate" result shall contain the QC code "LD2" in R20F5.

9.4 Sample Header Record (Type 21)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"21"
2	Delimiter	||
3	LEVEL	"LOW"/"MED"
2	Delimiter	||
3	PREPARATION CODE	CHARACTER[11]
1	Delimiter	|
6	NRAS NUMBER	CHARACTER
1	Delimiter	|
14	LAB SAMPLE ID	CHARACTER
1	Delimiter	|
4	PREPARATION YEAR	YYYY
1	Delimiter	|
2	PREPARATION MONTH	MM
1	Delimiter	|
2	PREPARATION DAY	DD
2	Delimiter	||
4	YEAR RECEIVED	YYYY
1	Delimiter	|
2	MONTH RECEIVED	MM

[11]Preparation Codes: A Preparation Code is required for all EPA sample numbers except "LRV", "BCD", and "TUNE##".
"HW1" - Hotplate/Block digestion for ICP-AES analysis of water samples.
"HW2" - Hotplate/Block digestion for ICP-MS analysis of water samples.
"MW1" - Microwave digestion for ICP-AES analysis of water samples.
"MW2" - Microwave digestion for ICP-AES analysis of water samples.
"HS1" - Hotplate/Block digestion for ICP-AES analysis of soil samples.
"HS2" - Hotplate/Block digestion for ICP-AES analysis of soil samples.
"MS1" - Microwave digestion for ICP-AES analysis of soil samples.
"CW1" - Preparation for the Manual Cold Vapor AA analysis of water samples.
"CS1" - Preparation for the Manual Cold Vapor AA analysis of soil samples.
"CW2" - Preparation for the Automated Cold Vapor analysis of water samples.
"DW1" - Distillation for the manual and semi-automated spectrophotometric analysis of water samples.
"DW2" - Midi-distillation for the semi-automated spectrophotometric analysis of water samples.
"DS1" - Distillation for the manual and semi-automated spectrophotometric analysis of soil samples.
"DS2" - Midi-distillation for the semi-automated spectrophotometric analysis of soil samples.
"NP1" - No preparation.

Sample Header Record (Type 21) (Con't)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
1	Delimiter	|
2	DAY RECEIVED	DD
1	Delimiter	|
9	SOLUTION SOURCE	CHARACTER[12]
1	Delimiter	|
8	INJECTION/ALIQUOT VOLUME	NUMERIC[13]
1	Delimiter	|
2	PREPARATION START HOUR	HH[14]
1	Delimiter	|
2	PREPARATION START MINUTE	MM[14]
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[12]This is the source of the solutions that are reported on Inorganic Forms IIA-IN, IIB-IN, IV-IN, and VII-IN of the hardcopy (ICV, CCV, CRI, ICS, and LCS), and the source of the instrument calibration standards.

[13]This is the portion of the sample that is injected into the instrument excitation system for the purpose of measuring the absorbence, emission, or concentration of an analyte.

[14]This is the time at which the preparation is started. It is used to differentiate between different batches on the same day.

9.5 Associated Injection and Counter Record (Type 22)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"22"
8	Delimiter	||||||||
5	VOLUME ADJUSTMENT FACTOR	NUMERIC[15]
2	Delimiter	||
8	FINAL VOLUME	NUMERIC[16]
1	Delimiter	|
8	DILUTION FACTOR	NUMERIC
3	Delimiter	|||
5	PERCENT SOLIDS	NUMERIC
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[15]This field is used to report any additional volume adjustments in the preparation method. As an example, the factor of 1.25 that results from the chloride interference volume adjustment in Preparation Method/Code HW2.

[16]This is the final volume that is currently reported on Form XII-IN of the hardcopy.

9.6 Results Data Record (Type 30)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"30"
1	Delimiter	|
1	ANALYTE IDENTIFIER	"C"[17]
1	Delimiter	|
9	ANALYTE CAS NUMBER	CHARACTER[18]
2	Delimiter	||
5	CONCENTRATION UNITS	"UG/L"/"MG/KG"
1	Delimiter	|
3	CONCENTRATION QUALIFIER	CHARACTER[19]
1	Delimiter	|
15	CONCENTRATION	NUMERIC [20,21]
1	Delimiter	|
1	VALUE DESCRIPTOR	"T"/"F"[22]
1	Delimiter	|
10	AMOUNT ADDED OR TRUE VALUE	NUMERIC
1	Delimiter	|
1	QC VALUE DESCRIPTOR, P	"P"[23]
1	Delimiter	|
10	QC VALUE	NUMERIC
1	Delimiter	|||
1	QC VALUE DESCRIPTOR, L	"L"[23]
1	Delimiter	|
10	QC VALUE	NUMERIC
1	Delimiter	|
1	MATRIX SPIKE QC LIMIT QUALIFIER	"N"[24]
1	Delimiter	|
10	QC LOWER LIMIT	NUMERIC [25]
1	Delimiter	|
10	QC UPPER LIMIT	NUMERIC [25]
1	Delimiter	|
1	QC LIMIT QUALIFIER	"*"/"E"[26]
1	Delimiter	|
1	MDL LABEL	"U"
1	Delimiter	|
10	MDL	NUMERIC[27]
2	Delimiter	||
15	RAW DATA AVERAGE	NUMERIC[28]
1	Delimiter	||
10	RAW DATA %RSD	NUMERIC
1	Delimiter	||
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

FORMAT OF THE RESULTS DATA RECORD (TYPE 30) FOOTNOTES

[17]"C" (CAS Registry Number) is used for all metals and cyanide.

[18]The CAS Numbers for metals and cyanide are in Exhibit B, Form IA-IN, and Table 1 - Inorganic Target Analyte List and Contract Required Quantitation Limits (CRQLs), in Exhibit C. NOTE: The CAS Numbers for the ICS non-target interferents are as follows: carbon (7440-44-0); chlorine (7782-50-5); molybdenum (7439-98-7); phosphorus (7723-14-0); sulfur (7704-34-9), and titanium (7440-32-6).

[19]"BDL" means below detection limit.

"NSQ" means there is not sufficient quantity to prepare sample according specification in Exhibit D; therefore, a smaller sample size is used.

"NAR" means no analysis result required.

"LTC" means less than the CRQL but greater than or equal to the MDL.

"FQC" means failed Quality Control (QC) criteria.

"GTL" means greater than the linear range. The result is reported from a re-analysis at an appropriate dilution.

"RIN" means that the analysis result was not used to report data in the SDG. The result is reported from a later re-analysis of the same sample aliquot.

"REX" means that the analysis result was not used to report data in the SDG. The result is reported from a later re-analysis of a repreparation of the same sample.

Note that, except for "NAR", none of these codes relieves the Contractor from reporting a valid result. They only explain why or if the result is qualified.

[20]EPA Field Samples reported on Traffic Report/Chain of Custody Record (QC codes FLD, FRB, FRM) shall have their analytes' results reported to four decimal places.

[21]Follow the instructions for the reporting of data in Exhibit B in reporting results for samples with QC codes. For example, the LD2 QC code sample results shall be reported to four decimal places because the duplicate results on Form VI-IN have to be reported to four decimal places. Refer to Section 9.3.1 for QC codes and definitions.

[22]"T" stands for an analyte's true value in a solution. This includes the concentration of all Instrument Calibration Standards for ALL methods of analysis. "F" stands for an added concentration to a sample such as a pre- or post-digestion spike.

[23]"P" equals Percent Recovery (%R), Percent Difference (%D), Relative Percent Difference (RPD), Percent Relative Standard Deviation (%RSD), Percent Relative Intensity (%RI), or correlation coefficient. "L" equals control limit for duplicates. The matrix spike sample %R shall be entered on the Type 30 record of the EPA sample number with the "S" suffix (QC code=LSF). The post digest spike sample %R shall be entered on the Type 30 record of the EPA sample number with the "A" suffix (QC code=PDF). The RPD and the control limit for duplicates shall be entered on the Type 30 record of the EPA sample number with the "D" suffix (QC code=LD2). The ICP serial dilutions %D shall be entered on the Type 30 record of the EPA sample number with the "L" suffix (QC code=LDF). The average %RSD for ICP-MS tune analyses shall be entered on the Type 30 record of the last EPA sample number "TUNE##" (QC code=LTS) in each run. The %RI for ICP-MS internal standards shall be entered on the Type 30 record of all EPA samples numbers (except "TUNE##", "ZZZZZZ", "MDL", and "LRV"). The correlation coefficient for the calibration for mercury and

cyanide analyses shall be reported on the Type 30 record of the EPA sample number associated with the final standard analyzed in the calibration curve (immediately preceding the ICV).

[24]"N" is the qualifier that is used on Form VA-IN of the hardcopy to indicate that the matrix or pre-digestion spike sample recovery for an analyte is not within the specified control limits. The "N" qualifier shall be entered on the Type 30 record of the EPA sample number with the "S" suffix (QC code=LSF).

[25]These are the control limits for the ICV/CCV percent recovery (%R) on Form IIA-IN, the CRI %R on Form IIB-IN, the ICSA/ICSAB %R on Forms IVA and IVB-IN, the matrix spike %R on Form VA-IN, and the LCSW %R and the LCSS upper and lower limits on Form VII-IN. The QC upper and lower limits for the Spike Sample Recovery shall be entered on the Type 30 record of the EPA sample number with the "S" suffix (QC code=LSF).

[26]"*" is the qualifier that is used on Form VI-IN of the hardcopy to indicate that the duplicate sample analysis for an analyte is out of control, and "E" is the qualifier that is used on Form VIII-IN of the hardcopy to indicate that the ICP serial dilution analysis results are estimated because of the existence of significant physical or chemical interferences. The "*" qualifier should be entered on the Type 30 record of the EPA sample number with the "D" suffix (QC code=LD2) The "E" qualifier shall be entered on the Type 30 record of the EPA sample number with the "L" suffix (QC code=LDF).

[27]The MDL shall be reported to 2 significant figures for values less than 10 and to 3 significant figures for values greater than or equal to 10. MDLs shall be reported in UG/L for water samples, ICV, ICB, CCV, CCB, CRI, ICSA, ICSAB and MIDRANGE (for cyanide), and any other samples with concentration results reported in "UG/L". MDLs shall be reported in MG/KG for soil samples.

[28]The average value of the replicate injections or exposures are reported in this field. The average values for mercury and cyanide analyses are also reported in this field. In addition, the raw data average value shall always be reported in units of UG/L to a minimum of four decimal places, regardless of the units the instrument readings are reported in, on record Type 31. The raw data average value shall not be corrected for dilutions or volume adjustments.

For Instrument Calibration Standards analyses and Instrument Tune Standards analyses, the raw data average is not required to be reported.

9.7 Instrumental Data Readout (Type 31)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"31"
1	Delimiter	|
1	TYPE OF DATA	"W"/"M" [29]
1	Delimiter	|
1	TYPE OF VALUE	CHARACTER[30]
2	Delimiter	||
8	ANALYTE WAVELENGTH/MASS	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	|
15	FIRST INSTRUMENT VALUE	NUMERIC [31]
2	Delimiter	||
15	SECOND INSTRUMENT VALUE	NUMERIC [31]
2	Delimiter	||
15	THIRD INSTRUMENT VALUE	NUMERIC [31]
2	Delimiter	||
15	FOURTH INSTRUMENT VALUE	NUMERIC [31]
2	Delimiter	||
15	FIFTH INSTRUMENT VALUE	NUMERIC [31]
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

[29]"W" equals wavelength, "M" equals mass.

[30]"C" equals concentration in µg/L, "B" equals absorbance, "I" equals intensity (counts per second or equivalent).

[31]Used to report data for method analyses that require replicate injections or exposures. If a single instrument measurement is used, then enter it in the first instrument value field, and leave the other four fields empty. If two instrument measurements are used, then enter them in the first and second instrument value fields in the order of their analyses, and leave the other three fields empty, etc. In addition, the instrument values shall be reported to a minimum of four decimal places.

9.8 Auxiliary Data Record (Type 32)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"32"
10	Delimiter	||||||||||
2	INTEGRATION TIME CODE	"IT"
1	Delimiter	|
10	INTEGRATION TIME	IN SECONDS
4	Delimiter	||||
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

9.9 QC Limit Record (Type 34)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"34"
4	Delimiter	||||
8	ANALYTE WAVELENGTH OR MASS	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	|
10	CRQL	NUMERIC
1	Delimiter	|
10	LINEAR RANGE VALUE	NUMERIC
6	Delimiter	||||||
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

9.10 Correction Data Record (Type 35)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"35"
1	Delimiter	|
3	TYPE OF CORRECTION	"ICP"
1	Delimiter	|||||
9	CAS NUMBER OF INTERFERING ANALYTE	CHARACTER
1	Delimiter	|
8	ANALYTE WAVELENGTH	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	|
10	CORRECTION FACTOR	NUMERIC
1	Delimiter	|
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

9.11 Comment Record (Type 90)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"90"
1	Delimiter	|
67	ANY COMMENT	CHARACTER
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

9.12 Sample Associated Data Record (Type 92)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"92"
1	Delimiter	|
9	COLOR BEFORE	CHARACTER
1	Delimiter	|
9	COLOR AFTER	CHARACTER
1	Delimiter	|
6	CLARITY BEFORE	CHARACTER
1	Delimiter	|
6	CLARITY AFTER	CHARACTER
1	Delimiter	|
6	TEXTURE	CHARACTER
1	Delimiter	|
3	ARTIFACTS	"YES"/BLANK
1	Delimiter	|
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

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APPENDIX A -- FORMAT OF RECORDS FOR SPECIFIC USES

DISCLAIMER

The USEPA does not warrant or guarantee the completeness and/or accuracy of the representative examples of record type uses provided in this appendix. This appendix serves as an example for the usage of record types and in no way redefines or supersedes the specifications or requirements stated in Exhibits A through H of ILM05.2.

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Appendix A -- Format of Records for Specific Uses

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1.0 ICP

1.1 ICP-AES

1.1.1 Start of an ICP-AES Run with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|17|09|06|P|ILM05.2|ABC|TESLAB||||68-D2-0039|P2||TEST LABSINC.|2|000001879
16|1999|09|17|12|03|Y|Y|Y|N|000012114

20|1|MDL|1|MDQ||||1999|07|15||||||04|000044B9D
21||||NP1|||||||||||||||000053CD5
30|C|7440-22-4||UG/L|||||||||||||||U|3.1||||||000065996
30|C|7429-90-5||UG/L|||||||||||||||U|21.8||||||0000767D1
30|C|7440-39-3||UG/L|||||||||||||||U|11.5||||||0000875CB
30|C|7440-41-7||UG/L|||||||||||||||U|1.1||||||0000983C5

20|1|MDL|1|MDQ||||1999|07|15||||||04|0000104B9D
21||||HW1||||1999|07|15|||||||08|00|0000113CD5
30|C|7440-22-4||UG/L|||||||||||||||U|3.4||||||0000125996
30|C|7429-90-5||UG/L|||||||||||||||U|22.8||||||00001367D1
30|C|7440-39-3||UG/L|||||||||||||||U|12.5||||||00001475CB
30|C|7440-41-7||UG/L|||||||||||||||U|2.1||||||00001583C5

20|1|MDL|F|MDQ||||1999|07|16||||||04|000164B9D
21||||HS1||||1999|07|15|||||||08|00|00017A212
30|C|7440-22-4||MG/KG|||||||||||||||U|0.82||||||00018C248
30|C|7429-90-5||MG/KG|||||||||||||||U|4.8||||||00019B321
30|C|7440-39-3||MG/KG|||||||||||||||U|3.1||||||00020CE75
30|C|7440-41-7||MG/KG|||||||||||||||U|0.42||||||00021A21B

20|1|LRV|1|LRQ||||1999|07|15||||||04|0002356C2
30|C|7440-22-4|||||||||||||||||||||||||0002463D1
32||||||||||IT|5.00||||000256CDA
34||||328.00|5|40000||||||000267591
30|C|7429-90-5|||||||||||||||||||||||||0002782AD
32||||||||||IT|5.00||||000288BB6
34||||308.20|200|1000000||||||0002994FB
30|C|7440-39-3|||||||||||||||||||||||||00030A211
32||||||||||IT|5.00||||00031AB1A
34||||493.40|20|100000||||||00032B436
30|C|7440-41-7|||||||||||||||||||||||||00033C149
32||||||||||IT|5.00||||00034CA52
34||||313.00|2|25000||||||00035D2DA

20|1|BCD||BCQ||||1999|07|01||||||04|0007894FB
30|C|7440-22-4|||||||||||||||||||||||||00079A20A
35|ICP|||||7439-89-6|259.90|-0.0002500|00080AC9B
35|ICP|||||7439-96-5|257.60|0.0002200|00081B6F4
30|C|7429-90-5|||||||||||||||||||||||||00082C410
35|ICP|||||7439-96-5|257.60|0.0004900|00083CE72
35|ICP|||||7440-62-2|292.40|-0.0419200|00084D8EF
30|C|7440-39-3|||||||||||||||||||||||||00085E605
35|ICP|||||7439-96-5|257.60|0.0000600|00086F060
30|C|7440-41-7|||||||||||||||||||||||||00087FD73
35|ICP|||||7440-50-8|324.70|0.0046200|0008914D1
35|ICP|||||7439-96-5|257.60|0.0015400|000901F30
```

1.1.2 ICP-AES Instrument Calibration Standards, S0 and S

```
20|1|S0|1|STB||20596|MAX123|1999|09|17|09|06||||04|00128D199
21||||NP1||STDB|1999|09|17|||||TESLAB||||00129DD31
22||||||||||||1.0||||00130E598
```

```
30|C|7440-22-4|||||T|0.0|||||||||||U|3.1||||||00131F8F5
31|W|I||328.00|0.0304||0.0374||0.0400|||||001320305
30|C|7429-90-5|||||T|0.0|||||||||||U|21.8||||||001331697
31|W|I||308.20|0.0104||0.0136||0.0120|||||001342137
30|C|7440-39-3|||||T|0.0|||||||||||U|11.5||||||00135348D
31|W|I||493.40|-0.0002||0.0002||0.0000|||||001363EA4
30|C|7440-41-7|||||T|0.0|||||||||||U|1.1||||||0013751FA
31|W|I||313.00|0.0006||0.0002||0.0004|||||001385C04

20|1|S|1|STD||20596|MAX123|1999|09|17|09|11||||04|00206314E
21||||NP1||STD1|1999|09|17|||||TESLAB||||002073CD5
22||||||||||||1.0||||00208453C
30|C|7440-22-4|||||T|5Q00|||||||||||U|3.1||||||002139157
31|W|I||328.00|1.9540||1.9610||1.9660|||||002149B6E
30|C|7429-90-5|||||T|1000|||||||||||U|21.8||||||00215ADE2
31|W|I||308.20|0.8384||0.8378||0.8440|||||00216B7EC
30|C|7440-39-3|||||T|5000|||||||||||U|11.5||||||00219E77D
31|W|I||493.40|1.9460||1.9510||1.9684|||||00220F18F
30|C|7440-41-7|||||T|5000|||||||||||U|1.1||||||002210410
31|W|I||313.00|0.9924||0.9910||1.0010|||||002220E25
```

1.1.3 Duplicates, Spike Sample Recovery, and Serial Dilutions Performed on the Same Field Sample (QC Codes FLD, LDO, LD2, LSF, LDF)

```
20|1|MAX123|F|FLD||20596|MAX123|1999|09|17|11|09||G|1.05|08|01568C5FD
21||LOW||HS1||S308233-01|1999|09|14||1999|08|24|||08|30|01569D451
22||||||||||200|1.0|||91.5|01570DE17
90|STONES|01571E154
92|GREY|GREY|||MEDIUM|YES|01572EA43
30|C|7440-22-4||MG/KG|BDL|2.0817||||||||||||U|0.82||1.1567||||01573FD12
31|W|C||328.00|4.2000||0.5500||-1.2800|||||0157409A5
30|C|7429-90-5||MG/KG||6227.0101||||||||||||U|4.8||29913.0000||||015751DCD
31|W|C||308.20|29992.0000||29654.0000||30093.0000|||||015762CA0
30|C|7440-39-3||MG/KG|LTC|21.9349||||||||||||U|3.1||105.3700||||01577400C
31|W|C||493.40|107.2400||101.6400||107.2300|||||015784DA6
30|C|7440-41-7||MG/KG|BDL|1.0409||||||||||||U|0.42||1.4900||||01579606A
31|W|C||313.00|1.4900||1.4900||1.4900|||||015806CD9

20|1|MAX123|F|LDO||20596|MAX123|1999|09|17|11|09||G|1.05|08|01650C630
21||LOW||HS1||S308233-01|1999|09|14||1999|08|24|||08|30|01651D484
22||||||||||200|1.0|||91.5|01652DE4A
30|C|7440-22-4||UG/L|BDL|10.00||||||||||||U|3.9||1.1567||||01655FC98
31|W|C||328.00|4.2000||0.5500||-1.2800|||||01656092B
30|C|7429-90-5||UG/L||29913.00||||||||||||U|23.1||29913.0000||||016571C09
31|W|C||308.20|29992.0000||29654.0000||30093.0000|||||016582ADC
30|C|7440-39-3||UG/L|LTC|105.37||||||||||||U|14.9||105.3700||||016593DCE
31|W|C||493.40|107.2400||101.6400||107.2300|||||016604B68
30|C|7440-41-7||UG/L|BDL|5.00||||||||||||U|2.0||1.4900||||01579606A
31|W|C||313.00|1.4900||1.4900||1.4900|||||015806CD9

20|1|MAX123D|F|LD2||20596|MAX123|1999|09|17|11|11||G|1.04|08|016913BCF
21||LOW||HS1||S308233-02|1999|09|14||1999|08|24|||08|30|016924A23
22||||||||||200|1.0|||90.9|0169353EC
30|C|7440-22-4||MG/KG|BDL|2.1017||||||||||||U|0.82||0.9600||||0169466BE
31|W|C||328.00|1.6400||1.6300||-0.3900|||||016957356
30|C|7429-90-5||MG/KG||6622.7406|||P|6.2||||||||U|4.8||31511.0000||||016968784
31|W|C||308.20|31993.0000||31313.0000||31227.0000|||||016979641
30|C|7440-39-3||MG/KG|LTC|25.1387|||P|13.6|||||||U|3.1||119.6100||||01698AAC5
31|W|C||493.40|121.4600||118.9300||118.4400|||||01699B86C
30|C|7440-41-7||MG/KG|BDL|1.0509||||||||||||U|0.42||1.5000||||01700CC13
31|W|C||313.00|1.5000||1.5000||1.5000|||||01701D86A
```

```
20|1|MAX123S|F|LSF||20596|MAX123|1999|09|17|11|14||G|1.01|08|01730BE3C
21||LOW||HS1||S308233-03|1999|09|14||1999|08|24|||08|30|01731CC90
22||||||||||200|1.0|||91.5|01732D656
30|C|7440-22-4||MG/KG||10.7212|F|10.82|P|99||||||75|125||U|0.82||49.5400||||01733EBC7
31|W|C||328.00|48.8400||49.2000||50.5800|||||01734F8DC
30|C|7429-90-5||MG/KG|NAR|6859.9253|||||||||||||U|4.8||31698.0000||||017350E27
31|W|C||308.20|31578.0000||31766.0000||31750.0000|||||017361CF1
30|C|7440-39-3||MG/KG||326.3539|F|432.83|P|70|||||N|75|125||U|3.1||1508.0000||||017373339
31|W|C||493.40|1524.0000||1504.4000||1495.6000|||||017384171
30|C|7440-41-7||MG/KG||10.4290|F|10.82|P|96||||||75|125||U|0.42||48.1900||||0173956E4
31|W|C||313.00|48.1900||48.2000||48.1800|||||0174063EB

20|1|MAX123L|F|LDF||20596|MAX123|1999|09|17|11|17||||08|017696573
21||LOW||||S308233-04|||||1999|08|24|||||017707255
22||||||||||||5.0|||91.5|017717B8D
30|C|7440-22-4||UG/L|BDL|50.00|||||||||||||U|3.9||0.6100||||017728DDF
31|W|C||328.00|1.4500||-0.3800||0.7600|||||017739A7B
30|C|7429-90-5||UG/L||25575.50|||P|15|||||||E|U|23.1||5115.1000||||01774AE69
31|W|C||308.20|5038.6000||5126.4000||5180.3000|||||01775BCAC
30|C|7440-39-3||UG/L|LTC|111.30|||P|6|||||||||U|14.9||22.2600||||01776D0AA
31|W|C||493.40|22.2600||22.7700||21.7500|||||01777DDB9
30|C|7440-41-7||UG/L|BDL|25.00|||||||||||||U|2.0||0.3000||||0173956E4
31|W|C||313.00|0.1900||0.2000||0.51|||||0174063EB
```

1.2 ICP-MS

1.2.1 Start of an ICP-MS Run with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|17|09|06|MS|ILM05.2|ABC|TESLAB||||68-D2-0039|P2||TEST LABSINC.|2|000001879
16|1999|09|17|12|03|Y|Y|Y|N|000012114

20|1|MDL|1|MDQ||||1999|07|15||||||04|000044B9D
21|||||NP1|||||||||||||||00005DD31
30|C|7440-22-4||UG/L|||||||||||||||||U|0.40||||||000065996
30|C|7429-90-5||UG/L|||||||||||||||||U|12.8||||||0000767D1
30|C|7440-39-3||UG/L|||||||||||||||||U|3.0||||||0000875CB
30|C|7440-41-7||UG/L|||||||||||||||||U|0.44||||||0000983C5

20|1|MDL|1|MDQ||||1999|07|15||||||04|000044B9D
21|||||HW2|||1999|07|15|||||||09|00|00005DD31
30|C|7440-22-4||UG/L|||||||||||||||||U|0.41||||||000065996
30|C|7429-90-5||UG/L|||||||||||||||||U|13.8||||||0000767D1
30|C|7440-39-3||UG/L|||||||||||||||||U|4.0||||||0000875CB
30|C|7440-41-7||UG/L|||||||||||||||||U|0.43||||||0000983C5

20|1|LRV|1|LRQ||||1999|07|15||||||04|0002356C2
30|C|7440-22-4|||||||||||||||||||||||||0002463D1
32||||||||||IT|5.00||||000256CDA
34||||107.00|5|40000||||||000267591
30|C|7429-90-5|||||||||||||||||||||||||0002782AD
32||||||||||IT|5.00||||000288BB6
34||||27.00|200|1000000||||||0002994FB
30|C|7440-39-3|||||||||||||||||||||||||00030A211
32||||||||||IT|5.00||||00031AB1A
34||||137.00|20|100000||||||00032B436
30|C|7440-41-7|||||||||||||||||||||||||00033C149
32||||||||||IT|5.00||||00034CA52
34||||111.00|2|25000||||||00035D2DA
```

1.2.2 ICP-MS Instrument Tune and Calibration Standards, S0 and S

```
20|3|TUNEA1|1|LTS||26791|MCSB00|1999|02|06|20|00||||5|000917DD7
```

```
21||||||TUNE1||||||||TESLAB||||000917DD8
22||||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|||||||||||||||||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|||||||||||||||||||000914DE2
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|||||||||||||||||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|||||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|||||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|3|TUNEA2|1|LTS||26791|MCSB00|1999|02|06|20|10||||5|000917DD7
21||||||TUNE2||||||||TESLAB||||000917DD8
22||||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|||||||||||||100000||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|||||||||||||||||||000914DE2
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|||||||||||||100000||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|||||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|||||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|3|TUNEA3|1|LTS||26791|MCSB00|1999|02|06|20|20||||5|000917DD7
21||||||TUNE3||||||||TESLAB||||000917DD8
22||||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|||||||||||||||||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|||||||||||||||||||000914DE2
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|||||||||||||||||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|||||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|||||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|3|TUNEA4|1|LTS||26791|MCSB00|1999|02|06|20|30||||5|000917DD7
21||||||TUNE4||||||||TESLAB||||000917DD8
22||||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|||||||||||||||||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|||||||||||||||||||000914DE2
```

```
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|||||||||||||||||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|||||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|||||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|3|TUNEA5|1|LTS||26791|MCSB00|1999|02|06|20|40||||5|000917DD7
21||||||TUNE5|||||||TESLAB||||000917DD8
22||||||||||||1.0||||000917DD9
30|C|7440-41-7|||||T|100|P|0.0|||||||||||||||||000917DE0
31|M|I||9.01|100000||100000||100000|||||000917DE1
30|C|7439-95-4|||||T|100|P|0.0|||||||||||||||||000914DE2
31|M|I||23.99|79000||79000||79000|||||000917DE3
31|M|I||24.99|10000||10000||10000|||||000917DE4
31|M|I||25.98|11000||11000||11000|||||000917DE5
30|C|7440-48-4|||||T|100|P|0.0|||||||||||||||||000917DE6
31|M|I||58.93|100000||100000||100000|||||000917DE7
30|C|7440-74-6|||||T|100|P|0.0|||||||||||||||||000917DE8
31|M|I||112.90|4000||4000||4000|||||000917DE9
31|M|I||114.90|96000||96000||96000|||||000917DF0
30|C|7439-92-1|||||T|100|P|0.0|||||||||||||||||000917DF1
31|M|I||205.97|24000||24000||24000|||||000917DF2
31|M|I||206.98|22000||22000||22000|||||000917DF3
31|M|I||207.98|52000||52000||52000|||||000917DF4

20|1|SO|1|STB||20596|MAX123|1999|09|17|09|06||||04|00128D199
21||||NP1||STDB|1999|09|17|||||TESLAB||||00129DD31
22||||||||||||1.0||||00130E598
30|C|7440-22-4|||||T|0.0||||||||||U|0.40||||||00131F8F5
31|M|I||107.00|0.0304||0.0374||0.0400|||||001320305
30|C|7429-90-5|||||T|0.0||||||||||U|12.8||||||001331697
31|M|I||27.00|0.0104||0.0136||0.0120|||||001342137
30|C|7440-39-3|||||T|0.0||||||||||U|3.0||||||00135348D
31|M|I||137.00|-0.0002||0.0002||0.0000|||||001363EA4
30|C|7440-41-7|||||T|0.0||||||||||U|0.44||||||0013751FA
31|M|I||111.00|0.0006||0.0002||0.0004|||||001385C04

20|1|S|1|STD||20596|MAX123|1999|09|17|09|11||||04|00206314E
21||||NP1||STD1|1999|09|17|||||TESLAB||||002073CD5
22||||||||||||1.0||||00208453C
30|C|7440-22-4|||||T|5000||||||||||U|0.40||||||002139157
31|M|I||107.00|1.9540||1.9610||1.9660|||||002149B6E
30|C|7429-90-5|||||T|1000||||||||||U|12.8||||||00215ADE2
31|M|I||27.00|0.8384||0.8378||0.8440|||||00216B7EC
30|C|7440-39-3|||||T|5000||||||||||U|3.0||||||00219E77D
31|M|I||136.00|1.9460||1.9510||1.9684|||||00220F18F
30|C|7440-41-7|||||T|5000||||||||||U|0.44||||||002210410
31|M|I||111.00|0.9924||0.9910||1.0010|||||002220E25
```

1.2.3 Field Samples

```
20|1|MAX123|1|FLD||20596|MAX123|1999|09|17|09|06||ML|100|04|00128D199
21||||HW2||S308233-01|1999|09|17||1999|09|16|TESLAB||09|30|00129DD31
22||||||||1.25||50|1.0|||0.0|00130E598
30|C|7440-22-4||UG/L|LTC|0.6625||||||||||||U|0.41||0.5300||||00131F8F5
```

```
31|M|I||107.00|0.5300||0.5300||0.5300|||||001320305
30|C|7429-90-5||UG/L||56.3750|||||||||||||U|13.8||45.1000||||001331697
31|M|I||27.00|45.1000||45.1000||45.1000|||||001342137
30|C|7440-39-3||UG/L||11.0000|||||||||||||U|4.0||8.8000||||00135348D
31|M|I||137.00|8.8000||8.8000||8.8000|||||001363EA4
30|C|7440-41-7||UG/L|BDL|1.000|||||||||||||U|0.43||0.3210||||0013751FA
31|M|I||111.00|0.3210||0.3210||0.3210|||||001385C04

20|1|MAX124|1|FLD||20596|MAX123|1999|09|17|09|06||ML|20|04|00128D199
21||||NP1||S308234-01|1999|09|17||1999|09|16|TESLAB||09|30|00129DD31
22||||||||||20|1.0|||0.0|00130E598
30|C|7440-22-4||UG/L|LTC|0.5300|||||||||||||U|0.40||0.5300||||00131F8F5
31|M|I||107.00|0.5300||0.5300||0.5300|||||001320305
30|C|7429-90-5||UG/L||45.1000|||||||||||||U|12.8||45.1000||||001331697
31|M|I||27.00|45.1000||45.1000||45.1000|||||001342137
30|C|7440-39-3||UG/L|LTC|8.8000|||||||||||||U|3.0||8.8000||||00135348D
31|M|I||137.00|8.8000||8.8000||8.8000|||||001363EA4
30|C|7440-41-7||UG/L|BDL|1.000|||||||||||||U|0.44||0.3210||||0013751FA
31|M|I||111.00|0.3210||0.3210||0.3210|||||001385C04
```

2.0 MERCURY

2.1 Start of a Mercury Run for Water Samples with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|09|08|44|CV|ILM05.2|ABC|TESLAB||||68-D2-0039|M3||TEST LABS INC.|6|0000018F7
16|1999|09|09|14|34|N||||000012099

20|1|MDL|1|MDQ||||1999|07|15||||||1|000044AEB
21||||CW1|||1999|07|15|||||||||000053CD5
30|C|7439-97-6||UG/L|||||||||||||||U|0.042||||||0000658F4

20|1|LRV|1|LRQ||||1999|09|09||||||1|0000666A6
30|C|7439-97-6|||||||||||||||||||||||||0000773CB
32|||||||||||||||000087D02
34||||253.70|0.2|5||||||00009852D
```

2.1.1 Start of a Mercury Run for Soil Samples with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|09|08|44|CV|ILM05.2|ABC|TESLAB||||68-D2-0039|M3||TEST LABS INC.|6|0000018F7
16|1999|09|09|14|34|N||||000012099

20|1|MDL|F|MDQ||||1999|07|16||||||1|000074AEB
21||||CS1|||1999|07|16|||||||09|00|000083CD5
30|C|7439-97-6||MG/KG|||||||||||||||U|0.0092||||||0000958F4

20|1|LRV|1|LRQ||||1999|09|09||||||1|0001066A6
30|C|7439-97-6|||||||||||||||||||||||||0001173CB
32|||||||||||||||000127D02
34||||253.70|0.2|5||||||00013852D
```

2.2 Mercury Instrument Calibration Standards: Blank (S0) and Four Other Standards

```
20|1|S0|1|STB||20596|MAX123|1999|09|09|08|44||||1|00010936F
21||||CS1||0PPB|1999|09|09|||||TESLAB||07|00|000119F0C
22||||||||||||1.0||||00012A773
30|C|7439-97-6|||||T|0.0|||||||||||U|0.018||0.0122||||00013BAD9
31|W|C||253.70|0.0122|||||||||00014C4EC

20|1|S0.2|1|STC||20596|MAX123|1999|09|09|08|48||||1|00015D392
21||||CS1||0.2PPB|1999|09|09|||||TESLAB||07|00|00016DF8F
22||||||||||||1.0||||00017E7F6
```

```
30|C|7439-97-6|||||T|0.2|||||||||||U|0.018||0.0896||||00018FB5E
31|W|C||253.70|0.0896|||||||||000190571

20|1|S1.0|1|STD||20596|MAX123|1999|09|09|08|53||||1|000201412
21||||CS1||1.0PPB|1999|09|09|||||TESLAB||07|00|00021200E
22|||||||||||1.0||||000222875
30|C|7439-97-6|||||T|1.0|||||||||||U|0.018||1.0128||||000233BDC
31|W|C||253.70|1.0128|||||||||0002445EF

20|1|S2.0|1|STD||20596|MAX123|1999|09|09|08|57||||1|000255495
21||||CS1||2.0PPB|1999|09|09|||||TESLAB||07|00|000266092
22|||||||||||1.0||||0002768F9
30|C|7439-97-6|||||T|2.0|||||||||||U|0.018||2.0055||||000287C61
31|W|C||253.70|2.0055|||||||||000298674

20|1|S5.0|1|STD||20596|MAX123|1999|09|09|09|01||||1|000309513
21||||CS1||5.0PPB|1999|09|09|||||TESLAB||07|00|00031A113
22|||||||||||1.0||||00032A97A
30|C|7439-97-6|||||T|5.0|P|0.9997|||||||||U|0.018||4.9952||||00033BCE5
31|W|C||253.70|4.9952|||||||||00034C6F8
```

2.3 Spike Sample Recovery and Duplicates Performed on Different Samples (QC Codes FLD, LSF, FLD, LD2)

```
20|1|MAX123|F|FLD||20596|MAX123|1999|09|09|13|20||G|0.20|1|002106798
21||LOW||CS1||S308233-01|1999|09|09||1999|08|24|||07|00|0021175EF
22|||||||||||100|1.0|||91.5|002127FB4
30|C|7439-97-6||MG/KG|BDL|0.1093|||||||||||||U|0.0092||0.0049||||002159EC0
31|W|C||253.70|0.0049|||||||||00216A8E3

20|1|MAX123S|F|LSF||20596|MAX123|1999|09|09|13|25||G|0.20|1|00229534B
21||LOW||CS1||S308233-03|1999|09|09||1999|08|24|||07|00|0023061A2
22|||||||||||100|1.0|||91.5|002316B67
30|C|7439-97-6||MG/KG||0.5664|F|0.55|P|103||||||75|125||U|0.0092||1.0366||||00232807A
31|W|C||253.70|1.0366|||||||||002338A9D

20|1|MAX126|F|FLD||20596|MAX123|1999|09|09|13|30||G|0.20|1|00217B9F5
21||LOW||CS1||S308233-06|1999|09|09||1999|08|24|||07|00|00218C84C
22|||||||||||100|1.0|||85.6|00219D211
30|C|7439-97-6||MG/KG||1.5053|||||||||||||U|0.0092||2.5771||||00222F11D
31|W|C||253.70|2.5771|||||||||00223FB40

20|1|MAX126D|F|LD2||20596|MAX123|1999|09|09|13|35||G|0.20|1|002240C9D
21||LOW||CS1||S308233-07|1999|09|09||1999|08|24|||07|00|002251AF4
22|||||||||||100|1.0|||85.1|0022624BC
30|C|7439-97-6||MG/KG|BDL|0.1175|||P|200|||L|0.0383||||*|U|0.0092||0.0028||||002273795
31|W|C||253.70|0.0028|||||||||0022841B9
```

2.4 Duplicates and Spike Sample Recovery Performed on the Same Sample (QC Codes FLD, LD2, LSF)

```
20|1|MAX126|F|FLD||20596|MAX123|1999|09|09|16|10||G|0.20|1|002106798
21||LOW||CS1||S308233-06|1999|09|09||1999|08|24|||07|00|0021175EF
22|||||||||||100|1.0|||91.5|002127FB4
30|C|7439-97-6||MG/KG||0.6429|||||||||||||U|0.0092||1.1765||||002159EC0
31|W|C||253.70|1.1765|||||||||00216A8E3

20|1|MAX126D|F|LD2||20596|MAX123|1999|09|09|16|15||G|0.20|1|002240C9D
21||LOW||CS1||S308233-07|1999|09|09||1999|08|24|||07|00|002251AF4
22|||||||||||100|1.0|||90.9|0022624BC
30|C|7439-97-6||MG/KG||0.2342|||P|94|||L|0.0364||||*|U|0.0092||0.4286||||002273795
31|W|C||253.70|0.4286|||||||||0022841B9
```

```
20|1|MAX126S|F|LS5||20596|MAX123|1999|09|09|16|20||G|0.20|1|00229534B
21||LOW||CS1||S308233-08|1999|09|09||1999|08|24|||07|00|0023061A2
22||||||||||100|1.0|||91.5|002316B67
30|C|7439-97-6||MG/KG||0.9710|F|0.55|P|60|||||N|75|125||U|0.0092||1.7769||||00232807A
31|W|C||253.70|1.7769|||||||||002338A9D
```

2.5 Initial Calibration Verification (ICV) with LVM QC Code

```
20|1|ICV1A|1|LVM||20596|MAX123|1999|09|09|09|06||||1|00035D687
21||||CS1||ICV-5|1999|09|09|||07|00|ICF(0791)||07|00|00036E25E
22||||||||||2.0||||00037EAC6
30|C|7439-97-6||UG/L||4.91|T|4.9|P|100||||||80.0|120.0||U|0.018||2.4559||||00038FFD0
31|W|C||253.70|2.4559|||||||||0003909FC
```

2.6 Laboratory Control Sample (Solid) with LCM QC Code

```
20|1|LCSSC3|F|LCM||20596|MAX123|1999|09|09|12|24||G|0.20|1|001256DBA
21||||CS1||LCSHG|1999|09|09|||||QAL-0287||07|00|001267B1B
22||||||||||100|1.0||||001278443
30|C|7439-97-6||MG/KG||4.6|T|4.2|P|110||||||2.8|6.0||U|0.0092||9.2000||||00128996D
31|W|C||253.70|2.7719|||||||||00129A39A
```

3.0 CYANIDE

3.1 Start of a Cyanide Run with Record Types 10 and 16 and the First Type 20 Records

```
10|1999|09|01|14|09|AS|ILM05.2|ABC|TESLAB||||68-D2-0039|C1||TEST LABS INC.|7|00000189C
16|1999|09|01|15|03|Y||||000012033

20|1|MDL|1|MDQ||||1999|07|15||||||1|000044A74
21||||NP1||||1999|07|15|||||||10|30|000053CD5
30|C|57-12-5||UG/L|||||||||||||||U|1.7||||||0000656DC

20|1|MDL|1|MDQ||||1999|07|15||||||1|000044A74
21||||DW1||||1999|07|15|||||||10|30|000053CD5
30|C|57-12-5||UG/L|||||||||||||||U|1.8||||||0000656DC

20|1|MDL|F|MDQ||||1999|07|16||||||1|000044A74
21||||DS2||||1999|07|16|||||||07|45|000053CD5
30|C|57-12-5||MG/KG|||||||||||||||U|0.092||||||0000656DC

20|1|LRV|1|LRQ||||1999|09|01||||||1|000066486
30|C|57-12-5|||||||||||||||||||||||||000076FDA
32||||||||||IT|45.00||||000087917
34||||620.00|10|400||||||000098169
```

3.2 Cyanide Instrument Calibration Standards: Blank (S0) and Five Other Standards

```
20|1|S0|1|STB||20596|MAX123|1999|09|01|14|09||||1|000108FA1
21||||NP1||0PPB|||||||TESLAB||||000119B3E
22||||||||||1.0||||00012A3A5
30|C|57-12-5|||||T|0.0|||||||||||U|1.7||0.3543||||00013B48B
31|W|C||620.00|0.3543|||||||||00014BD34

20|1|S10|1|STC||20596|MAX123|1999|09|01|14|10||||1|00015CB95
21||||NP1||10PPB|||||||TESLAB||||00016D763
22||||||||||1.0||||00017DFCA
30|C|57-12-5|||||T|10.0|||||||||||U|1.7||11.1700||||00018F0D2
31|W|C||620.00|11.1700|||||||||00019F97B

20|1|S40|1|STD||20596|MAX123|1999|09|01|14|11|||||1|0002007E0
21||||NP1||40PPB|||||||TESLAB||||0002113B1
22||||||||||1.0||||000221C18
```

```
30|C|57-12-5|||||T|40.0|||||||||||U|1.7||38.4000||||000232D23
31|W|C||620.00|38.4000|||||||||0002435CC

20|1|S100|1|STD||20596|MAX123|1999|09|01|14|12||||1|00025445F
21||||NP1||100PPB|||||||TESLAB||||00026505D
22|||||||||||1.0||||0002758C4
30|C|57-12-5|||||T|100.0|||||||||||U|1.7||99.7400||||000232D23
31|W|C||620.00|99.7400|||||||||0002972A5

20|1|S200|1|STD||20596|MAX123|1999|09|01|14|12||||1|000308139
21||||NP1||200PPB|||||||TESLAB||||000318D38
22|||||||||||1.0||||00032959F
30|C|57-12-5|||||T|200.0|||||||||||U|1.7||201.3000||||00033A6D8
31|W|C||620.00|201.3000|||||||||00034AF81

20|1|S400|1|STD||20596|MAX123|1999|09|01|14|13||||1|00035BE18
21||||NP1||400 PPB|||||||TESLAB||||00036CA19
22|||||||||||1.0||||00037D280
30|C|57-12-5|||||T|400.0|P|1.0000|||||||||U|1.7||399.5000||||00038E3BB
31|W|C||620.00|399.5000|||||||||00039EC64
```

3.3 Preparation Blank (Soil) with LRB QC Code

```
20|1|PBSD1|F|LRB||20596|MAX123|1999|09|01|14|23||G|1.00|1|000928FA0
21||||DS2||PB|1999|08|30|||||||08|30|000939A40
22|||||||||||50|1.0||||00094A30C
30|C|57-12-5||MG/KG|BDL|1.0000|||||||||||||U|0.092||-0.0030||||00095B433
31|W|C||620.00|-0.0030|||||||||00096BE6F
```

3.4 Laboratory Control Sample (Soil) with LCM QC Code

```
20|1|LCSSD1|F|LCM||20596|MAX123|1999|09|01|14|24||G|1.00|1|00097CF4D
21||||DS2||LCSCN|1999|08|30|||||QAL-0689||08|30|00098DCB0
22|||||||||||50|1.0||||00099E57C
30|C|57-12-5||MG/KG||5.0|T|5.6|P|89||||||4.3|6.9||U|0.092||100.0933||||00100F89B
31|W|C||620.00|100.0933|||||||||001010315
```

3.5 Continuing Calibration Verification (CCV) with LVC QC Code

```
20|1|CCV11|1|LVC||20596|MAX123|1999|09|01|14|30||||1|0015045A3
21||||NP1||200 PPB|||||||TESLAB||||0015151A2
22|||||||||||1.0||||001525A09
30|C|57-12-5||UG/L||188.48|T|200.0|P|94||||||85.0|115.0||U|1.7||188.4772||||001536E87
31|W|C||620.00|188.4772|||||||||001547916
```

3.6 Spike Sample Recovery and Post Distillation Spike Sample Recovery Performed on the Same Sample (QC Codes FLD, PDO, LSF, PDF)

```
20|1|MAX123|F|FLD||20596|MAX123|1999|09|01|14|35||G|1.06|1|001955D8E
21||LOW||DS2||S308233-01|1999|08|30||1999|08|24|||08|30|001966BDF
22|||||||||||50|1.0|||71.0|001977578
30|C|57-12-5||MG/KG|LTC|0.2952|||||||||||||U|0.092||4.4441||||002009309
31|W|C||620.00|4.4441|||||||||002019D4B

20|1|MAX123|F|PDO||20596|MAX123|1999|09|01|14|35||G|1.06|1|00202AE62
21||LOW||DS2||S308233-01|1999|08|30||1999|08|24|||08|30|00203BCB3
22|||||||||||50|1.0|||71.0|00204C64C
30|C|57-12-5||UG/L|LTC|4.44|||||||||||||U|1.4||4.4441||||00207E3DD
31|W|C||620.00|4.4441|||||||||00208EE1F

20|1|MAX123S|F|LSF||20596|MAX123|1999|09|01|14|36||G|1.02|1|00209FF7A
21||LOW||DS2||S308233-02|1999|08|30||1999|08|24|||08|30|002100DCB
22|||||||||||50|1.0|||71.0|002111767
```

```
30|C|57-12-5||MG/KG||4.6341|F|6.90|P|63|||||N|75|125||U|0.092||67.1210||||0021228D6
31|W|C||620.00|67.1210|||||||||002133324

20|1|MAX123A|F|[redacted]||20596|MAX123|1999|09|01|14|37||G|1.06|1|0021444AD
21||LOW||||S308233-03|1999|08|30||1999|08|24|||||0021552FE
22||||||||||50|1.0|||71.0|002165C98
30|C|57-12-5||UG/L||21.23|F|20.0|P|84|||||||||U|1.4||21.2279||||0021770C0
31|W|C||620.00|21.2279|||||||||002187B4E
```

APPENDIX B - Modified Analysis

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Appendix B - Modified Analysis

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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, analysis of additional analytes and/or lower quantitation limits. These requests will be made by the USEPA Regional CLP Project Officer (CLP PO), USEPA OERR Analytical Operations/Data Quality Center (AOC) Inorganic Program Manager (PM), and USEPA Contracting Officer (CO) in writing, prior to sample scheduling. If the Contractor voluntarily elects to perform these modified analyses, these analyses will be performed with no increase in per sample price. All contract requirements specified in the SOW/Specifications will remain in effect unless the USEPA CO provides written approval for the modification(s) and a waiver for associated defects. The USEPA CO approval must be obtained prior to sample scheduling.

GRAPHITE FURNACE ATOMIC ABSORPTION METHOD

1.0 SCOPE AND APPLICATION

This method is a graphite furnace atomic absorption spectroscopy procedure that is used to analyze water, sediment, sludge, and soil samples taken from hazardous waste sites. The following metals: arsenic, lead, selenium, and thallium that are contained in the Target Analyte List (TAL) in Exhibit C may be quantitated by the Graphite Furnace Atomic Absorption (GFAA) method.

2.0 SUMMARY OF METHOD

Water and soil samples are treated with acids and heat to solubilize the metals present. These digestates are then analyzed for trace metals by the Graphite Furnace Atomic Absorption (GFAA) spectroscopic technique. In this technique, a tube of graphite is located in the sample compartment of the Atomic Absorption (AA) spectrometer, with the light passing through it. A small volume of sample solution is quantitatively placed into the tube, normally through a sample injection hole located in the center of the tube wall. The tube is heated through a programmed temperature sequence until finally the analyte present in the sample is dissociated into atoms and atomic absorption occurs.

3.0 DEFINITIONS

See Exhibit G 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 water and soil/sediments. Dissolved elements are determined in filtered and acidified samples. 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 milligrams per Liter (mg/L). In addition, total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps must be taken to correct for potential interference effects.

Interferences from the Graphite Furnace Atomic Absorption (GFAA) technique can be divided into two broad categories, spectral and nonspectral interferences. Spectral interferences are those resulting from light absorption by molecules or by atoms other than those of the analyte element; that is, spectral interference exists if the atomic absorption profile of an element overlaps the emission line of another. Nonspectral interferences are those which affect the production or availability of analyte atoms which create the measured atomic absorption.

4.1 Spectral Interferences

4.1.1 Emission Interference - this interference arises when the intense light emitted by the hot graphite tube reaches the instrument's light detector, the Photomultiplier Tube (PMT). This problem is manifested by increased signal variability (noise) which degrades analytical performance. In severe circumstances, emission interference may temporarily blind the PMT, resulting in erratic, meaningless readings at atomization.

4.1.2 Background Absorption - this is the most severe spectral interference encountered with graphite furnace analyses. Background absorption is a nonspecific attenuation of light at the analyte wavelength caused by matrix components in the sample. Unlike atomic absorption, background absorption is broad band, sometimes covering tens or even hundreds of nanometers. This broad band absorption normally is due to molecular absorption or light scattering caused by undissociated sample matrix components in the light path at atomization. Since background absorption is broad band, the chance of overlap with a desired analyte wavelength is significant.

4.1.3 Emission interference is controlled by primarily by spectrometer optical design. Techniques for controlling and reducing background absorption include matrix modification (sample treatment) and optical background correction. Through matrix modification, a reagent or "matrix modifier" is added to the sample or standard. The matrix modifier is selected to generate either an increased matrix volatility or decreased analyte volatility. One type of background correction, Zeeman, can correct for higher and more spectrally complicated background absorption and provide more precise and accurate analytical results. Zeeman background correction uses the principle that the electronic energy levels of an atom placed in a strong magnetic field are changed thereby changing the atomic spectra; the spectral nature of background absorption, on the other hand is unaffected by a magnetic field.

4.2 Nonspectral Interferences

In order for atomic absorption to occur, free atoms of the analyte element must be present in the spectrometer light path. Nonspectral interferences result when diverse components in the sample matrix inhibit the formation of free analyte atoms. An often used approach to compensate for nonspectral interferences is known as the "Method of Standard Additions".

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 milliliter (mL) beaker or other appropriate vessel

6.1.2 Watch glasses

6.1.3 Funnels

6.1.4 Graduated cylinders

6.1.5 Various volumetric flasks (Type A)

6.1.6 Thermometer that covers a range of 0-200°C

6.1.7 Whatman No. 42 filter paper or equivalent

6.1.8 Hot plate, block digester, or other heating source capable of maintaining 92-95°C

6.1.9 Balances - Analytical Balance, 300 gram (g) capacity, and minimum ±0.01 g.

6.2 Atomic Absorption Spectrophotometer - with graphite furnace atomizer and background correction. Hollow Cathode Lamp (HCL) and/or Electrodeless Discharge Lamp (EDL).

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. (Redistilled acids are acceptable.)

7.1.1 Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-77). Use this preparation for all reagents, standards, and dilutions of solutions.

7.1.2 Nitric acid - Concentrated (specific gravity 1.41).

7.1.3 Nitric acid, 5% (v/v) - Add 50 milliliters (mL) conc. HNO_3 to 500 mL reagent water; dilute to 1 Liter (L).

7.1.4 Hydrochloric acid - Concentrated (specific gravity 1.19).

7.1.5 Hydrogen peroxide (30%)

7.1.6 Matrix Modifiers

7.1.6.1 Ammonium Phosphate solution (40%): Dissolve 40 grams (g) of ammonium phosphate, $(NH_4)_2PO_4$ (analytical reagent grade) in reagent water and dilute to 100 mL.

7.1.6.2 Calcium Nitrate solution: Dissolve 11.8 g of calcium nitrate, $Ca(NO_3)_2 \cdot 4H_2O$ (analytical reagent grade) in reagent water and dilute to 100 mL. 1 mL = 20 mg Ca.

7.1.6.3 Lanthanum Nitrate solution: Dissolve 58.64 g of American Chemical Society (ACS) reagent grade 2. La_2O_3 in 100 mL conc. HNO_3 and dilute to 1000 mL with reagent water. 1 mL = 50 mg La.

7.1.6.4 Nickel Nitrate solution, 5%: Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \cdot 6H_2O$ in reagent water and make up to 100 mL.

7.1.6.5 Nickel Nitrate solution, 1%: Dilute 20 mL of the 5% nickel nitrate to 100 mL with reagent water.

7.2 Standards

7.2.1 Introduction

The Contractor must provide all standards 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 or prepared from ultra high purity grade chemicals or metals. 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.) Typical stock solution preparation procedures follow.

7.2.2.2 Arsenic solution, stock [1 mL = 1 mg As (1000 mg/1)] - Dissolve 1.320 g of As_2O_3 in 100 mL of reagent water containing 0.4 g NaOH. Acidify the solution with 20 mL conc. HNO_3 and dilute to 1 L.

7.2.2.3 Lead solution, stock [1 mL = 1 mg Pb (1000 mg/L)] - Dissolve 1.599 g of $Pb(NO_3)_2$ in reagent water. When solution is complete, acidify with 10 mL of conc. HNO_3 and dilute to 1 L with reagent water.

7.2.2.4 Selenium solution, stock [1 mL = 1 mg Se (1000 mg/L)] - Dissolve 0.3453 g of H_2SeO_3 (actual assay 94.6%) in reagent water and make up to 200 mL.

7.2.2.5 Thallium solution stock [1 mL = 1 mg Tl (1000 mg/L)] - Dissolve 1.303 g of $TlNO_3$ in reagent water. Add 10 mL of conc. nitric acid and dilute to 1 L with reagent water.

7.2.3 Working Standards

7.2.3.1 Secondary Dilution Standards

Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions". The calibration standards must be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed after sample preparation.

7.2.3.2 Calibration Blank

Prepared by diluting 1 mL of (1+1) HNO_3 and 2 mL 30% H_2O_2 to 100 mL with reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All samples must be collected in glass or polyethylene containers. Water/aqueous samples must be preserved with nitric acid to pH less than 2 immediately after collection. 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 (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 2 immediately after filtration.

8.2 Procedure for Sample Storage

The 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 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 365 days 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 Conditions

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 must be within the instrument calibrated range. It is the responsibility of the analyst to verify that 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 Graphite Furnace Atomic Absorption (GFAA) Instrument Calibration Procedure

9.2.1 Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument standardization date and time shall be included in the raw data.

9.2.2 Calibration standards shall be prepared fresh daily or each time an analysis is to be made and discarded after use. Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range. One atomic absorption calibration standard shall be at the CRQL. The calibration standards shall be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following sample preparation.

9.2.3 Calibration standards are prepared by diluting the stock metal solutions at the time of analysis. Date and time of preparation and analysis shall be given in the raw data.

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 Sample Management Office (SMO) to inform them of the problem. SMO will contact the Region for instructions. The 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 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 sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the Region. If all phases of the sample are amenable to analysis, the 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 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 Region's decision in the SDG Narrative.

10.1.3 Water/Aqueous Sample Preparation

10.1.3.1 Shake sample and transfer 50-100 mL of well-mixed sample to a 250 mL heating vessel, add 1 milliliter (mL) of (1+1) HNO_3 and 2 mL of 30% H_2O_2 to the sample. Cover with watch glass or similar cover and heat on a hot plate, block digester, or equivalent heating source which is adjustable and capable of maintaining a temperature of 92-95°C for 2 hours or until sample volume is reduced to between 25 and 50 mL, making certain sample does not boil. Cool sample and filter to remove insoluble material.

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.

Adjust sample volume to 50-100 mL with reagent water. The sample is now ready for analysis. Concentrations so determined shall be reported as "total". If volumes less than 100 mL are used, all other reagents shall be reduced appropriately (e.g., if 50 mL is

used, reduce reagent volumes by one-half). The final volume of the digestate must equal the initial volume of the sample aliquot.

10.1.4 Soil/Sediment Sample Preparation

10.1.4.1 A representative 1.0 gram (g) (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Nitric acid is employed as the final reflux acid for the Graphite Furnace Atomic Absorption (GFAA) analysis of As, Pb, Se, and Tl. A separate sample shall be dried for a percent solids determination.

10.1.4.2 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a beaker.

10.1.4.3 Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 92-95°C on hot plate or block digestor, and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watch glass, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.

10.1.4.4 After the second reflux step has been completed and the sample has cooled, add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the heat source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heat vessel.

Continue to add 30% H_2O_2 in 1 mL aliquots 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 30% H_2O_2.

10.1.4.5 If the sample is being prepared for the GFAA analysis of As, Pb, Se, and Tl, continue heating the acid-peroxide digestate until the volume has been reduced to approximately 2 mL, add 10 mL of reagent water, and warm the mixture. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 mL with reagent water.

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.

Dilute the digestate 1:1 (200 mL final volume) with acidified water to maintain constant acid strength. For analysis, withdraw aliquots of appropriate volume, and add any required reagent or matrix modifier. The sample is now ready for analysis.

10.2 Sample Analysis

10.2.1 Set up instrument with proper operating parameters established in Section 9.1.

10.2.2 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using calibration standard solutions.

10.2.3 Instrument Parameters - Suggested Conditions

10.2.3.1 Arsenic

10.2.3.1.1 Wavelength: 193.7 nm

10.2.3.1.2 Operating parameters should be set as specified by the particular instrument manufacturer.

10.2.3.1.3 The use of background correction is required. Background correction made by the deuterium arc method does not adequately compensate for high levels of certain interferents (i.e., Al, Fe). If conditions occur where significant interference is suspected, the laboratory must switch to an alternate wavelength or take other appropriate actions to compensate for the interference effects.

10.2.3.1.4 The use of the Electrodeless Discharge Lamps (EDLs) for the light source is recommended.

10.2.3.2 Lead

10.2.3.2.1 Wavelength: 283.3 nm

10.2.3.2.2 Operating parameters should be set as specified by the particular instrument manufacturer.

10.2.3.2.3 The use of background correction is required.

10.2.3.2.4 Greater sensitivity can be achieved using the 217.0 nm line, but the optimum concentration range is reduced. The use of an EDL at this lower wavelength has been found to be advantageous. Also a lower atomization temperature (2400°C) may be preferred.

10.2.3.2.5 To suppress sulfate interference (up to 1500 ppm), lanthanum is added as the nitrate to both samples and calibration standards.

10.2.3.2.6 Since glassware contamination is a severe problem in lead analysis, all glassware should be cleaned immediately prior to use, and once cleaned, should not be open to the atmosphere except when necessary.

10.2.3.3 Selenium

10.2.3.3.1 Wavelength: 196.0 nm

10.2.3.3.2 Operating parameters should be set as specified by the particular instrument manufacturer.

10.2.3.3.3 Selenium analysis suffers interference from chlorides (>800 mg/L) and sulfate (>200 mg/L). For the analysis of industrial effluents and samples with concentrations of sulfate from 200 to 2000 mg/L, both samples and standards should be prepared to contain 1% nickel.

10.2.3.3.4 The use of the EDL for the light source is recommended.

10.2.3.4 Thallium

10.2.3.4.1 Wavelength: 276.8 nm

10.2.3.4.2 Operating parameters should be set as specified by the particular instrument manufacturer.

10.2.3.4.3 The use of background correction is required.

10.2.3.4.4 Nitrogen may also be used as the purge gas.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Water/Aqueous Sample Calculation

The concentrations determined in the digestate are to be reported in units of microgram per Liter (µg/L):

EQ. 1 Aqueous Sample Concentration

$$\text{Concentration} = C \times \frac{V_f}{V_i}$$

WHERE,

C = Instrument value in µg/L

V_f = Final digestion volume

V_i = Initial digestion volume

11.2 Soil 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 milligram per kilogram (mg/kg):

EQ. 2 Soil Sample Concentration

$$\text{Concentration (dry wt.) (mg/kg)} = \frac{C \times V}{W \times S}$$

WHERE,

C = Concentration (mg/L)

V = Final volume in liters after sample preparation

W = Weight in kg of wet sample

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

11.3 Corrections For Sample Dilutions

If dilutions were performed, the appropriate factor shall be applied to the sample values as follows:

EQ. 3 Correction for Dilution

$$C\ (\mu g/L) = C_i \times DF$$

WHERE, C = Concentration of analyte in sample

C_i = Instrument value concentration

DF = Dilution Factor

12.0 QUALITY CONTROL

For specific Quality Control (QC) requirements, the Contractor shall follow the instructions provided by the USEPA Region requesting the analysis.

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 206.2. March 1983.

16.2 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 239.2. March 1983.

16.3 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 270.2. March 1983.

16.4 US Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes. Method 279.2. March 1983.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Not applicable.

EXHIBIT B

INORGANIC FORMS

USEPA - CLP

COVER PAGE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS 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)	_____	_____

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 computer-readable data submitted on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) 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.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Level: (low/med) _____ Date Received: _____

% Solids: _____

Concentration Units (**ug/L** 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:

_____

_____

_____

_____

1B-IN
INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Level: (low/med) _____ Date Received: _____

% Solids: _____

Concentration Units (**ug/L** or **mg/kg** dry weight): _____

CAS No.	Analyte	Concentration	C	Q	M

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

_____

_____

_____

_____

2A-IN
INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS 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 80-120; Other Metals 90-110; Cyanide 85-115

2B-IN
CRQL CHECK STANDARD

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

CRQL Check Standard Source: _____

Concentration Units: ug/L

Analyte	CRQL Check Standard				
	Initial			Final	
	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: 70-130 with the following exceptions:
ICP-AES - Antimony, Lead, and Thallium: 50-150.
ICP-MS - Cobalt, Manganese, and Zinc: 50-150.

* If applicable, enter the concentration qualifier "J" or "U" after the concentration in these columns (e.g., 0.20U for Mercury).

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3-IN
BLANKS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Preparation Blank Matrix (soil/water): _____

Preparation Blank Concentration Units (ug/L or mg/kg): _____

Analyte	Initial Calibration Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Preparation Blank	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											

4A-IN
ICP-AES INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-AES Instrument ID: _____ ICS Source: _____

Concentration Units: ug/L

Analyte	True		Initial Found				Final Found			
	Sol. A	Sol. AB	Sol. A	%R	Sol. AB	%R	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										

4B-IN
ICP-MS INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ ICS 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						
Carbon						
Chloride						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Molybdenum						
Nickel						
Phosphorus						
Potassium						
Selenium						
Silver						
Sodium						
Sulfur						
Thallium						
Titanium						
Vanadium						
Zinc						

5A-IN
MATRIX SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

% 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.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

Concentration Units: ug/L

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.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

% Solids for Sample: _____ % Solids for Duplicate: _____

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.: _____ NRAS No.: _____ SDG No.: _____

Solid LCS Source: _____

Aqueous LCS Source: _____

Analyte	Aqueous (ug/L)			Solid (mg/kg)					
	True	Found	%R	True	Found	C	Limits		%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									

8-IN
ICP-AES and ICP-MS SERIAL DILUTIONS

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Level: (low/med) _____

Concentration Units: ug/L

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							

9-IN
METHOD DETECTION LIMITS (ANNUALLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Instrument Type: _____ Instrument ID: _____ Date: _____

Preparation Method: _____

Concentration Units (ug/L or mg/kg): _____

Analyte	Wavelength /Mass	CRQL	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:

_____

_____

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_____

10A-IN
ICP-AES INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS 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:

_____

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_____

10B-IN
ICP-AES INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS 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:

_____

_____

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_____

11-IN
ICP-AES and ICP-MS LINEAR RANGES (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP Instrument ID: _____ Date: _____

Analyte	Integ. Time (Sec.)	Concentration (ug/L)	M
Aluminum			
Antimony			
Arsenic			
Barium			
Beryllium			
Cadmium			
Calcium			
Chromium			
Cobalt			
Copper			
Iron			
Lead			
Magnesium			
Manganese			
Nickel			
Potassium			
Selenium			
Silver			
Sodium			
Thallium			
Vanadium			
Zinc			

Comments:

_____

_____

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_____

12-IN
PREPARATION LOG

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

Preparation Method: _____

EPA Sample No.	Preparation Date	Weight (gram)	Volume (mL)

USEPA - CLP

13-IN
ANALYSIS RUN LOG

Lab Name: ______________________ Contract: ______________

Lab Code: ________ Case No.: ________ NRAS 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		

14-IN
ICP-MS Tune

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS No.: _____ SDG No.: _____

ICP-MS Instrument ID: _____ Date: _____

Element - Mass	Avg. Measured Mass (amu)	Avg. Peak Width at 5% Peak Height (amu)	%RSD
Be - 9			
Mg - 24			
Mg - 25			
Mg - 26			
Co - 59			
In - 113			
In - 115			
Pb - 206			
Pb - 207			
Pb - 208			

Comments:

_____

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_____

15-IN
ICP-MS Internal Standards Relative Intensity Summary

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ NRAS 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

SAMPLE LOG-IN SHEET

ab Name	Page __ of __
eceived By (Print Name)	Log-in Date
eceived By (Signature)	
ase Number	Sample Delivery Group No. / NRAS Number

emarks:		EPA Sample #	Aqueous Sample pH	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
. Custody Seal(s)	Present/Absent* Intact/Broken					
. Custody Seal Nos.	_____					
	_____					
. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*					
. Airbill	Airbill/Sticker Present/Absent*					
. Airbill No.	_____					
	_____					
. Sample Tags	Present/Absent*					
Sample Tag Numbers	Listed/Not Listed on Traffic Report/Chain of Custody Record					
. Sample Condition	Intact/Broken*/ Leaking					
. Cooler Temperature Indicator Bottle	Present/Absent*					
. Cooler Temperature	_____					
0. Does information on Traffic Reports/Chain of Custody Records and sample tags agree?	Yes/No*					
1. Date Received at Lab	_____					
2. Time Received	_____					
Sample Transfer						
raction	Fraction					
rea #	Area #					
y	By					
n	On					

Contact SMO and attach record of resolution

eviewed By		Logbook No.
ate		Logbook Page No.

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____

CITY/STATE _____

CASE NO. _____ SDG NO. _____

SDG NOs. TO FOLLOW _____ _____ _____

NRAS 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) (Do not number)				
2.	Sample Log-In Sheet (DC-1)				
3.	Traffic Report/Chain of Custody Record				
4.	Cover Page				
5.	SDG Narrative				
	Inorganic Analysis				
6.	Data Sheet (Form I-IN)				
7.	Initial & Continuing Calibration Verification (Form IIA-IN)				
8.	CRQL Standard (Form IIB-IN)				
9.	Blanks (Form III-IN)				
10.	ICP-AES Interference Check Sample (Form IVA-IN)				
11.	ICP-MS Interference Check Sample (Form IVB-IN)				
12.	Matrix Spike Sample Recovery (Form VA-IN)				
13.	Post-Digestion Spike Sample Recovery (Form VB-IN)				
14.	Duplicates (Form VI-IN)				
15.	Laboratory Control Sample (Form VII-IN)				
16.	ICP-AES and ICP-MS Serial Dilutions (Form VIII-IN)				
17.	Method Detection Limits (Annually) (Form IX-IN)				
18.	ICP-AES Interelement Correction Factors (Quarterly) (Form XA-IN)				
19.	ICP-AES Interelement Correction Factors (Quarterly) (Form XB-IN)				
20.	ICP-AES and ICP-MS Linear Ranges (Quarterly) (Form XI-IN)				
21.	Preparation Log (Form XII-IN)				
22.	Analysis Run Log (Form XIII-IN)				

	PAGE NOs. FROM	TO	CHECK LAB	REGION
23. ICP-MS Tune (Form XIV-IN)	____	____	____	____
24. ICP-MS Internal Standards Relative Intensity Summary (Form XV-IN)	____	____	____	____
25. ICP-AES Raw Data	____	____	____	____
26. GFAA Raw Data (If Applicable)	____	____	____	____
27. ICP-MS Raw Data	____	____	____	____
28. Mercury Raw Data	____	____	____	____
29. Cyanide Raw Data	____	____	____	____
30. Preparation Logs Raw Data	____	____	____	____
31. Percent Solids Determination Log	____	____	____	____
32. USEPA Shipping/Receiving Documents				
Airbill (No. of Shipments ____)	____	____	____	____
Sample Tags			____	____
Sample Log-In Sheet (Lab)	____	____	____	____
33. Misc. Shipping/Receiving Records (list all individual records)				
Telephone Logs	____	____	____	____
_____	____	____	____	____
_____	____	____	____	____
34. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)				
_____	____	____	____	____
_____	____	____	____	____
35. Internal Original Sample Prep & Analysis Records (describe or list)				
Prep Records _____	____	____	____	____
Analysis Records _____	____	____	____	____
Description _____	____	____	____	____
36. Other Records (describe or list)				
Telephone Communications Log	____	____	____	____
_____	____	____	____	____
_____	____	____	____	____

37. Comments: _____

_____

_____

_____

Completed by:

(CLP Lab) _____ _____ _____

(Signature) (Print Name & Title) (Date)

Audited by:

(USEPA) _____ _____ _____

(Signature) (Print Name & Title) (Date)