United States
Environmental Protection
Agency

Office of
Solid Waste and
Emergency Response

9240.1-32
EPA/540/R-96/016
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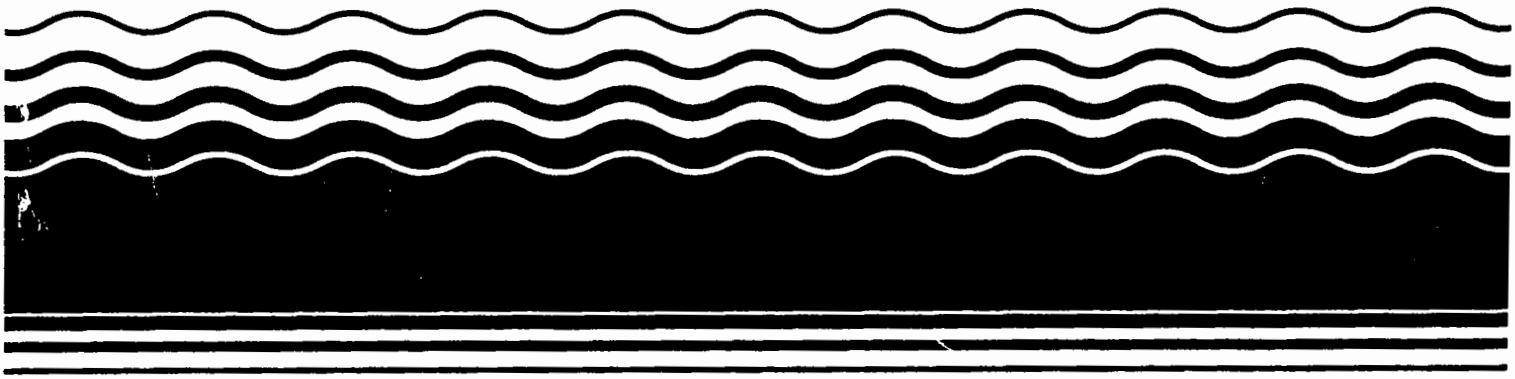
Solid Waste

EPA

Statement of Work for Low Concentration Inorganic Analytes in Water

ILC03.1

Draft Version



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DRAFT

STATEMENT OF WORK

FOR
LOW CONCENTRATION INORGANIC ANALYTES
IN WATER

ANALYTICAL SERVICES FOR SUPERFUND DOCUMENT NO. ILC03.1

U.S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM

CONTENTS

EXHIBIT A: Summary of Requirements

CONTRACTOR OPERATED:

SAMPLE MANAGEMENT OFFICE

The Sample Management Office (SMO) is operated by the Contract Laboratory Analytical Services Support (CLASS) contract awarded and administered by the U.S. Environmental Protection Agency (EPA). Laboratory contractors are advised that wherever in this document the words "Sample Management Office", "SMO", "Contract Laboratory Analytical Services Support" or "CLASS" appear, EPA is referring to those contractor employees. The contract is currently held by DynCorp•Viar under Contract No. 68-D4-0104. Laboratory contractors are also advised that DynCorp•Viar employees are not representatives or agents of EPA. As such, DynCorp•Viar nor its employees, nor any successor contractor, may change, waive, or interpret any terms and conditions in this contract, including this document (ILC03.1). All such questions or inquiries should be addressed to the responsible party within EPA.

QUALITY ASSURANCE TECHNICAL SUPPORT LABORATORY

The Quality Assurance Technical Support (QATS) Laboratory contract was awarded and is administered by the U.S. Environmental Protection Agency (EPA). Laboratory contractors are advised that wherever in this document the "Quality Assurance Technical Support Laboratory" or "QATS" appear, EPA is referring to those employees. The contract is currently held by ICF Kaiser Engineers, Inc. (ICF), under Contract No. 68-D5-0002. Laboratory contractors are also advised that ICF employees are not representatives or agents of EPA. As such, ICF nor its employees, nor any successor contractor, may change, waive, or interpret any terms and conditions in this contract, including this document (ILC03.1). All such questions or inquiries should be addressed to the responsible party within EPA.

SECTION I: General Requirements

1. The data generated using the procedures in this Statement of Work (SOW), with its associated quality control procedures, criteria, and documentation, will be used for several purposes in the Environmental Protection Agency (EPA) Superfund Program.

2. The data quality must meet the Office of Drinking Water (Safe Drinking Water Act) Standards, including Maximum Contamination Levels (MCLs) for drinking water and groundwater. In addition, the data will be used for performing risk and health assessments in order to determine the appropriateness of Superfund remedy options. The data will also be used in public health assessments, where it is necessary to provide data to support a proposed risk range of 10^{-4} to 10^{-7} Cancer Risk Factors. Information may also be used for Superfund enforcement and litigation and in cost recovery. Finally, these data will be used to ascertain compliance with ambient water quality criteria. Because of the multidimensional use of the data, and its importance in meeting these goals, it is imperative that the data meet the contract requirements.

3. Under this SOW, Contractor laboratories (herein called Contractor or Laboratory) shall employ procedures specified here in the preparation and analysis of low concentration water samples for the presence and quantitation of 23 indicated elements, cyanide.

4. This Exhibit (A) summarizes the requirements, and Exhibits B-G present specific information. Because of the low detection levels that are required under this SOW, the Contractor shall exercise caution during the preparation, analysis, and storage of the samples to prevent contamination. Following sample analysis, the Contractor shall perform data reduction and shall report analytical activities, sample data, and quality control documentation as designated in Exhibit B.

5. The Contractor shall use proven instruments and techniques to identify and measure the elements and inorganic species presented in the Target Analyte List (TAL) (Exhibit C). The Contractor shall perform sample preparation and analysis procedures as prescribed in Exhibit D, meeting specified sample preservation and holding time requirements.

6. The Contractor shall adhere to the quality assurance/quality control (QA/QC) protocol specified in Exhibit E for all samples analyzed under this contract.

7. Exhibit F contains chain-of-custody and document control requirements that the Contractor shall follow in processing samples and specifies requirements for written laboratory standard operating procedures.

8. To ensure proper understanding of language utilized in this contract, Exhibit G contains a glossary of terms. When a term is used in the text without explanation, the glossary meaning shall be applicable. Glossary definitions do not replace or take precedence over specific information included in the SOW text.

SECTION II: Specific Requirements

Part A - Task Performance

For each sample, the Contractor shall perform the following tasks:

Task I: Receive and Prepare Low Concentration Water Samples

1. Chain-of-Custody. The Contractor shall receive and maintain samples under proper chain-of-custody and sample documentation procedures described in Exhibit F. A sample consists of all components, contained inside appropriate receptacles. Containers may be glass or plastic. More than one container may be used for a single sample; individual containers may contain preservatives for different analysis portions. All associated document control and inventory procedures shall be developed and followed. Documentation, as described therein, shall be required to show that all procedures are being strictly followed. This documentation shall be reported as the Complete Sample Delivery Group File (CSF) (see Exhibit B). The Contractor shall establish and use appropriate procedures to handle confidential information received from the Agency.

2. Sample Scheduling/Shipments. Sample shipments to the Contractor's facility will be scheduled and coordinated by the EPA Contract Laboratory Program (CLP) Sample Management Office (SMO). 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.

2.1 Samples will routinely be shipped directly to the Contractor through a delivery service. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including Saturdays and holidays. As necessary, the Contractor shall be responsible for any handling or processing for the receipt of sample shipments, including pick-up of samples at the nearest servicing airport, bus station, or other carrier service within the Contractor's geographical area.

2.2 The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation expressed in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

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

2.4 The Contractor shall be required to routinely return sample shipping containers (i.e., coolers) to the appropriate sampling office within

fourteen (14) calendar days following shipment receipt (see Clause entitled Government Furnished Supplies and Materials).

2.5 If there are problems with the samples (e.g., mixed media, containers broken or leaking) or the sample documentation/paperwork (e.g., Traffic Reports not with shipment, or sample and Traffic Report do not correspond), the Contractor shall immediately notify SMO regarding any problems and/or laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date. The Contractor shall document all communications with SMO and/or the Regional representative(s) in the SDG narrative.

3. All samples and blanks submitted to the Contractor should have measured amounts of preservatives, i.e., 2 mL 1:1 HNO_3/L to pH < 2 as recorded on the Traffic Report. Before sample preparation is initiated on a sample received in shipment, the Contractor shall check the pH of the sample and note the pH in the laboratory's preparation log. The laboratory shall verify that the pH is <2 for the metals sample, and that the pH is > 12 for cyanide samples, as appropriate. If the sample has not been properly preserved (i.e., the pH of the metals samples is >2, and the pH of the cyanide samples is <12), the Contractor shall immediately contact SMO. SMO will contact the Region from which the samples were shipped for instructions on how to proceed. The Region will either require that the pH be adjusted, and the analysis(es) be performed or that the Contractor proceed with the analysis(es). SMO will notify the Contractor of the Region's decision. The Contractor shall document the Region's decision and list the EPA sample number for all affected samples in the SDG narrative.

4. The Contractor shall prepare samples as described in Exhibit D. If dissolved metals are required by the EPA Region, the Contractor shall follow the instructions provided on the Traffic Report(s). If there are no instructions on the Traffic Report(s), the Contractor shall digest the samples designated as dissolved metals. If the Regional office indicates on the Traffic Report(s) that a digestion is not to be performed when analyzing field samples for dissolved metals, then a laboratory control sample (LCS) (Form IX) is not required. Dissolved metals samples that are not digested shall be matrix-matched to instrument calibration standards. Matrix matching shall be applied without affecting the original sample volume by more than 10 percent.

5. The Contractor shall prepare and analyze samples within the maximum holding time specified in Section II of Exhibit D even if these times are less than the maximum data submission time allowed in this contract.

6. To more effectively monitor the temperature of the sample shipping cooler, 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.

6.1 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.

6.2 The temperature of the sample shipping cooler shall be measured and recorded immediately upon opening the cooler, and prior to unpacking the samples or removing the packing material.

6.3 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 shall have a measurable range of 0 to 50 degrees Celsius.

6.4 If the temperature of the sample shipping cooler's temperature indicator exceeds 10 degrees Celsius, 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 in the SDG narrative. Also, in the SDG narrative, the Contractor shall list, by the USEPA sample number, all samples which were shipped in a cooler which exceeded 10 degrees Celsius.

6.5 The Contractor shall record the temperature of the cooler on the DC-1 Form, under Remark #8 - Sample Conditions, and in the SDG narrative.

Task II: Analyze Samples for Identification and Quantitation of Specific Inorganic Constituents

1. Aliquots and digestates prepared in Task I shall be analyzed by the analytical procedures described in the methodologies given in Exhibit D. The documentation that accompanies the sample(s) to the Contractor facility shall indicate specific analytical requirements for that sample or set of samples.

2. Exhibit D contains instructions and references for preparation of samples containing low concentrations of inorganics for inductively coupled plasma - atomic emission spectroscopy (ICP), inductively coupled plasma - mass spectrometry (ICP-MS), graphite furnace, flame, and cold vapor atomic absorption (AA), and cyanide (CN) analyses. The identification and quantitation of each analyte shall be accomplished using the appropriate methods as specified by the following:

Analyte	Approved Analytical Method
CN	Manual and semiautomated colorimetric
Hg	Cold vapor AA
Sb, As, Se, Zn, V, Tl, Ag, Ni, Fe, Cu, Co, Cr, Cd, Be, Ba, Al Mn, Pb	ICP, ICP-MS, furnace AA

Ca, Mg, K, Na	ICP, ICP-MS, flame AA, furnace AA

3. All samples shall initially be run undiluted (i.e., the final product of the sample preparation procedure). When an analyte concentration exceeds the calibrated or linear range, appropriate dilution (but not below the CRDL) and reanalysis of the sample is required, as specified in Exhibit D. The Contractor should maintain the same acid concentration in the diluted sample as was present in the undiluted sample used for analysis.

4. For the purpose of this contract, a full sample analysis is defined as analysis for all of the TAL constituents identified in Exhibit C in accordance with the methods in Exhibit D, and performance of related QA/QC as specified in Exhibit E. All QA/QC required sample analyses, including Duplicate sample, spike sample, and Laboratory Control Sample (LCS) analyses are considered an inherent part of this SOW and are included in the contract sample unit price.

Task III: Perform Required Quality Assurance/Quality Control Procedures

1. All specific QA/QC procedures prescribed in Exhibit E shall be strictly adhered to by the Contractor. 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 Exhibit B requirements.

2. The Contractor shall establish and use on a continuing basis the QA/QC procedures in Exhibit E. These procedures include the use of standard reference materials at the required frequency where available at appropriate concentrations, from EPA, the National Institute of Standards and Technology or secondary standards traceable thereto, where available at appropriate concentrations (i.e., standard solutions designed to ensure that operating parameters of equipment and procedures, from sample receipt through identification and quantitation, produce reliable data). Exhibit E specifies the QA/QC procedures required.

3. The Contractor shall maintain a Quality Assurance Plan (QAP) as defined in Exhibit E 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. The Performance Evaluation Sample (PES) is an external laboratory QC sample prepared by the Agency to assess, on the basis of Sample Delivery Group (SDG), the baseline capability of the Contractor to perform the analytical methods listed in Exhibit D.

4.1 The PES will be received from the Agency in ampules or as full volume samples. If they are received in ampules, the Contractor will receive instructions concerning the dilution procedure to bring the samples to full volume prior to preparation and analysis.

4.2 The PES shall be prepared and analyzed concurrently with each SDG, when available and provided by the Agency. The PES may be provided as a

single-blind QC sample (i.e., the Contractor will know that the sample is a PES, but will not know which analytes are in the PES or their concentration) or as a double-blind QC sample (i.e., the Contractor will receive a PES as a full volume sample which will appear to be an environmental field sample).

4.3 The Contractor will prepare and analyze the PES according to requirements listed in Exhibit D, and report the results according to the requirements in Exhibit B.

4.4 The Contractor will be responsible for correctly identifying and quantifying the analytes included in the PES. The Agency will notify the Contractor of unacceptable performance. Exhibit E defines acceptable performance and describes the corrective action procedures required under this contract.

5. The Laboratory Control Sample (LCS) is an internal laboratory QC sample designed to assess, on a SDG-by-SDG basis, the baseline capability of the Contractor to perform the analytical methods listed in Exhibit D. The Contractor shall prepare and analyze an LCS once per SDG, concurrently with samples in the SDG.

Part B - Reporting

1. EPA has provided the Contractor a format for the reporting of data (Exhibit B). The Contractor shall be responsible for completing and returning analysis data sheets in the format specified in this SOW and within the time specified in the Contract Performance/Delivery Schedule (see Exhibit B).

2. Use of formats other than those designated by EPA will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to the government will be required.

3. Computer-generated forms may be submitted in the hard copy data package(s) provided that the forms are in EXACT EPA FORMAT. This means that the order of data elements is the same as on each EPA-required form, including form numbers and titles, page numbers and header information, columns and lines.

Part C - Systems

1. The Contractor shall provide analytical equipment and technical expertise for the analyses of TAL analytes at concentrations equal to or lower than the contract required detection limits specified in Exhibit C. The Contractor shall maintain, at a minimum, the following:

1.1 ICP emission spectrometer, with the capability to analyze metals sequentially or simultaneously,

1.2 AA spectrometer equipped with graphite furnace, flame, and cold vapor AA (or a specific mercury analyzer) analysis capabilities. Note: Deuterium background may not meet all of the analytical requirements of this contract.

2. The Contractor shall maintain a complete system(s) applicable to the determination of cyanide.

3. Analytical equipment and apparatus for the determination of analytes not analyzed by the above instrumentation (e.g., microwave digestion units, hot plates, a block digester, wet chemistry apparatus, spectrometers, etc.). In addition, the following list of optional equipment may be used if the instrumentation can meet the required CRDLs and QC requirements listed in Exhibits C and E, respectively:

3.1 An ICP-MS with the capability to analyze metals.

Part D - Functions and Operations

1. The Contractor shall designate and utilize qualified key personnel to perform the functions specified in this SOW.

2. EPA reserves the right to review personnel qualifications and experience.

3. The Contractor shall respond within 7 days to written requests from data recipients for additional information or explanations that result from the government's inspection activities, unless otherwise specified in the contract (see Exhibit E for details on Government inspection activities).

4. The Contractor is required to retain unused sample volumes and used sample containers for a period of 60 days after data submission.

5. 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. A Case may consist of one or more SDG(s). An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received, or
- Each 20 field samples within a Case, or
- Each 7 calendar day period during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

5.1 Data for all samples in an SDG shall be submitted together, including the PES, in one package and in the order specified in Exhibit B. The SDG number is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number is the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. The SDG number is reported on all data reporting forms. The SDG Receipt Date is the day that the last sample in the SDG is received.

5.2 The Contractor is responsible for identifying each SDG as samples are received, through proper sample documentation (see Exhibit B) and communication with SMO personnel.

6. Each sample, including the PES, received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report form bearing the sample number and descriptive information regarding the sample. EPA field 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, 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 in choosing the QC samples when such information is provided.

6.1 The Contractor shall submit signed copies of Traffic Reports for all samples in an SDG to SMO within FIVE (5) WORKING days following receipt of the last sample in the SDG. Traffic Reports shall be submitted in SDG sets (i.e., all Traffic Reports for a SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

7. EPA Case numbers (including SDG numbers) and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally, in reports, and correspondence.

SECTION III: Technical and Management Requirements

1. Personnel - The Contractor shall have adequate personnel at all times during the performance of the contract to ensure that EPA receives data that meet the terms and conditions of the contract.

2. Instrumentation - The Contractor shall have sufficient inductively coupled plasma (ICP) emission spectrometers with the capability to analyze metals sequentially or simultaneously, atomic absorption (AA) spectrometers equipped with graphite furnace, flame, and cold vapor AA (or specific mercury analyzers) analysis capabilities or equivalent for the analysis of metals, and analytical equipment/apparatus for analysis of cyanide as described in Exhibit D to meet all the terms and conditions of the contract.

3. Facilities - The Contractor shall maintain a facility suitable for the receipt, storage, analysis, and delivery of the product meeting the terms and conditions of the contract.

EXHIBIT B: Reporting and Deliverables Requirements

SECTION I: Contract Reports and Deliverables Distribution

The following table reiterates the Contract reporting and deliverables requirements specified in the Contract Schedule and specifies the distribution that is required for each deliverable.

Item	No. Copies	Delivery Schedule	Distribution† (1)	(2)	(3)
*****A. Standard Operating Procedures	1	60 days after contract award, and as required in Exhibit E	As Directed		
B. Sample Traffic Reports	1	5 working days after receipt of last sample in Sample Delivery Group (SDG)***		X	
**C. Sample Data Package	1	14 days after receipt of last sample in SDG	X		#
****D. Complete SDG File	1	14 days after receipt of last sample in SDG**		X	
*E. Quarterly/Annual Verification of Instrument Parameters	2	Quarterly: 15th day of January, April July, October	X	X	
*F. ICP-MS Diskettes/Tapes	Lot	Retain for 365 days after submission; or submit them within 7 days of written request by APO or EMSL-LV	As directed		
*****G. Quality Assurance Plan	1	60 days after contract award, and as required in Exhibit E	As directed		

†Distribution

(1) USEPA Contract Laboratory Program (CLP)
Sample Management Office (SMO)[1]
P. O. Box 818
Alexandria, VA 22313

For overnight delivery service, use street address:

300 N. Lee Street, 5th Floor
Alexandria, VA 22314

[1] The Sample Management Office (SMO) is a contractor-operated facility operating under the CLASS contract.

(2) USEPA REGIONS: The CLP SMO, acting on behalf of the Administrative Project Officer (APO), will provide the Contractor with the list of addressees for the ten EPA Regions. SMO will provide the Contractor with updated regional name and address lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

(3) 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

[2] The Quality Assurance Technical Support (QATS) Laboratory is a contractor-operated facility.

Footnotes:

Contractor concurrent delivery to QATS may be required upon written request by the Regional Technical Project Officer (TPO) or APO. The Contractor shall retain a copy of the sample data package for 365 days after final data acceptance of the reconciled data package, and submit within seven (7) days after receipt of written request by the TPO or APO.

* Also required in each Sample Data Package.

** Concurrent delivery of these items to all recipients is required.

*** A Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. (See SOW Exhibit A, paragraph I., for further description).

**** A Complete SDG File will contain the sample data package plus all of the original documents described in Exhibit B under "Complete SDG File."

The Complete SDG File must be delivered concurrently with the Sample Data Package.

***** See Exhibit E for a more detailed description.

Note 1: Specific recipient names and addresses are subject to change during the term of the contract. The APO will notify the Contractor in writing of such changes when they occur.

Note 2: As specified in the Contract Schedule (Government Furnished Supplies and Materials), unless otherwise instructed by the Contract Laboratory Program (CLP) SMO, the Contractor shall dispose of unused sample volume and used sample bottles or containers no earlier than sixty (60) days following submission of analytical data. Sample disposal and disposal of unused sample bottles or containers is the responsibility of the Contractor and should be done in accordance with all applicable laws and regulations governing the disposal of such material.

SECTION II: Report Descriptions and Order of Data Deliverables

1. The Contractor shall provide reports and other deliverables as specified in the Contract Performance and Delivery Schedule (see Contract Schedule). 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 in ascending order, and
- Double-sided.

2. If submitted documentation does not conform to the above criteria, the Contractor shall be required to resubmit such documentation with the deficiency(ies) corrected, at no additional cost to the government.

3. Hold status may be applied by the APO due to any technical or administrative contractual deficiency. During contract hold, samples may not be scheduled with the Contractor. Reasons for contract hold include, but are not limited to, PES results, laboratory Contract Compliance Screening (CCS) results, laboratory audit results, laboratory instrument or personnel deficiencies, and late data.

4. The Contractor must be prepared to receive the full monthly sample contract requirement at the time of contract award.

5. When the Contractor is required to submit or resubmit data because of an on-site laboratory evaluation, a CCS assessment, an APO or Technical Project Officer (TPO) action, or a regional data reviewer's request, the data shall be clearly marked "Additional Data," and shall be sent to the following contractual data recipients:

- Sample Management Office (SMO)
- Client Region.

5.1 A cover letter shall be included that describes which data are being delivered, to which EPA case(s) the data pertain, and who requested the data.

6. When the Contractor is required or requested to respond to CCS review by SMO, the laboratory response shall be sent to the contractual data recipients (SMO, and the client Region). Each response shall be accompanied by a color-coded Cover Sheet (Laboratory Response to Results of Contract Compliance Screening), which shall be provided in generic format by SMO.

7. Section IV of this Exhibit contains the required Inorganic Analysis Data Reporting Forms in Agency-specified formats. Section III of this Exhibit contains instructions for completing the data reporting forms that must be provided to the Agency.

8. Descriptions of the requirements for each deliverable item cited in the Contract Performance and Delivery Schedule (see Contract Schedule) are specified in Parts A-F of this Section. Items submitted concurrently shall be arranged in the order listed. Additionally, the components of each deliverable item shall be arranged in the order presented herein when the item is submitted.

Part A - Standard Operating Procedures (SOPs) and Quality Assurance Plan (QAP)

1. Submit updated SOPs and QAPs according to the instructions in Exhibit E.

Part B - Sample Traffic Reports

1. The original Sample Traffic Report page marked "Lab Copy for Return to SMO," with laboratory receipt information and original Contractor signature, shall be submitted for each sample in the SDG.

2. Traffic Reports (TRs) shall be submitted in Sample Delivery Group (SDG) sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached.

3. The SDG Cover Sheet shall contain the following items:

- Laboratory name
- Contract number
- Sample Analysis Price - full sample price from contract.
- Case Number
- List of EPA sample numbers of all samples in the SDG, identifying the first and last samples received, and their dates of receipt at the laboratory.

Note: When more than one sample is received in the first or last SDG shipment, the "first" sample received is the lowest sample number (considering alpha and numeric designations); the "last" sample received is the highest sample number (considering both alpha and numeric designations).

4. Each TR shall be clearly marked with the SDG number, which is the sample number of the first sample in the SDG (as described in the following paragraph). This information shall be entered below "Lab Receipt Date" on the TR. In addition, the TR for the last sample received in the SDG shall be clearly marked "SDG - FINAL SAMPLE."

5. The EPA sample number of the first sample received in the SDG is the SDG number. EPA field sample numbers contain six digits. If the Contractor receives a sample number of any other length, the Contractor shall contact SMO immediately. When more than one sample is received in the first SDG shipment, the SDG number shall be the lowest sample number (considering alpha and numeric designations) in the first group of samples received under the SDG. (The SDG number is also reported on all data reporting forms. See Section III, Form Instruction Guide.)

6. If samples are received at the laboratory with multisample Traffic Reports (TRs), all the samples on one multisample TR may not be in the same SDG. In this instance, the laboratory shall submit a copy of the TR with each SDG cover sheet.

Part C - Sample Data Package

1. The sample data package shall include data for analysis of all samples in one SDG, including but not limited to analytical samples, field samples, reanalyses, blanks, spikes, duplicates, Laboratory Control Samples (LCSs), and Performance Evaluation Samples (PESs). The sample data package shall be complete before submission. It shall be consecutively paginated in ascending order, and shall include the following.

2. The Cover Page for the LC-Inorganic Analyses Data Package (COVER PAGE - LCIN), shall include:

- Laboratory name,
- Laboratory code,
- Contract number,
- Case No.,
- SDG No.,
- EPA sample numbers in alphanumeric order, showing EPA sample numbers cross-referenced with laboratory ID numbers, and,
- Completion of the statement on use of inductively coupled plasma (ICP) background and elemental expression corrections for the samples.

2.1 The Cover Page shall contain the following statement, *verbatim*: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. Release of the data contained in this hard copy data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature." This statement shall be followed by the signature of the Laboratory Manager or the Manager's designee with a typed line below it containing the signer's name, title, and date of signature.

3. The Comments Page [COMMENTS PAGE - LCIN] shall include:

- details of the problems encountered in processing the samples in the data package,
- details of technical or administrative problems encountered,
- corrective action taken, and
- resolution of the problem.

The Contractor shall retain a copy of the Sample Data Package for 365 days after final acceptance of the reconciled data package. After this time, the Contractor may dispose of the package.

NOTE: The use of the terms "Comments Page" and "Case Narrative" are used interchangeably in this document, when one or the other is used it is meant to refer to the form ([COMMENTS PAGE - LCIN]) in Exhibit B, Section IV - Data Report Forms.

4. Sample Data: Sample data shall be submitted with the Low Concentration Inorganic Analysis Data Reporting Forms for all samples in the SDG, including

the PES, arranged in increasing alphanumeric EPA Sample Number order, followed by the QC analyses data, and Verification of Instrument Parameters forms, raw data, and copies of the preparation logs.

4.1 Results -- Low Concentration Inorganics Analysis Data Sheet [FORM I - LCIN]:

4.1.1 Tabulated analytical results (identification and quantitation) of the specified analytes (Exhibit C). The validation and release of these results are authorized by a specific, signed statement on the Cover Page. If the Laboratory Manager cannot verify all data reported for each sample, he/she shall provide a detailed description of the problems associated with the sample(s) on the Comments Page.

4.1.2 The quantitative values shall be reported in units of micrograms per liter (ug/L) for all samples. No other units are acceptable. Analytical results shall be reported to two significant figures if the result value is less than 10; to three significant figures if the value is greater than or equal to 10.

4.2 Quality Control Data

4.2.1 Initial and Continuing Calibration Verification [FORM II-LCIN]

4.2.2 Contract Required Detection Limit (CRDL) Standards [FORM III-LCIN]

4.2.3 Linear Range Determination Standards [FORM IV-LCIN]

4.2.4 Blanks [FORM V-LCIN]

4.2.5 ICP-AES and ICP-MS Interference Check Sample Results [FORM VI-LCIN]

4.2.6 Spike Sample Recovery [FORM VII-LCIN]

4.2.7 Duplicates [FORM VIII-LCIN]

4.2.8 Laboratory Control Sample [FORM IX-LCIN]

4.2.9 Serial Dilution [FORM X-LCIN]

4.2.10 Standard Addition Results [FORM XI-LCIN]

4.2.11 Instrument Detection Limits [FORM XII-LCIN]

4.2.12 ICP-AES and ICP-MS Elemental Expression (A) Factors [FORM XIII-LCIN]

4.2.13 ICP-AES and ICP-MS Elemental Expression (B) Factors [FORM XIV-LCIN]

4.2.14 ICP-AES and ICP-MS Tuning and Response Factor Criteria [FORM XV-LCIN] [FORM XVI-LCIN]

4.2.15 ICP-MS Internal Standards Relative Intensity Summary [FORM XVI-LCIN]

4.2.16 Analysis Run Log (A) [FORM XVII-LCIN]

4.2.17 Analysis Run Log (B) [FORM XVIII-LCIN]

4.2.18 Standard Solutions Sources [FORM XIX-LCIN]

4.2.19 Sample Log-In Sheet [FORM DC-1]

4.2.20 Document Inventory Sheet [FORM DC-2]

4.3 Raw Data: For each reported value, the Contractor shall include in the data package all raw data from the instrument used to obtain that value and the QA/QC values reported (except for raw data for quarterly and annual verifications of instrument parameters). Raw data shall contain all instrument readouts used for the sample results, including those readouts that may fall below the IDL. All instruments shall provide a legible hard copy of the direct, real-time instrument readout (i.e., strip charts, printer tapes, etc.). A photocopy or other accurate facsimile of the direct instrument readout shall be included.

4.3.1 The order of raw data in the data package shall be: ICP-AES, ICP-MS, flame atomic absorption (AA), graphite furnace AA (GFAA), mercury, and cyanide.

4.3.2 All raw data shall include intensities or concentration for ICP-AES, ICP-MS, absorbance or concentration for AA, and spectrophotometric measurements.

4.3.3 Raw data shall be labeled with an EPA Sample Number and appropriate codes, specified in Table B-1, to unequivocally identify 4.3.3.1 through 4.3.3.12.

TABLE B-1 CODES FOR LABELING DATA

Sample	XXXXXX
Duplicate	XXXXXXD
Matrix Spike	XXXXXS
Serial Dilution	XXXXXXL
Analytical (Post Digestion) Spike	XXXXXXA
Method of Standard Additions:	
Zero Addition	XXXXXX0
First Addition	XXXXXX1
Second Addition	XXXXXX2
Third Addition	XXXXXX3
Instrument Calibration Standards:	
ICP-AES, ICP-MS,	S or S0 for blank standard
graphite furnace AA and Cyanide	0, 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
CRDL Standard	CRI
Laboratory Control Samples	LCS
Preparation Blank	PBW
Linear Range Analysis Standard	LRS
Memory Test Solution	MTS
Tuning Solution	TS

1.1 When an analytical spike or MSA is performed on samples other than field samples, the "A", "0", "1", "2" or "3" suffixes must be the last to be added to the EPA Sample Number. For instance, an analytical spike of a duplicate must be formatted "XXXXXXDA".

1.2 The numeric suffix that follows the "S" suffix for the standards indicates the true value of the concentration of the standard in ug/L.

1.3 ICP 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 must be formatted "S0".

4.3.3.1 Calibration standards, including source and preparation date.

4.3.3.2 Initial and continuing calibration blanks and preparation blanks.

4.3.3.3 Initial and continuing calibration verification standards, interference check samples, CRDL standards, LCS solutions, linear range standards, tuning standards, memory test solutions, and serial dilution samples.

4.3.3.4 Diluted and undiluted samples (by EPA Sample Number) and all dilutions and volumes used to obtain the reported values. (If the volumes and dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters may be reported in the SDG Narrative).

4.3.3.5 Duplicates.

4.3.3.6 Spikes (indicating standard solutions used, final spike concentrations, volumes involved). If spike information (source, concentration, volume) is consistent for a given SDG, a general statement outlining these parameters may be reported in the SDG Narrative).

4.3.3.7 Instrument and background correction used, any instrument adjustments, data corrections, or other apparent anomalies in the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation in the SDG Narrative.

4.3.3.8 All information, for flame AA and furnace AA analysis, clearly and sequentially identified in the raw data, including EPA Sample Number and date of analysis, sample and analytical spike data, percent recovery, coefficient of variation, full Method of Standard Additions (MSA) data, MSA correlation coefficient, slope and y intercept of linear fit, and final sample concentration (standard addition concentration).

4.3.3.9 All ICP-MS tuning and mass calibration data, in addition to all internal standard results including the elements and concentration used.

4.3.3.10 Time and date of each analysis. Instrument run logs may be submitted if they contain this information. If the instrument does not automatically provide times of analysis, they shall be entered manually on all raw data for initial and continuing calibration verification and blanks, as well as on data for tuning solutions, CRDL standards, interference check samples, and the linear range standard.

4.3.3.11 Integration times for all analyses.

4.4 Preparation Logs: Preparation Logs shall be submitted in the following order: ICP-AES, ICP-MS, flame AA, graphite furnace AA, mercury, and cyanide. These logs shall include:

- Preparation date,
- Sample volume (initial and final),
- Sufficient information to unequivocally identify which QC samples (i.e., laboratory control sample (LCS), preparation blank) correspond to each batch prepared,
- Comments describing any significant sample changes or reactions that occurred during preparation, and
- Report pH <2 or >12, as applicable.

5. The Contractor shall provide a copy of the Sample Traffic Report described in Part B for all of the samples in the SDG. The Traffic Reports shall be arranged in increasing alphanumeric EPA Sample Number order.

Part D - Complete Sample Delivery Group (SDG) File (CSF)

1. As specified in the Delivery Schedule, one CSF, including the original Sample Data Package, shall be delivered to the Region concurrently with delivery of a copy of the Sample Data Package to SMO. The contents of the CSF will be numbered according to the specifications described in Section III and IV of Exhibit B. The Document Inventory Sheet, Form DC-2, is contained in Section IV. The CSF will contain all original documents where possible. Copies will not be placed in the CSF unless the original documents are bound in a logbook maintained by the laboratory. The CSF will contain all original documents specified in Section III and IV, and Form DC-2 of Exhibit B of the SOW.

2. The CSF will consist of the following original documents in addition to the documents in the Sample Data Package.

2.1 Original Sample Data Package (See Exhibit B, Section II, Part C)

2.2 A completed and signed Document Inventory Sheet (Form DC-2)

2.3 All original shipping documents including, but not limited to, the following documents:

2.3.1 EPA Chain-of-Custody Record.

2.3.2 Airbills.

2.3.3 EPA (SMO) Traffic Reports.

2.3.4 Sample Tags (if present) sealed in plastic bags.

2.4 All original receiving documents including, but not limited to, the following documents:

2.4.1 Form DC-1.

2.4.2 Other receiving forms or copies of receiving logbooks.

2.4.3 SDG Cover Sheet.

2.5 All original laboratory records of sample transfer, preparation, and analysis including, but not limited to, the following documents:

2.5.1 Original preparation and analysis forms or copies of preparation and analysis logbook pages.

2.5.2 Internal sample and sample extract transfer chain-of-custody records.

2.5.3 All instrument output, including strip charts from screening activities.

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

2.6.1 Telephone contact logs.

2.6.2 Copies of personal logbook pages.

2.6.3 All handwritten Case-specific notes.

2.6.4 Any other Case-specific documents not covered by the above.

2.6.4.1 Note that 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 EPA, as well as copies that are altered in any fashion, are also deliverables to EPA (original to the Region and a copy to SMO).

2.6.4.2 If the laboratory submits Case-specific documents to EPA after submission of the CSF, the documents should be numbered as an addendum to the CSF and a revised DC-2 Form should be submitted; or the documents should be numbered as a new CSF and a new DC-2 Form should be submitted to the Region only.

Part E - Quarterly and Annual Verification of Instrument Parameters

1. The Contractor shall perform and report quarterly verification of instrument detection limits by methods specified in Exhibit E for each instrument used under this contract. For the ICP-AES and ICP-MS instrumentation, the Contractor shall also perform and report annually elemental expression factors (including method of determination), elemental expressions used, and integration times. Forms for quarterly and annual verification of instrument parameters for the current year shall be submitted in each SDG data package, on Forms XII and XIII as specified in Section III of this Exhibit. Submission of quarterly and annual verification of instrument parameters shall include the raw data used to determine those values reported.

2. If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to, the following actions:

- reduction in the number of samples sent under the contract,
- suspension of sample shipment to the Contractor,
- ICP-MS tape audit (if appropriate),
- data package audit,
- on-site laboratory evaluation,
- remedial performance evaluation sample, and/or
- contract sanctions, such as a Cure Notice.

Part F - Results of Laboratory Control Sample (LCS)

1. Analytical results and QC for the method reference sample analysis, as specified in Exhibit E, shall be tabulated on Form IX.

Part G - Results of Performance Evaluation Sample (PES)

1. Analytical results for the PES analysis, as specified in Exhibit E, shall be tabulated on Form I.

SECTION III - Form Instruction Guide

This section contains specific instructions for the completion of all required Inorganic Data Reporting Forms. This section is organized into the following Parts:

Part A - General Information and Header Information

1. Values must be reported on the hard copy data reporting forms according to the individual form instructions in this Section. Each form submitted must be filled out completely for all analytes and samples before proceeding to the next form of the same type. Multiple forms cannot be submitted in place of one form if the information on those forms can be submitted on one form.

2. All typed characters that appear on the data reporting forms presented in the contract (Exhibit B, Section IV) must be reproduced by the Contractor when submitting data, and the format of the forms submitted must 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 EPA Administrative Project Officer (APO). The names of the various fields and analytes (i.e., "Lab Code," "Aluminum") must appear as they do on the forms in the contract, including the options specified in the form.

3. Five pieces of information are common to the header sections of each data reporting form. These are: Lab Name, Contract, Lab Code, Case No., and SDG No. This information must be entered on every form and must match on all forms.

3.1 "Lab Name" is the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.

3.2 "Contract" is the number of the EPA contract, including hyphens, under which the analyses were performed.

3.3 "Lab Code" is an alphabetic code of up to 6 characters, to identify the laboratory. This code shall be assigned by the EPA at the time a contract is awarded, and must not be modified by the Contractor, except at the direction of EPA.

3.4 "Case No." is the EPA-assigned Case number (5 spaces maximum) associated with the sample and reported on the Traffic Report (TR).

3.5 "SDG No." is the EPA Sample Number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number must be the lowest alphanumeric sample number in the first group of samples received under the SDG.

4. "EPA Sample No." is common to several of the forms.

4.1 This number appears in the upper right-hand corner of the form or at the left column of a table summarizing data from a number of samples. When "EPA Sample No." is entered into a triple-spaced box in the upper right-hand corner of a form, it must be centered.

4.2 All field samples and quality control (QC) samples must be identified with an EPA Sample Number. For field samples, the EPA Sample Number is the unique identifying number given in the TR that accompanied that sample.

The QC samples abbreviations listed in Table B-1 must be used as appropriate.

5. "Run No." refers to a number assigned to a collective sequential analytical run which starts, ends, and encompasses all of the QA/QC relevant to the analytical run from Exhibit E. A run number of 1 is assigned to the chronologically oldest analytical run used to report data. A run number of 2 is associated with the next oldest analytical run, and so on.

6. A Form Suffix for each form must appear in the two-character space provided after the form number in the bottom section of the form. The Form Suffix is used to sequentially distinguish between different forms of the same type (Form Number).

7. All the values substituted in the formulas given in the forms instructions must be exactly those values reported on the form for which the formula applies.

8. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

8.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

9. All results must be transcribed to Forms II-XIX from the raw data to the specified number of decimal places that are described in Exhibit B. 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 on that form. If there are not enough figures in the raw data result to enter in the specified space for that result, then zeros must be used for decimal places to the specified number of reporting decimals for that result for a specific form. The following examples of floating decimal places are provided:

Raw Data Result	Specified Format	Correct Entry on Form
5.9	6.3	5.900
5.99653	6.3	5.997
95.99653	6.3	95.997
995.99653	6.3	996.00
9995.996	6.3	9996.0
99995.9	6.3	99996.
999995.9	6.3	invalid

Note: The specified format of 6.3 means that a maximum of 6 spaces may be used to report the data, of which a maximum of 3 decimal places may be used.

10. To roundnumbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals 5 and there are no digits to the right of the 5 or all digits to the right of the 5 equal zero, then round up if the digit to be retained is odd, or round down if that digit is even. See also Rounding Rules entry in Glossary (Exhibit G).

11. Before evaluating a number for being in or out of control, round the number using EPA rounding rules to the significance required. For instance, the control limit for an ICV is plus or minus 10 percent of the true value. A percent recovery value of 110.4 would be considered in control while a value of 110.6 would be considered out of control. In addition, a value of 110.50 would be in control while a value of 110.51 would be out of control.

Part B - Cover Page [COVER PAGE - LCIN]: This form is used to list all field samples, duplicates, spikes, and performance evaluation samples (PES) analyzed within a SDG, and to provide certain analytical information. The form is signed by the Laboratory Manager to authorize and release all data and deliverables associated with the SDG.

1. Complete the header information according to the instructions in Part A and as follows.

2. The "SOW No." is the EPA-designated number that indicates the SOW version used for the data package being reported. For samples analyzed using this SOW, enter "ILC03.1" for "SOW No."

3. Under "EPA Sample No.," enter the EPA Sample No. of each field sample, (including spikes, duplicates, and the PES) to eight spaces, for the sample analyzed within the SDG. Spikes must contain an "S" suffix and duplicates a "D" suffix. These sample numbers must be listed on the form in ascending alphanumeric order using the EBCDIC convention. Thus, if MAB123 is the lowest (considering both alpha and numeric characters) EPA Sample No. within the SDG, it would be entered in the first EPA Sample No. field. Samples would be listed below it, in ascending sequence - MAB124, MAB125, MAC111, MA1111, MA1111D, MA1111S, etc.

4. All EPA Sample Nos. must be listed in ascending alphanumeric order, continuing to additional Cover Pages if necessary.

5. Under "Lab Sample ID.," a Lab Sample ID. (to 10 spaces) may be entered for each EPA Sample No. If a Lab Sample ID is entered, it must be entered identically (for each EPA Sample No.) on all associated forms.

6. Enter "YES" or "NO" in answer to each of the two questions concerning ICP-AES and ICP-MS corrections. Each question must be explicitly answered with a "YES" or a "NO." The third question must be answered with a "YES" or "NO" if the answer to the second question is "YES." It should be left blank if the answer to the second question is "NO."

7. Each Cover Page original must be signed and dated 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.

8. For "Name," enter the first and last name (to 25 spaces) of the person whose signature appears on the Cover Page.

9. For "Date," enter the date (formatted MM/DD/YY) on which the Cover Page is signed.

10. For "Title," enter the title (to 25 spaces) of the person whose signature appears on the Cover Page.

Part C - Comments Page [COMMENTS PAGE - LCIN]: This form is used to enter comments that are relevant to the analyses performed under the SDG as a whole.

1. Complete the header information according to the instructions in Part A, Part B (as applicable), and as follows.

2. Comments should describe in detail any problems encountered in processing the samples in the data package, any technical or administrative problems encountered, the corrective actions taken, all communication with SMO and/or the Regional representative(s), and the resolution of the problems.

3. Also included should be explanations detailing the rationale used for the elemental expressions which differ from those recommended by this SOW. These explanations should include the limitations of the elemental expressions, and their applicability to the samples analyzed.

Part D - Analysis Data Sheet [FORM I - LCIN]: This form is used to tabulate and report sample analysis results for target analytes (Exhibit C).

1. Complete the header information according to the instructions in Part A and as follows.

2. "Lab Sample ID," is the laboratory sample ID of the EPA sample number listed on the form if one was designated on the Cover Page.

3. "Date Received" is the date (formatted MM/DD/YY) of sample receipt at the laboratory, as recorded on the TR, i.e., the Validated Time of Sample Receipt (VTSR).

4. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte. For each blank, enter the concentration of each analyte (positive or negative) whose absolute value exceeds the IDL.

4.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

5. Under the column labeled "Concentration," enter for each analyte the value of the result.

5.1 Analytical results must be reported to two significant figures if the result value is less than 10; to three significant figures if the result value is greater than or equal to 10.

5.1.1 Note: This requirement for reporting results to two or three significant figures applies to Form I-LCIN only. Follow the specific instructions for reporting all other results on required forms as described in this exhibit.

6. Under the columns labeled "C," "Q," and "M," enter result qualifiers as identified below. If additional qualifiers are used, their explicit definitions must be included on the Cover Page in the Comments section.

6.1 C (Concentration) qualifier -- Enter "U" if the reported value was obtained from a reading that was less than the IDL. Use "B" if the value is less than the CRDL and greater than or equal to the IDL. Leave blank if greater than or equal to the CRDL.

6.2 Q qualifier -- Specified entries and their meanings are as follows:

- C - CRDL Standard not within control limits.
- E - The reported value is estimated because of the presence of an interference.
- M - Duplicate injection (exposure) precision not met.
- N - Spiked sample recovery not within control limits.
- S - The reported value was determined by the Method of Standard Additions (MSA).
- W - Analytical spike recovery not witin control limits
- Asterisk (*) - Duplicate analysis not within control limits.
- Plus (+) - Correlation coefficient for the MSA is less than 0.995.
- More than one of these qualifiers may be used in the Q column, except that entering "E," "S," or "+" is mutually exclusive. No combination of these qualifiers can appear in the same field for an analyte.

6.3 M (Method) qualifier -- Enter:

- "P" for ICP-AES when open beaker digestion is used
- "M" for ICP-MS when open beaker digestion is used
- "F" for graphite furnace AA when open beaker digestion is used
- "A" for flame AA when open beaker digestion is used
- "PM" for ICP-AES when microwave digestion is used
- "MM" for ICP-MS when microwave digestion is used
- "FM" for graphite furnace AA when microwave digestion is used
- "AM" for flame AA when microwave digestion is used
- "PB" for ICP-AES when block digestion is used
- "MB" for ICP-MS when block digestion is used
- "FB" for graphite furnace AA when block digestion is used
- "AB" for flame AA when block digestion is used
- "CV" for Cold Vapor AA
- "AV" for automated cold vapor AA
- "AS" for semi-automated Spectrophotometric
- "C" for manual spectrophotometric
- "CA" for midi-distillation Spectrophotometric
- "AC" for automated spectrophotometric
- "NR" if the analyte is not required to be analyzed
- " " if no results for the analyte appear on the form

6.4 A brief physical description of the sample before and after preparation must be reported:

- Color - red, blue, yellow, green, orange, violet, white, colorless, brown, grey, or black
- Clarity - clear, cloudy, or opaque
- Viscosity - nonviscous or viscous

6.5 Under "Comments" note any significant changes that occur during sample preparation i.e., emulsion formation, or sample-specific comments concerning the analyte results.

Part E - Initial and Continuing Calibration Verification [FORM II - HCIN]: This form is used to report analyte recoveries from calibration solutions.

1. Complete the header information according to the instructions in Part A and as follows.

2. "Run No.", refers to a number assigned to a collective sequential analytical run which starts, ends, and encompasses all of the QA/QC relevant to the analytical run from Exhibit E. A run number of 1 is assigned to the chronologically oldest analytical run used to report data. A run number of 2 is associated with the next oldest analytical run, and so on.

3. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

3.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

4. Three items are listed under "Initial Calibration."

4.1 Under "True," enter the value (in ug/L, to two decimal places) of the concentration of each analyte in the Initial Calibration Verification solution (ICV). If an analysis was not performed on the analyte, leave the field blank.

4.2 Under "Found," enter the value (in ug/L, to three decimal places), of the concentration of each analyte measured in the ICV.

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

$$\%R = \frac{\text{Found (ICV)}}{\text{True (ICV)}} \times 100$$

where True(ICV) is the true concentration of the analyte in the ICV and Found(ICV) is the found concentration of the analyte in the ICV.

5. "Continuing Calibration has three items:

5.1 Under "True," enter the value (in ug/L, to two decimal places) of the concentration of each analyte in the Continuing Calibration Verification solution (CCV). If an analysis is not performed on the analyte, leave the field blank.

5.2 Under "Found," enter the value (in ug/L, to three decimal places) of the concentration of each analyte measured in the CCV.

5.2.1 Note that the form contains two "Found" columns. The column to the left must contain values for the first CCV and the column to the right must contain values for the second CCV. The column to the right should be left blank if no second CCV was performed during the run.

5.2.2 If more than one Form II is required to report multiple CCVs, then the column to the left on the second form must contain values for the third CCV, the column to the right must contain values for the fourth CCV, and so on.

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

$$\%R = \frac{\text{Found (CCV)}}{\text{True (CCV)}} \times 100$$

where True(CCV) is the true concentration of each analyte, and Found(CCV) is the found concentration of the analyte measured in the CCVs.

5.3.1 Note that the form contains two "Continuing Calibration %R" columns. Entries to these columns must follow the sequence detailed above for entries to the "Continuing Calibration Found" columns.

6. Under "M," enter the method used, as explained in Part D.

7. Under "Comments" give additional relevant information.

7.1 Note that the order of reporting ICVs and CCVs for each analyte must follow the chronological order in which the standards were run, starting with the first Form II and moving from the left to the right continuing to additional Form IIs as appropriate. For instance, the first ICV for all analytes must be reported on the first Form II. In a run where three CCVs were analyzed, the first CCV must be reported in the left CCV column on the first Form II and the second CCV must be reported in the right column of the same form. The third CCV must be reported in the left CCV column of

the second Form II. On the second Form II, the ICV column and the right CCV column must be left empty in this example. In the previous example, if a second run for an analyte was needed, the ICV of that run must be reported on a third Form II and the CCVs follow in the same fashion as previously explained. Where more than one elemental expression is used for an analyte in the SDG, all ICV and CCV results using "EE" number 1 must be reported before proceeding to report the results from "EE" number 2, and so on.

Part F - CRDL Standards [FORM III - LCIN]: This form is used to report analyte recoveries from analyses of the CRDL Standards.

1. Complete the header information according to the instructions in Part A and as follows.

2. "Run No.," refers to a number assigned to a collective sequential analytical run which starts, ends, and encompasses all of the QA/QC relevant to the analytical run from Exhibit E. A run number of 1 is assigned to the chronologically oldest analytical run used to report data. A run number of 2 is associated with the next oldest analytical run, and so on.

3. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

3.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

4. The "Initial" and "Final" columns contain several parts.

4.1 Under "Initial True," enter the value (in ug/L, to two decimal places) of the concentration of each analyte in the CRDL Standard Source Solution that was analyzed for analytical samples associated with the SDG.

4.2 Under "Initial Found," enter the value (in ug/L, to three decimal places) of the concentration of each analyte measured in the CRDL Standard Solution analyzed at the beginning of each run.

4.3 Under "Final Found," enter the value (in ug/L, to three decimal places) of the concentration of each analyte measured in the CRDL Standard Solution analyzed at the end of each run.

4.4 Under "Initial %R" and "Final %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{\text{CRDL Standard Found}}{\text{CRDL Standard True}} \times 100$$

4.5 Note that for every initial solution reported there must be a final one. However, the opposite is not true. If a CRDL Standard was required to be analyzed in the middle of a run (to avoid exceeding the 8-h limit), it must be reported in the "Final Found" section of this form.

5. Under "M," enter the method used, as explained in Part D.

5.1 If more CRDL standards analyses were required or analyses were performed using more than one elemental expression per analyte, submit additional Form IIIs in the order explained in Part E as appropriate.

6. Under "Comments" give additional relevant information.

7. The order of reporting CRDL standards for each analyte must follow the chronological order in which the standards were run starting with the first Form III and continuing to the following Form IIIs as appropriate. When multiple elemental expressions are used for one analyte, all results using "EE" number 1 must be reported before proceeding to report the results from "EE" number 2, and so on.

Part G - Linear Range Determination Standard (LRS) [FORM IV - LCIN]: This form is used to report the upper limit of the linear range of all analysis systems and the analyte recoveries from analyses of the Linear Range Determination Standards (LRS).

1. Complete the header information according to the instructions in Part A and as follows.

2. "Run No.," refers to a number assigned to a collective sequential analytical run which starts, ends, and encompasses all of the QA/QC relevant to the analytical run from Exhibit E. A run number of 1 is assigned to the chronologically oldest analytical run used to report data. A run number of 2 is associated with the next oldest analytical run, and so on.

3. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used

and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

3.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

4. The "Initial" and "Final" columns contain several parts.

4.1 Under "Initial True," enter the highest calibration standard used or the value (in ug/L, to two decimal places) of the concentration of each analyte in the LRS that was analyzed for analytical samples associated with the SDG.

4.2 Under "Initial Found," enter the value (in ug/L, to three decimal places) of the concentration of each analyte measured in the LRS analyzed.

4.3 Under "Final Found," if a second LRS was used enter the value (in ug/L, to three decimal places) of the concentration of each analyte measured in the LRS analyzed at the end of each run.

4.4 Under "Initial %R" and "Final %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

$$\%R = \frac{\text{LRS Standard Found}}{\text{LRS Standard True}} \times 100$$

5. Under "M," enter the method of analysis (two characters maximum) for which the elemental expressions listed on the form were made as follows:

- "P" for ICP-AES
- "M" for ICP-MS
- "F" for graphite furnace AA
- "A" for flame AA

6. If more LRS standards analyses were required or analyses were performed using more than one elemental expression per analyte, submit additional Form IVs in chronological order as explained in Part E as appropriate.

6.1 The order of reporting LRS standards for each analyte must follow the chronological order in which the standards were run starting with the first Form IV and continuing to the following Form IVs as appropriate. When multiple elemental expressions are used for an analyte, all the results of "EE" number 1 must be reported before proceeding to "EE" number 2, and so on.

7. Under "Comments" give additional relevant information.

Part H - Blanks [FORM V - LCIN]: This form is used to report analyte concentrations found in the Initial Calibration Blank (ICB), in Continuing Calibration Blanks (CCB), and in the Preparation Blank (PB).

1. Complete the header information according to the instructions in Part A and as follows.

2. "Run No.," refers to a number assigned to a collective sequential analytical run which starts, ends, and encompasses all of the QA/QC relevant to the analytical run from Exhibit E. A run number of 1 is assigned to the chronologically oldest analytical run used to report data. A run number of 2 is associated with the next oldest analytical run, and so on.

3. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

3.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

4. Under "Initial Calib. Blank," enter the value (in ug/L, to three decimal places) of the concentration of each analyte measured in the ICB.

4.1 Under the "C" qualifier field, for any analyte, enter "U" if the absolute value of the analyte in the blank is less than the IDL. Use "B" if the value is less than the CRDL and greater than or equal to the IDL.

5. Under "Continuing Calibration Blank" several items are listed.

5.1 Under "Continuing Calibration Blank 1," enter the value (in ug/L, to three decimal places) of the measured concentration of each analyte detected in the first required CCB analyzed after the ICB.

5.2 Enter an appropriate qualifier, as explained for the "Initial Calib. Blank," (4.1 above) to the "C" qualifier column immediately following the "Continuing Calibration Blank 1" column.

5.3 If only two CCBs were analyzed, then leave the columns labeled "3" blank. If an additional CCB was analyzed, complete the columns labeled "3," in accordance with the instructions for the "Continuing Calibration Blank 1" column. If more than three CCBs were analyzed, then complete additional Form Vs as appropriate, following the chronological order in which they were run.

6. Under "Prep. Blank," enter the value (in ug/L, to three decimal places) of the measured concentration of each analyte in the Preparation Blank.

6.1 Enter any appropriate qualifier, as explained for the "Initial Calibration Blank," (4.1 above) to the "C" qualifier column immediately following the "Prep. Blank" column.

7. Under "M," enter the method used, as explained in Part D.

8. Under "Comments" give additional relevant information.

9. If more than one elemental expression is used for an analyte, submit additional Form Vs as appropriate.

10. The order of reporting ICBs and CCBs for each analyte must follow the chronological order in which the blanks were run starting with the first Form V and moving from left to right and continuing to the following Form Vs as explained in Part E. When multiple elemental expressions are used for the analysis of one analyte, all the results of "EE" number 1 must be reported before proceeding to "EE" number 2, and so on.

Part I - ICP-AES AND ICP-MS Interference Check Sample [FORM VI - LCIN]: This form is used to report Interference Check Sample (ICS) results for each ICP-AES or ICP-MS instrument used for each SDG.

1. Complete the header information according to the instructions in Part A and as follows.

2. For "Instrument ID Number," enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two Instruments within a laboratory may have the same Instrument ID Number.

3. The "Run No." found on many of the forms, refers to a number assigned to a collective sequential analytical run which starts, ends, and encompasses all of the QA/QC relevant to the analytical from Exhibit E. A run number of 1 is assigned to the chronologically oldest analytical run used to report data. A run number of 2 is associated with the next oldest analytical run, and so on.

4. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

4.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

5. True and Found Values

5.1 For all found values of solutions A and AB, enter the concentration (positive, negative, or zero) of each analyte not present in Solution A at each elemental expression used for analysis by the instrument.

5.2 Under "True Sol. A," enter the true concentration (in ug/L, to two decimal places) of each analyte analyzed by ICP that is present in Solution A. A concentration of zero "0" must be entered for the analytes analyzed by ICP-AES or ICP-MS that have no true value.

5.3 Under "True Sol. AB," enter the true concentration (in ug/L, to two decimal places) of each analyte present in Solution AB. A concentration of zero "0" must be entered for the analytes analyzed by ICP-AES or ICP-MS that have no true value.

5.4 Under "Initial Found Sol. A," enter the value (in ug/L, to three decimal places) of the measured concentration for each analyte analyzed by ICP-AES or ICP-MS that resulted from the initial analysis of Solution A as required in Exhibit E.

5.5 Under "Initial Found Sol. AB," enter the value (in ug/L, to three decimal places) of the measured concentration for each analyte analyzed by ICP-AES or ICP-MS that resulted from the initial analysis of Solution AB as required in Exhibit E.

5.6 For ICP-AES analysis under "Final Found Sol. A," enter the value (in ug/L, to three decimal places) of the measured concentration which resulted from the final analysis of Solution A as required in Exhibit E. ICP-MS analysis does not require a final analysis.

5.7 For ICP-AES analysis under "Final Found Sol. AB," enter the value (in ug/L, to three decimal places) of the measured concentration which resulted from the final analysis of Solution AB as required in Exhibit E. ICP-MS analysis does not require a final analysis.

5.8 Under "Initial Found %R" and "Final Found %R," enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

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

5.8.1 Leave the field empty if the True Solution A or AB is equal to zero.

5.9 Note that, except in the case of ICP-MS, for every initial solution reported there must be a final one. However, the opposite is not true. If an ICS was required to be analyzed in the middle of a run (to avoid exceeding the 8-h limit), it must be reported in the "Final Found" section of this form.

6. Under "M," enter the method of analysis (two characters maximum) for which the elemental expressions listed on the form were made as follows,

- "P" for ICP-AES
- "M" for ICP-MS
- "F" for graphite furnace AA
- "A" for flame AA

7. Under "Comments" give additional relevant information.

8. If more ICS analyses were required, submit additional Form VIs as appropriate. The order of reporting ICSs for each analyte must follow the chronological order in which they were run, starting with the first Form VI and continuing to the following Form VIs as appropriate. When multiple elemental expressions are used for one analyte, all the results of "EE" number

1 must be reported before proceeding to "EE" number 2, in the same manner as described in Part E.

Part J - Spike Sample Recovery [FORM VII - LCIN]: This form is used to report results for the matrix spike.

1. Complete the header information according to the instructions in Part A and as follows.

2. In the "EPA Sample No." box, enter the EPA Sample Number (8 places maximum) of the sample from which the spike results on this form were obtained. The number must be centered in the box.

3. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

3.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

4. Under "Sample Result (SR)," enter the value (in ug/L, to three decimal places) of the measured concentration for each analyte in the sample (reported in the EPA Sample No. box) on which the matrix spike was performed. Enter the IDL value if the analyte was not detected. Enter any appropriate qualifier, as explained in Part D, to the "C" qualifier column immediately following the "Sample Result (SR)" column.

5. Under "Spiked Sample Result (SSR)," enter the value (in ug/L, to three decimal places) of the measured concentration for each analyte in the matrix spike sample. Enter the IDL value if the analyte was not detected. Enter any appropriate qualifier, as explained in Part D, to the "C" qualifier column immediately following the "Spiked Sample Result (SSR)" column.

6. Under "Spike Added (SA)," enter the value (in ug/L, to three decimal places) of the concentration of each analyte added to the sample. If the "Spike Added" concentration is specified in the contract, the value added and reported must be that specific concentration in ug/L.

7. Under "%R," enter the value (to the nearest whole number) of the percent recovery for all spiked analytes computed according to the following equation:

$$\%R = \frac{SSR}{(SA + SR)} \times 100$$

7.1 %R must be reported, whether it is negative, positive or zero.

7.2 A value of zero must be used for SSR or SR if the analyte value is less than the IDL.

8. Under "Q," enter "N" if the Spike Recovery (%R) is out of the control limits (75-125).

9. Under "M," enter method used as explained in Part D.

10. Under "Comments" give additional relevant information.

11. If different samples were used for spike sample analysis of different analytes, additional Form VIIs must be submitted for each sample as appropriate.

12. Use additional Form VIIs for each sample on which a required spike sample analysis was performed. If multiple elemental expressions are used for an analyte, the results of "EE" number 1 must be reported proceeding to "EE" number 2, and so on.

Part K - Duplicates [FORM VIII - LCIN]: The duplicates form is used to report results of duplicate analyses.

1. Complete the header information according to the instructions in Part A and as follows.

2. In the "EPA Sample No." box, enter the EPA Sample Number (8 places maximum) of the sample from which the duplicate results on this form were obtained. The number must be centered in the box.

3. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

3.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a

number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

4. Under "Control Limit," enter the numerical value of the CRDL (in ug/L, to two decimal places) for the analyte if the sample or duplicate values were less than 5 times the CRDL. If both the sample and duplicate values were less than the CRDL or both were greater than or equal to 5 times the CRDL, leave the field empty.

5. Under "Sample (S)," enter the original value (in ug/L, to three decimal places) of the concentration of each analyte in the sample (reported in the EPA Sample No. box) on which a duplicate analysis was performed. Enter the IDL value if the analyte was not detected. Enter any appropriate qualifier, as explained in Part D, to the "C" qualifier column immediately following the "Sample (S)" column.

6. Under "Duplicate (D)," enter the value (in ug/L, to three decimal places) of each analyte in the Duplicate sample (reported in the EPA Sample No. box). Enter the IDL value if the analyte was not detected. Enter any appropriate qualifier, as explained in Part D, to the "C" qualifier column immediately following the "Duplicate (D)" column.

7. Under "RPD," enter the absolute value (to the nearest whole number) of the Relative Percent Difference for all analytes detected above the CRDL in either the sample or the duplicate, computed according to the following equation:

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

7.1 A value of zero must be substituted for S or D if the analyte concentration is less than the IDL in either one. If the analyte concentration is less than the IDL in both S and D, leave the RPD field blank.

8. 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 CRDL, then the RPD must be less than or equal to 20 percent to be in control. If either sample or duplicate values are less than 5 times the CRDL, then the absolute difference between the two values must be less than or equal

to the CRDL to be in control. If both values are below the CRDL, then no control limit is applicable.

9. Under "M," enter method used as explained in Part D.

10. Under "Comments" give additional relevant information.

11. Use additional Form VIIIs for each sample on which a required duplicate sample analysis was performed. If multiple elemental expressions are used for an analyte, the results of "EE" number 1 must be reported before proceeding to "EE" number 2, and so on.

Part L - Laboratory Control Sample [FORM IX - LCIN]: This form is used to report results for the Laboratory Control Sample.

1. Complete the header information according to the instructions in Part A and as follows. If an analyte was not required to be analyzed then leave the appropriate spaces blank.

2. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

2.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

3. Under "Limits," enter the lower limit (in ug/L, to two decimal places) in the left column, and the upper limit (in ug/L, to one decimal place) in the right column for each analyte in the LCS Source solutions.

4. Under "True," enter the value (in ug/L, to two decimal places) of the concentration of each analyte in the LCS Standard Source.

5. Under "Found," enter the measured concentration (in ug/L, to three decimal places) of each analyte found in the LCS solutions. Enter the IDL value if the analyte was not detected.

6. Enter any appropriate qualifier as explained in Part D to the "C" qualifier column immediately following the "Found" column.

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

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

7.1 If the analyte concentration is less than the IDL, a value of zero must be substituted for the LCS found.

8. Under "M," enter method used as explained in Part D.

9. Under "Comments" give additional relevant information.

10. Submit additional Form IXs as appropriate, if more than one LCS was required. If multiple elemental expressions are used for an analyte, the results of "EE" number 1 must be reported before proceeding to "EE" number 2, and so on.

Part M - Serial Dilution [FORM X - LCIN]: The Serial Dilution Form is used to report results of serial dilution analyses.

1. Complete the header information according to the instructions in Part A and as follows.

2. In the "EPA Sample No." box, enter the EPA Sample Number (8 places maximum) of the sample from which the duplicate results on this form were obtained.

3. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

3.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte

in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

4. Under "Initial Sample Result (I)," enter the value (in ug/L, to three decimal places) of the measured concentration for each analyte in the undiluted sample (reported in the EPA Sample No. box), which is within the linear range of the instrument, on which a Serial Dilution analysis was performed.

4.1 If the measured concentration of an analyte exceeds the linear range of the instrument, leave the field blank.

4.2 Enter the IDL value if the analyte was not detected.

4.3 Enter any appropriate qualifier, as explained in Part D, to the "C" qualifier column immediately following the "Initial Sample Result (I)" column.

4.4 Note that the Initial Sample concentration for an analyte does not have to equal the value for that analyte reported on Form I. The Initial Sample Concentration is the value of the analyte concentration (uncorrected for dilution) that is within the linear range of the instrument.

5. Under "Serial Dilution Result (S)," enter the measured concentration value (in ug/L, to three decimal places) of each analyte in the serially diluted sample (reported in the EPA Sample No. box) multiplied by 5.

5.1 If the measured concentration of an analyte exceeds the linear range of the instrument, leave the field blank.

5.2 Enter the IDL value multiplied by five if the analyte was not detected.

5.3 Enter any appropriate qualifier, as explained in Part D, to the "C" qualifier column immediately following the "Serial Dilution Result (S)" column.

5.4 Note that the Serial Dilution Result (S) is obtained by multiplying by five the instrument measured value (in ug/L) of the serially diluted sample. In addition, the "C" qualifier must be established based on the instrument measured value of the serially diluted result, before correcting it for the dilution. A value or "0" may be substituted for S if the analyte concentration is less than the IDL.

6. Under "% Difference," enter the value (to the nearest whole number) of the percent difference computed according to the following equation:

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

6.1 As mentioned in 5.4 above, a value of "0" must be substituted for S if the analyte concentration is less than the IDL. If I is less than the IDL, or I is blank, leave the "% Difference" field empty.

7. Under "Q," enter "E" if the % Difference value is greater than 10 percent and the sample concentration of the undiluted sample is within the linear range of the instrument and is greater than 20 times the CRDL.

8. Under "M," enter the method used as explained in Part D.

9. Under "Comments" give additional relevant information.

10. Use additional Form Xs for each sample on which a required serial dilution analysis was performed. If multiple elemental expressions are used for an analyte, the results of "EE" number 1 must be reported before proceeding to "EE" number 2, and so on.

Part N - Standard Addition Results [FORM XI - LCIN]: This form is used to report the results of samples analyzed using the Method of Standard Additions (MSA).

1. Complete the header information according to the instructions in Part A.

2. Under "EPA Sample No.," enter the EPA Sample Numbers (8 places maximum) of all analytical samples analyzed using MSA. This includes reruns by MSA (if the first MSA was out of control) as explained in Exhibit E.

2.1 A maximum of 32 standard addition results can be entered on this form. If additional MSAs were required, submit additional Form XIs.

2.2 Standard addition results must be listed in ascending alphanumeric order by EPA sample number using the EBCDIC convention. If more than one analyte for a sample required MSA, then the standard addition results for that sample must be listed in ascending alphabetic order by analyte. All analyte results must be reported before proceeding to the analyte results for the next sample number, continuing to the next Form XI if applicable. If multiple elemental expressions are used for an analyte, the results of "EE" number 1 must be reported before proceeding to "EE" number 2, and so on.

3. Under "An," enter the chemical symbol (3 spaces maximum) for each analyte for which MSA was required for each sample listed. The analytes must be listed in ascending alphabetic order.

4. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

4.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

5. "Additions" has several items.

5.1 Under "Zero Found" (y_1), enter the measured value in absorbance or intensity units (to 3 decimal places) for the analyte before any addition is performed.

5.2 Under "First Added" (x_2), enter the concentration in ug/L (to 3 decimal places) of the analyte added to the first addition of the sample analyzed by MSA.

5.3 Under "First Found" (y_2), enter the measured value in absorbance or intensity units (to 3 decimal places) for the sample solution spiked with the first addition.

5.4 Under "Second Added" (x_3), enter the concentration in ug/L (to 3 decimal places) of the analyte to the second addition of the sample analyzed by MSA.

5.5 Under "Second Found" (y_3), enter the measured value in absorbance or intensity units (to 3 decimal places) for the sample solution spiked with the second addition.

5.6 Under "Third Added" (x_4), enter the concentration in ug/L (to 3 decimal places) of the analyte (excluding sample contribution) after the third addition to the sample analyzed by MSA.

5.7 Under "Third Found" (y_4), enter the measured value in absorbance or intensity units (to 3 decimal places) for the analyte in the sample solution spiked with the third addition.

5.8 Note that "Zero Found," "First Found," "Second Found," and "Third Found" must have the same dilution factor.

6. Under "Final Conc.," enter the final analyte concentration (in ug/L, to 3 decimal places) in the sample as determined by MSA computed as follows, using the ordinary least-squares regression line (unweighted):

where,

$$m = \frac{4 \times \sum_{n=1}^{4} x_i \times y_i - \sum_{n=1}^{4} x_i \times \sum_{n=1}^{4} y_i}{4 \times \sum_{n=1}^{4} x_i^2 - \left(\sum_{n=1}^{4} x_i\right)^2}$$

$x_1 = 0$ $y_1 =$ "Zero Found"
$x_2 =$ "First Added $y_2 =$ "First Found"
$x_3 =$ "Second Added $y_3 =$ "Second Found"
$x_4 =$ "Third Added $y_4 =$ "Third Found"

and

$$b = \frac{1}{4} \times \sum_{n=1}^{4} y_i - \frac{m}{4} \times \sum_{n=1}^{4} x_i$$

then

$$\text{Final Concentration} = \frac{b}{m}$$

7. Under "r," enter the correlation coefficient (to 3 decimal places) computed as follows:

$$r = \frac{4 \times \sum_{n=1}^{4} x_i \times y_i - \sum_{n=1}^{4} x_i \times \sum_{n=1}^{4} y_i}{\left[4 \times \sum_{n=1}^{4} x_i^2\right]^{1/2} \times \left[4 \times \sum_{n=1}^{4} y_i^2 - \left(\sum_{n=1}^{4} y_i\right)^2\right]^{1/2}}$$

Note that the final concentration of an analyte does not have to equal the value for that analyte which is reported on Form I for that sample.

8. Under "Q," enter "+" if r is less than 0.995. If r is greater than or equal to 0.995, then leave the field empty.

9. Under "M," enter method used as explained in Part D.

Part O - Instrument Detection Limits (IDL) [FORM XII - LCIN]: This form documents the IDL for each instrument that the laboratory used to obtain data

for the SDG. Only the instrument and elemental expressions used to generate data for the SDG must be included.

1. Complete the header information according to the instructions in Part A and as follows.

2. Enter the "Instrument ID Number" for the instrument used to produce data for the SDG, and for which IDLs are being reported, as explained in Section H.

3. For "Method," enter the method of analysis as explained in Part D.

4. Enter the date (formatted MM/DD/YY) on which the IDL values were determined. This date must not exceed any of the analysis dates for that instrument in the SDG data package. Also, it must not precede them by more than 3 calendar months.

5. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

5.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

5.2 If multiple instruments are used for an analyte the IDLs for the lowest numeric instrument must be reported before proceeding to the next highest instrument. If multiple elemental expressions are used for an analyte, the results of "EE" number 1 must be reported before proceeding to "EE" number 2, and so on. After all "EE" numbers have been reported, then additional instruments may be reported if they were used.

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

7. Under "Background," enter the type of background correction used to obtain furnace AA data. Enter "BS" for Smith Hieftje, "BD" for Deuterium Arc, or "BZ" for Zeeman background correction. If ICP-MS was used, leave the field blank.

8. Under "CRDL," enter the Contract Required Detection Limit (in ug/L), as established in Exhibit C.

9. Under "IDL," enter the Instrument Detection Limit (in ug/L) as determined by the laboratory for each analyte analyzed by the instrument for which the ID Number is listed on this form. IDLs must be reported to two significant figures if the IDL value is less than 100 and to three significant figures for values above or equal to 100.

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

11. Use additional Form XIIs if more instruments, wavelengths, or elemental expressions are used.

Part P - ICP-AES and ICP-MS Elemental Expression Factors (A) [FORM XIII - LCIN]: This form documents for each ICP-AES and ICP-MS instrument the elemental expression factors, for each analyte, applied by the Contractor to obtain data for the SDG. Although the correction factors are determined annually (every 12 calendar months), a copy of the results of the annual elemental expression factors must be included with each SDG data package on Form XIII.

1. Complete the header information according to instructions in Part A and as follows.

2. Enter the "Instrument ID Number" for each ICP-AES, AA, and ICP-MS instrument used to produce data for the SDG, as explained in Section H. If more than one ICP instrument is used, submit additional Form XIIIs as appropriate.

3. For "Method," enter the method of analysis (two characters maximum) for which the elemental expressions listed on the form were made.

- "P" for ICP-AES
- "M" for ICP-MS
- "F" for graphite furnace AA
- "A" for flame AA

4. Report the date (formatted as MM/DD/YY) on which these correction factors were determined for use. This date must not exceed any of the analysis dates reported for that instrument in the SDG data package. Also, it must not precede them by more than 12 calendar months.

5. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES and AA, the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

5.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

6. Under "Wavelength or Mass," list the wavelength (in nanometers, to two decimal places) for ICP-AES instruments, or the mass-to-charge ratio (m/z, to nominal unit mass) for ICP-MS instruments for each analyte analyzed.

6.1 If more than one mass-to-charge ratio is used in the elemental expression to provide quantitation, then the mass-to-charge ratio entered should be the analyte's primary mass in the equation used for quantitation. For example, if the elemental expression for the first selenium (EE)is Se = (1.0000)(m/z 78)-(0.1869)(m/z 76) then the mass reported should be 78.

6.2 If more than one elemental expression is used, submit additional Form XIIIs as appropriate.

7. Under "EOM," enter the element symbol (for ICP-AES or AA) or the mass-to-charge ratio (m/z., to the nominal mass unit) (for ICP-MS), which will be used as a correction factor, for each correction that will be applied as part of the elemental expression. If a correction term is not needed, then leave the field blank.

8. Under "Factor," enter the correction factor (negative, positive, or zero, to 5 significant places, 7 spaces maximum) associated with the EE number to the left of the factor. If an "EOM" was not identified in the column to the left, leave the field blank.

9. Under "IS," enter the element symbol of the internal standard used for the analyte listed.

10. Under "ISEE" enter the "EE" number associated with the "IS." The "EE" number is the same as that found on Form XIV.

11. Under "Comments" give additional relevant information.

12. Use additional Form XIIIs as appropriate if correction factors for more than five analytes were applied. All correction factors for "EE" number 1 must be reported before proceeding to "EE" number 2 and so on.

Part Q - ICP-AES and ICP-MS Elemental Expression Factors (B) [FORM XIV - LCIN]: This form documents for each ICP-AES and ICP-MS instrument the elemental expression factors applied by the Contractor for internal standards

when they are used to obtain data for the SDG. This form is required for all ICP-MS analyses. Although the correction factors are determined annually (every 12 calendar months), a copy of the results of the annual elemental expression factors must be included with each SDG data package on Form XIV (B), if internal standards are used.

1. Complete the header information according to instructions in Part A and as follows.

2. Enter the "Instrument ID Number" for each ICP-AES, AA, and ICP-MS instrument used to produce data for the SDG, as explained in Section H. If more than one ICP instrument is used, submit additional Form XIVs as appropriate.

3. For "Method," enter the method of analysis (two characters maximum) for which the elemental expressions listed on the form were made as follows,

- "P" for ICP-AES
- "M" for ICP-MS
- "F" for graphite furnace AA
- "A" for flame AA

4. Under "Analyte" enter the name of the internal standard or analyte being described by the elemental expression.

5. Report the date (formatted as MM/DD/YY) on which these correction factors were determined for use. This date must not exceed any of the analysis dates reported for that instrument in the SDG data package. Also, it must not precede them by more than 12 calendar months.

6. "EE" is the elemental expression used to obtain data for each analyte. For ICP-AES the elemental expression identifies the wavelength used and interference correction terms (if any). For ICP-MS, the elemental expression identifies the primary quantitation mass and isobaric interference correction terms (if any). The actual elemental expressions are specified on Form XIII and are assigned an individual identifying number (the "EE" number) if more than one expression is specified for a given analyte.

6.1 Under "EE," enter the number of the elemental expression that was used to derive the results for each analyte reported on the form. The "EE" is a number assigned to each elemental expression when more than one elemental expression is used to obtain data for an analyte in the SDG. An "EE" number of 1 should be assigned to the most frequently used elemental expression for a given analyte in the SDG. An "EE" number of 2 should be assigned to the second most frequently used elemental expression, and so on. If only one elemental expression is used to obtain data for an analyte in the SDG, an "EE" number of 1 should be assigned to the elemental expression. The "EE" number must be consistently applied to the elemental expression it identifies for the entire SDG.

7. Under "Wavelength or Mass," list the wavelength (in nanometers, to two decimal places) for ICP-AES or AA instruments, or the primary mass-to-charge

ratio (m/z, to nominal unit mass) for ICP-MS instruments as explained in Part P for each analyte analyzed.

7.1 If more than one mass-to-charge ratio is used in the elemental expression to provide quantitation, then the mass-to-charge ratio entered should be the analyte's primary mass in the equation used for quantitation. For example, if the elemental expression for the first selenium EE is Se = (1.0000)(m/z 78)-(0.1869)(m/z 76) then the mass reported should be 78. If more than one elemental expression is used, submit additional Form XIVs as appropriate.

8. Under "EOM," enter the element symbol (for ICP-AES or AA;) or the mass-to-charge ratio (m/z, to the nominal mass unit) (for ICP-MS), which will be used as a correction factor, for each correction that will be applied as part of the elemental expression. If a correction term is not needed, then leave the field blank.

9. Under "Factor," enter the correction factor (negative, positive or zero, to five significant places, 7 spaces maximum) associated with the EOM to the left of the factor. If an "EOM" was not identified in the column to the left, leave the field blank.

10. Under "IS," enter the chemical symbol of the primary internal standard used. The chemical symbols must be listed in ascending atomic number.

11. Under "Comments" give additional relevant information.

12. Use additional Form XIVs as appropriate additional correction factors were applied. All correction factors for "EE" number 1 must be reported before proceeding to "EE" number 2 and so on.

13. Report all internal standard elemental expressions that were used to report analytical results under this contract.

Part R - ICP-MS Tuning and Response Factor Criteria [FORM XV - LCIN]: This form is used for reporting tuning, response factor, and mass calibration verification results for each ICP-MS run used to report data in the SDG.

1. Complete the header information according to the instructions in Part A and as follows.

2. Enter the "Instrument ID Number" for the ICP-MS instrument used to produce data on the form, as explained in Section H. A Form XV must be submitted for each ICP-MS analysis run in the SDG.

3. For "Run No.," enter the run number (two spaces maximum) from which the information on the form was taken. The run number is a sequential number for each run in the SDG that identifies the different analytical runs that are performed on the same instrument. The first run number for an instrument must be 1, the second must be 2, and so on.

4. For "Analysis Date," enter the date (formatted MM/DD/YY) of analysis of the initial tuning solution from which the information on the form was taken.

5. "Analysis Times" has two items.

5.1 For "Analysis Times, Initial," enter the time (in military format - HHMM) of analysis of the initial tuning solution from which the information on this form was taken.

5.2 For "Analysis Times, Final," enter the time (in military format - HHMM) of analysis of the final tuning solution from which the information on this form was taken.

6. The remainder of the form contains three tables.

6.1 Tuning

6.1.1 Under "% Relative Abundance, Initial," enter the percent relative abundance (to 2 decimal places) calculated from the intensities listed under "Response Factor," for each of the isotopes listed, as a result of analyzing the 100 ppb tuning solution (Table VII in Section C) at the beginning of each ICP-MS run. The isotopes are listed in the first column from the left in the Tuning Section of the Form.

6.1.2 Under "% Relative Abundance, Final," enter the percent relative abundance (to 2 decimal places) calculated from the intensities listed under "Response Factor," for each of the isotopes listed, as a result of analyzing the 100 ppb tuning solution (Table VII in Section C) at the end of each ICP-MS run. The isotopes are listed in the first column from the left in the Tuning Section of the Form.

6.2 Response Factor (counts per second)

6.2.1 Under "RF_{100} Response, Initial," enter the measured value of the response (in counts per second, to the nearest whole number) in the 100 ppb tuning solution (Table VII in Section C) analyzed at the beginning of each ICP-MS run, for each mass-to-charge ratio listed in the first column from the left in the Response Factor Section of the Form.

6.2.2 Under "RF_{100} Response, Final," enter the measured value of the response (in counts per second, to the nearest whole number) in the 100 ppb tuning solution (Table VII in Section C) analyzed at the end of each ICP-MS run for each mass-to-charge ratio listed in the first column from the left in the Response Factor Section of the Form.

6.3 Mass Calibration

6.3.1 Under "Observed Mass," enter the actual mass observed of each analyte's peak center (to two decimal places) in the 100 ppb tuning solution (Table VII in Section C) analyzed at the beginning of each ICP-MS run for each mass-to-charge ratio listed in the first column from the left in the Mass Calibration Section of the Form.

6.3.2 The values measured and reported in the Mass Calibration Section of the Form must be within the control limits listed in the second column from the left in each of the Sections.

7. Note that for every initial solution reported there must be a final one. However, the opposite is not true. If a tuning solution was required to be analyzed in the middle of a run (to avoid exceeding the 8-hour limit), it must be reported in the "Final" section of this form.

8. If more tuning solutions analyses were required, submit additional Form XVs reporting the runs for the lowest numeric instrument before proceeding to the next lowest instrument.

9. The order of reporting the tuning solution results must follow the chronological order in which the solutions were run starting with the first Form XV and continuing to the following Form XV, as appropriate.

Part S - ICP-MS Internal Standards Relative Intensity Summary (A) [FORM XVI - LCIN]: 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.

1. A run is defined as the continuous totality of analyses performed by an instrument throughout the sequence initiated by the first SOW-required calibration standard and terminated by, and including, the continuing calibration verification and blank following the last SOW-required analytical sample.

2. All field samples and all QC analyses (including calibration standards, ICVs, CCVs, ICBs, CCBs, MTS, CRIs, ICSs, LRSs, LCSs, PBs, duplicates, PE Samples, and spikes) associated with the SDG must be reported on Form XVI. The run must be continuous and inclusive of all analyses performed on the particular instrument during the run.

3. Submit one Form XVI 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 XVIs as appropriate. Each new run must be started on the first line of Form XVI.

4. An identical number of Form XVIIs with ICP-MS methods and Form XVIs must exist.

5. Complete the header information according to the instructions in Part A, and as follows:

6. For "Instrument ID Number," enter the instrument ID number (12 spaces maximum) which must be an identifier designated by the laboratory to uniquely identify each instrument used to produce data which are required to be reported in the SDG deliverable. If more than one ICP-MS instrument or run is used, submit additional Form XVIs 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.

7. For "Run No.,",enter the run number as explained in Part R.

8. For "Method," enter the method code (two characters maximum) as follows,

- "P" for ICP-AES
- "M" for ICP-MS
- "F" for graphite furnace AA
- "A" for flame AA

9. For "Start Time," enter the time (in military format - HHMM) that the analysis run was started.

10. For "End Time," enter the date (in military format - HHMM) that the analysis run was ended.

11. Under "EPA Sample No.," enter the EPA sample number of each analysis, including all QC operations applicable to the SDG (formatted according to Table B-1, Exhibit B). All EPA sample numbers must be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XVI 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, must be reported. Those analyses must be identified with the EPA Sample No. of "ZZZZZZ." Samples identified as "ZZZZZZ" need not have intensities reported for internal standards.

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

12.1 For any particular ICP-MS run, the EPA Sample No. and time sequence on Form XVI and XVII must be identical.

13. Under "Internal Standards %RI For:," enter the chemical symbol and elemental expression number of the internal standard in the three-space "Element" header field provided, to indicate the internal standard and elemental expression for which the relative intensity of the internal standards will be calculated in that column.

13.1 In the "Element" column, enter the internal standard relative intensity (to the nearest whole number) the internal standard in the EPA Sample No. for each sample analysis listed on the form (excluding "ZZZZZZ"). The internal standard relative intensity (%RI) is calculated using the following formula:

$$\% \text{ RI} = \frac{I_n}{I_o}$$

Where I_o is the intensity of the internal standard in the blank calibration standard, and

I_n is the intensity of internal standard in the EPA Sample No. in the same units.

13.2 Under the "Q" column to the right of each "Element" column, enter an "R" if the RI for a field sample, PES, duplicate, or spike is less than 0.30. If the percent relative intensity is greater than 0.30, leave the field blank.

13.3 Columns of internal standard RI must be entered left to right starting with the internal standards of the lower mass on the first Form XVI and proceeding to the following Form XVI as appropriate. All Form XVIs for the lowest numeric instrument must be reported in ascending order by the run number, before proceeding to the next Form XVI.

Part T - Analysis Run Log (A) [FORM XVII - LCIN]: This form is used to report the sample analysis run log for ICP-AES, AA, and ICP-MS only. In addition, the samples reported on this form must have been prepared in the same manner using no pre-preparation dilution or concentration steps. The results reported on Form I for the samples listed on this form for each analyte must be obtained by multiplying each analyte's concentration (in ug/L) from the instrument by the dilution factor listed on the form.

1. A run is defined as the continuous totality of analyses performed by an instrument throughout the sequence initiated by, and including, the initial and the final tuning solution, the first SOW-required calibration standard and terminated by, and including, the continuing calibration verification and blank following the last SOW-required analytical sample.

2. All field samples and all quality control analyses (including tuning solutions, ICP serial dilutions, calibration standards, ICVs, CCVs, ICBs, CCBs, MTS, CRIs, ICSs, LRSs, LCSs, PBs, duplicates, PE samples, and spikes) associated with the SDG must be reported on Form XVII. The run must be continuous and inclusive of all analyses performed on the particular instrument during the run.

3. Submit one Form XVII 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 XVIIs as appropriate.

4. Complete the header information according to the instructions in Part A, and as follows.

5. For "Instrument ID Number," enter the instrument ID number (12 spaces maximum) which must be an identifier designated by the laboratory to uniquely identify each instrument used to produce data which are required to be reported in the SDG deliverable. If more than one ICP-AES or ICP-MS instrument is used, submit additional Form XVIIs as appropriate.

6. For "Run No.," enter the run number as explained in Part R.

7. For "Method," enter the method code (two characters maximum) as follows,

- "P" for ICP-AES
- "M" for ICP-MS
- "F" for graphite furnace AA
- "A" for flame AA

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

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

10. Under "EPA Sample No.," enter the EPA sample number of each analysis, including all QC operations applicable to the SDG (formatted according to Table B-1, Exhibit B). All EPA sample numbers must be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XVII 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, must be reported. Those analyses must be identified with the EPA Sample No. of "ZZZZZZ."

11. Under "Prep. Batch Number," enter the preparation batch number for each sample and quality control sample preparation (including duplicates, spikes, LCSs, PBs, and PE samples) that are reported on the Form. The preparation batch number is used to link the sample analysis with the appropriate preparation batch. It consists of an ordered combination of the date of preparation (formatted MMDDYY), the hour of preparation (in military format - HH), and the method of preparation. The preparation batch number must be left-justified and may not have any blank spaces between its components. It may not have more than one leading blank. Single digit hours and months must be padded to the left with zeros. The following are examples of preparation batch numbers:

Prep. Batch Number	Preparation Hour	Date	Method
"11308915CV"	15	12/25/92	CV
"11038915P "	15	12/25/92	P
"01029008F "	8	01/01/93	F
"12908F "	invalid		

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

13. Under "D/F," note that for a particular sample a dilution factor (D/F) of "1" must be entered if the preparation product was analyzed without adding any further volume of dilutant or any other solutions to the sample or an aliquot of that sample taken for preparation.

13.1 For solutions such as ICVs, ICSs, PESs, and LCSs, a dilution factor must 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 must be that which would make the reported true values on the appropriate form for the solution equal those that were provided with the solution. For example, ICV-2(0887), an UEPA solution, has a true value of 104.0 ug/L at a 20 fold dilution. Not all required QC solution will be supplied by the USEPA. If the solution is prepared at a 40-fold dilution, a dilution factor of "2" must be entered on Form XVII and the uncorrected instrument reading is compared to a true value of 52 ug/L. In this example, Form II will have a true value of 104.0 regardless of the dilution used. The found value for the ICV must be corrected for the dilution listed on Form XVII using the following formula:

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

14. 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 on any of the forms in the SDG. Leave the column blank for each analyte if the analysis was not used to report the particular analyte.

Part U - Analysis Run Log (B) [FORM XVIII - LCIN]: This form is used to report the sample analysis run log for each instrument used for analysis in the SDG. This includes ICP-AES and ICP-MS analysis runs where conditions for reporting on Form XVII were not met. Form XVIII is analyte and method specific.

1. A run is defined as the continuous totality of analyses performed by an instrument throughout the sequence initiated by, and including, the initial and the final tuning solution, the first SOW-required calibration standard and terminated by, and including, the continuing calibration verification and blank following the last SOW-required analytical sample.

2. All field samples and all QC analyses (including tuning solutions, serial dilutions, calibration standards, ICVs, CCVs, ICBs, CCBs, MTS, CRIs, ICSs, LRS s, LCSs, PBs, duplicates, PE Samples, matrix spikes, analytical spikes, and each addition analyzed for MSA determination) associated with the SDG must be reported on Form XVIII if they were not reported on Form XVII. The run must be continuous and inclusive of all analyses performed on that instrument during the run.

3. Submit one Form XVIII 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 XVIIIs as appropriate.

4. Complete the header information according to the instructions in Part A, and as follows.

5. For "Instrument ID Number," enter the instrument ID number (12 spaces maximum) which must be an identifier designated by the laboratory 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 XVIIIs as appropriate.

6. For "Run No.," enter the run number as explained in Part Q.

7. For "Method," enter the method code (two characters maximum) as follows,

- "P" for ICP-AES
- "M" for ICP-MS
- "F" for graphite furnace AA
- "A" for flame AA

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

9. For "Analyte," enter the analyte's chemical symbol (three spaces maximum) for which the analysis run is being reported on the Form. Submit a Form XIX for each analyte analyzed which was not reported on Form XVIII.

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

11. Under "EPA Sample No.," enter the EPA sample number of each analysis, including all QC operations applicable to the SDG (formatted according to Table B-1, Exhibit B). All EPA sample numbers must be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XVIII 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, must be reported. Those analyses must be identified with the EPA Sample No. of "ZZZZZZ."

12. Under "Prep. Batch Number," enter the preparation batch number as explained in Part T.

13. Under "Int. Vol.," enter the initial volume (in milliliters, to the nearest whole number) of each sample or aliquot of the sample taken for preparation (distillation, digestion, etc.) for analysis by the method indicated in the header section of the Form. This field must have a value for each field sample listed.

14. Under "Fin. Vol.," enter the final volume (in milliliters, 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 must have a value for each field sample listed.

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

16. Under "D/F," enter the dilution factor (to two decimal places) by which the final product of preparation procedure (digestate or distillate) needed to be diluted for each analysis performed.

16.1 Note that for a particular sample, a dilution factor of "1" must be entered if the preparation product was analyzed without adding any further volume of dilutant or any other solution to the "Fin. Vol." of the sample or an aliquot of that "Fin. Vol." listed for that sample on this form.

16.2 For solutions such as ICVs, ICSs, and LCSs, a dilution factor must 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 must be that which would make the reported true values on the appropriate form for the solution equal those that were provided with the solution. For example, ICV-2(0887), an USEPA solution, has a true value of 104.0 ug/L at a 20 fold dilution. Not all required QC solutions will be supplied by the USEPA. If the solution is prepared at a 40-fold dilution, a dilution factor of "2" must be entered on Form XVIII and the uncorrected instrument reading is compared to a true value of 52 ug/L. In this example, Form II will have a true value of 104.0 regardless of the dilution used. The found value for the ICV must be corrected for the dilution listed on Form XVIII using the following formula:

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

17. Under "%R," enter the percent recovery (to two decimal places) for each analytical spike analyzed. Leave the field blank if the analysis reported is not an analytical spike. A %R of "-9999" must be entered for the analytical spike if either the sample or the analytical spike result is greater than the calibration range of the instrument.

18. Under "%RSD," enter the relative standard deviation of the replicate exposures or injections for each analysis reported on this form.

Part V - Standard Solutions Sources [FORM XIX - LCIN]: This form is used to report the source of each standard solution on an analyte-by-analyte basis used for initial and continuing calibration verifications, CRDL, LRS, ICS, and LCS standards used as a QC analysis in the SDG.

1. Complete the header information according to the instructions in Part A, and as follows.

2. Under "ICV Standard Source," enter the initial calibration source (10 spaces maximum) for each analyte for which ICV results were reported on Form II. Enter sufficient information in the available 12 spaces to unequivocally identify the manufacturer and the solution used.

3. Under "CCV Standard Source," enter the continuing calibration source (10 spaces maximum) for each analyte for which CCV results were reported on Form II, as described for the initial calibration source.

4. Under "CRI Standard Source," enter the CRDL standard source (10 spaces maximum) for each analyte for which CRDL standard results were reported on Form III, as described for the initial calibration source.

5. Under "LRS Standard Source," enter the linear range analysis source (10 spaces maximum) for each analyte for which LRS standard results were reported on Form IV, as described for the initial calibration source.

6. Under "ICS Standard Source," enter the ICP-AES and ICP-MS interference source (10 spaces maximum) for each analyte for which ICS standard results were reported on Form VI, as described for the initial calibration source.

7. Under "LCS Standard Source," enter the laboratory control sample source (10 spaces maximum) for each analyte for which LCS standard results were reported on Form IX, as described for the initial calibration source.

Part W - Sample Log-In Sheet [FORM DC - 1]: This form is used to document the receipt and inspection of samples and containers. 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 Arabic number and a copy of Form DC-1 must 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 each copy.

1. Sign and date the airbill (if present). Examine the shipping container and record in item 1 on Form DC-1 the presence/absence of custody seals and their condition (i.e., intact, broken).

2. Record the custody seal numbers in item 2.

3. Open the container, remove the enclosed sample documentation, and record the presence/absence of chain-of-custody record(s), SMO forms (i.e., traffic reports, packing lists), and airbills or airbill stickers in items 3-5 on Form DC-1. Specify if there is an airbill present or an airbill sticker in item 5 on Form DC-1. Record the airbill or sticker number in item 6.

4. Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record in items 7 and 8 on Form DC-1 the condition of the sample bottles (i.e., intact, broken, leaking) and presence or absence of sample tags.

5. Review the sample shipping documents and complete the header information described in Part A. Compare the information recorded on all the documents and samples and mark the appropriate answer in item 9 on Form DC-1.

6. If there are no problems observed during receipt, sign and date (include time) Form DC-1, the chain-of-custody record, and Traffic Report, and write the sample numbers on Form DC-1. Record the appropriate sample tags and assigned laboratory numbers if applicable. The log-in date should be recorded at the top of Form DC-1 and the date and time of cooler receipt at the

laboratory should be recorded in items 10 and 11. Cross out unused columns and spaces.

7. If there are problems observed during receipt, 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.

8. 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.

Part X - Document Inventory Sheet (FORM DC - 2): This form is used to record the inventory of the Complete SDG File (CSF) documents which are sent to the Region.

1. Organize all EPA-CSF documents as described in Exhibit B, Section II and Section III.

2. Assemble the documents in the order specified on Form DC-2 and Section II and III, and stamp each page with the consecutive number. (Do not number the DC-2 form).

3. Inventory the CSF by reviewing the document numbers and recording page numbers ranges in the column provided on the Form DC-2. If there are no documents for a specific document type, enter an "NA" in the blank space.

4. 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 the documents under No. 32, 33, 34, or 35. Category 35 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

SECTION IV - Data Reporting Forms

U.S. EPA - CLP

Low Concentration Inorganics

Cover Page

Lab Name: _____ Contract: _____

Lab Code: _____ Case 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 and ICP-MS background corrections applied?	(Yes/No)	___	___
If yes, were raw data generated before application of background corrections?	(Yes/No)	___	___

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed on the Comments Page. Release of the data contained in this hard copy data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

U.S. EPA - CLP

Low Concentration Inorganics

Comments Page/Case Narrative

Lab Name: _____ Contract: _____

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

SOW No.: ____

Comments:

U.S. EPA - CLP

Low Concentration Inorganics

Analysis Data Sheet

EPA Sample No.

Lab Name: _____ Contract: _____

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

Lab Sample ID: _____ Date Received: _____

Concentration Units: ug/L

CAS No.	Analyte	E	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					
7440-47-3	Calcium					
7440-70-2	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					
7440-02-7	Potassium					
7440-02-0	Nickel					
7782-49-2	Selenium					
7440-22-4	Silver					
7440-23-5	Sodium					
7440-28-0	Thallium					
7440-62-2	Vanadium					
7440-66-6	Zinc					
	Cyanide	X				
		X				

	Color	Clarity	Viscosity
Before:			
After:			

Comments:

U.S. EPA - CLP

Low Concentration Inorganics

Initial and Continuing Calibration Verification

Lab Name: _____ Contract: _____

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

Run No.: ___

Concentration Units: ug/L

Analyte	E E	Initial Calibration			Continuing Calibration					M
		True	Found	%R	True	Found 1	%R 1	Found 2	%R 2	
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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U.S. EPA - CLP

Low Concentration Inorganics

CRDL Standards

Lab Name: _____ Contract: _____

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

Run No.: ___

Concentration Units: ug/L

Analyte	E E	Initial			Final		M
		True	Found	%R	Found	%R	
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Mercury							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							
Cyanide							

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

Linear Range Determination Standards (LRS)

Lab Name: _____ Contract: _____

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

Run No.: ___

Concentration Units: ug/L

Analyte	E E	Initial			Final		M
		True	Found	%R	Found	%R	
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Mercury							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							
Cyanide							

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

Blanks

Lab Name: _____ Contract: _____

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

Run No.: ___

Concentration Units: ug/L

Analyte	E	Initial Calib. Blank	C	Continuing Calibration Blanks 1	C	2	C	3	C	Prep. 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												

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

ICP and ICP-MS Interference Check Sample

Lab Name: _____ Contract: _____

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

Instrument ID Number: _____ Run No.: ___

Concentration Units: ug/L

Analyte	E	True Sol. A	True Sol. AB	Initial Found Sol. A	Initial Found Sol. AB	%R	Final Found Sol. A	Final Found Sol. AB	%R	M
Aluminum										
Antimony										
Arsenic										
Barium										
Beryllium										
Cadmium										
Calcium										
Chromium										
Cobalt										
Copper										
Iron										
Lead										
Magnesium										
Manganese										
Mercury										
Nickel										
Potassium										
Selenium										
Silver										
Sodium										
Thallium										
Vanadium										
Zinc										

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

Spike Sample Recovery

EPA Sample No.

Lab Name: _____ Contract: _____

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

Concentration Units: ug/L

Analyte	E	Control Limit %R	Sample Result (SR)	C	Spiked Sample Result (SSR)	C	Spike Added (SA)	%R	Q	M
		75-125								
Aluminum		75-125								
Antimony		75-125								
Arsenic		75-125								
Barium		75-125								
Beryllium		75-125								
Cadmium		75-125								
Calcium		75-125								
Chromium		75-125								
Cobalt		75-125								
Copper		75-125								
Iron		75-125								
Lead		75-125								
Magnesium		75-125								
Manganese		75-125								
Mercury		75-125								
Nickel		75-125								
Potassium		75-125								
Selenium		75-125								
Silver		75-125								
Sodium		75-125								
Thallium		75-125								
Vanadium		75-125								
Zinc		75-125								
Cyanide		75-125								

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

Duplicates

EPA Sample No.

Lab Name: _____ Contract: _____

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

Concentration Units: ug/L

Analyte	E	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									

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

Laboratory Control Sample

Lab Name: _____ Contract: _____

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

Concentration Units: ug/L

Analyte	EE	Limits Lower	Limits Upper	True	Found	C	%R	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:

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U.S. EPA - CLP

Low Concentration Inorganics

Serial Dilution

EPA Sample No.

Lab Name: _____ Contract: _____

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

Concentration Units: ug/L

Analyte	E	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								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

Standard Addition Results

Lab Name: ____________________________ Contract: _________

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

Concentration Units: ug/L

EPA Sample No.	An	E E	Zero Found	ADDITIONS First Added	First Found	Second Added	Second Found	Third Added	Third Found	Final Conc.	r	Q	M

U.S. EPA - CLP

Low Concentration Inorganics

Instrument Detection Limits

Lab Name: _____ Contract: _____

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

Instrument ID Number: _____ Method: __ Date: _____

Concentration Units: ug/L

Analyte	Elemental Expression (EE)	Integ. Time (sec)	Back-ground	CRDL	IDL
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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U.S. EPA - CLP

Low Concentration Inorganics

Elemental Expression Factors (A)

Lab Name: _____ Contract: _____

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

Instrument ID Number: _____ Method: __ Date: _____

Analyte	EE	Wave-Length or Mass	EOM	Factor	EOM	Factor	EOM	Factor	EOM	Factor	EOM	Factor	IS	ISEE
Aluminum														
Antimony														
Arsenic														
Barium														
Beryllium														
Cadmium														
Calcium														
Chromium														
Cobalt														
Copper														
Iron														
Lead														
Magnesium														
Manganese														
Mercury														
Nickel														
Potassium														
Selenium														
Silver														
Sodium														
Thallium														
Vanadium														
Zinc														

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

Elemental Expression Factors (B)

Lab Name: _____ Contract: _____

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

Instrument ID Number: _____ Method: __ Date: _____

Analyte	EE	Wave-Length or Mass	EOM	Factor	EOM	Factor	EOM	Factor	EOM	Factor	EOM	Factor	IS

Comments:

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U.S. EPA - CLP

Low Concentration Inorganics

ICP-MS Tuning and Response Factor Criteria

Lab Name: _____ Contract: _____

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

Instrument ID Number: _____ Run No.: __

Analysis Date: _____ Analysis Times: (Initial) _____ (Final) _____

Tuning

m/z	Ion Abundance Criteria	% Relative Abundance	
		Initial	Final
7Li/59Co	(0.20 - 1.00)		
59Co/59Co	(1.00)		
115In/59Co	(0.75 - 2.00)		
205Tl/59Co	(0.50 - 1.20)		

Response Factor (counts per second)

m/z	Response Factor Criteria	RF_{100}	Response
		Initial	Final
7Li	(> 2,000)		
59Co	(>20,000)		
115In	(>10,000)		
102Ru	(< 25)		
205Tl	(> 1,000)		

Mass Calibration

m/z	Acceptable Mass Range	Observed Mass
7Li	(6.92 - 7.12)	
59Co	(58.83 - 59.03)	
115In	(114.80 - 115.00)	
205Tl	(204.87 - 205.07)	

U.S. EPA - CLP

Low Concentration Inorganics

ICP-MS Internal Standards Relative Intensity Summary (A)

Lab Name: ______________________ Contract: ________

Lab Code: ______ Case No.: ______ SDG No.: ______

Instrument ID Number: ____________ Run No.: __ Method: __

Start Time: ________ End Time: ________

EPA Sample No.	Time	Internal Standards %RI For:									
		Element __	Q	Element __	Q	Element __	Q	Element __	Q	Element __	Q

U.S. EPA - CLP

Low Concentration Inorganics

Analysis Run Log (A)

Lab Name: ______________________ Contract: ________

Lab Code: ______ Case No.: ______ SDG No.: ______

Instrument ID Number: ____________ Run No.: __ Method: __

Start Date: ________ End Date: ________

EPA Sample No.	Prep. Batch Number	Time	D/F	Analytes																						
				Al	Sb	As	Ba	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Hg	Ni	K	Se	Ag	Na	Tl	V	Zn

U.S. EPA - CLP

Low Concentration Inorganics

Analysis Run Log (B)

Lab Name: ______________________ Contract: ________

Lab Code: ______ Case No.: ______ SDG No.: ______

Instrument ID Number: _____________ Run No.: __ Method: __

Start Date: ________ Analyte: ___ End Date: ________

Retention Time Window: ______ Lower Limit: ______ Upper Limit: _____

EPA Sample No.	Prep. Batch Number	Int. Vol.	Fin. Vol.	Time	D/F	%R	%RSD	Retention Time

U.S. EPA - CLP

Low Concentration Inorganics

Standard Solutions Sources

Lab Name: _____ Contract: _____

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

Analyte	ICV Standard Source	CCV Standard Source	CRI Standard Source	LRS Standard Source	ICS Standard Source	LCS Standard Source
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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EXHIBIT C - Tables

TABLE I. - Inorganic Target Analyte List

Analyte	CRDL[1,2] (ug/L)
Aluminum	100
Antimony	5
Arsenic	2
Barium	20
Beryllium	1
Cadmium	1
Calcium	500
Chromium	5
Cobalt	5
Copper	5
Iron	100
Lead	2
Magnesium	500
Manganese	10
Mercury	0.2
Nickel	20
Potassium	750
Selenium	3
Silver	10
Sodium	500
Thallium	2
Vanadium	10
Zinc	5
Cyanide	10

(1) Any analytical method specified in Exhibit D may be utilized, except for mercury and cyanide, provided the documented instrument detection limits (IDLs) meet the CRDL requirements. Higher detection limits may only be used in the following circumstance:

If the sample concentration exceeds five times the detection limit of the instrument or method in use, the value may be reported even though the IDL may not equal the CRDL. This is illustrated in the example below:

For lead:

Method in use = ICP
IDL = 40
Sample concentration = 220
CRDL = 2
The value of 220 may be reported even though the IDL is greater than CRDL. The IDL must be documented as described in Exhibit E.

(2) The CRDL is the IDL obtained in pure water that must be met using the procedure in Exhibit E. The detection limits for samples may be considerably higher depending on the sample matrix.

TABLE II. - Initial and Continuing Calibration Verification, CRDL, Standard Control Limits, and LCS Standard Control Limits for Inorganic Analyses

INITIAL AND CONTINUING CALIBRATION VERIFICATION LIMITS

Analytical Method	Inorganic Species	% of True Value Limit (EPA Set) Low	High
ICP and AA	Metals	90	110
ICP-MS	Metals	90	110
Cold Vapor AA	Mercury	80	120
Other	Cyanide	85	115

CRDL STANDARD CONTROL LIMITS

Analytical Method	Inorganic Species	% of True Value Limit (EPA Set) Low	High
ICP/OES and AA	Metals	50	150
ICP/MS	Metals	50	150
Cold Vapor AA	Mercury	50	150
Other	Cyanide	50	150

LCS STANDARD CONTROL LIMITS

The LCS Standard Control Limits are the same for all inorganic species. The limits are 80 - 120.

TABLE III. - Spiking Levels for Matrix Spike

Element	Water (ug/L)
Aluminum	*
Antimony	200[2]
Arsenic	100[3]
Barium	500
Beryllium	50
Cadmium	50
Calcium	*
Chromium	200
Cobalt	200
Copper	200
Iron	1000
Lead	100
Magnesium	*
Manganese	200
Nickel	200
Potassium	*
Selenium	50[4]
Silver	50
Sodium	*
Thallium	50
Vanadium	200
Zinc	500
Mercury	1
Cyanide	100

(1) The levels shown indicate concentrations added in the final digestate of the spiked sample.

(2) The spike must be made with a solution containing antimony in the +5 oxidation state.

(3) The spike must be made with a solution containing arsenic in the +5 oxidation state.

(4) The spike must be made with a solution containing selenium in the +6 oxidation state.

*No spike required.

TABLE IV. - Interference Check Sample Components and Concentrations for ICP and ICP/MS

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Aluminum	100.0	100.0
Calcium	300.0	300.0
Iron	250.0	250.0
Magnesium	100.0	100.0
Sodium	250.0	250.0
Phosphorous	100.0	100.0
Potassium	100.0	100.0
Sulfur	100.0	100.0
Carbon	200.0	200.0
Chlorine	2121.5	2121.5
Molybenum	2.0	2.0
Titanium	2.0	2.0
Arsenic	0.0	0.100
Cadium	0.0	.100
Chromium	0.0	.200
Cobalt	0.0	0.200
Copper	0.0	.200
Manganese	0.0	.200
Nickel	0.0	0.200
Selenium	0.0	0.100
Silver	0.0	.050
Vanadium	0.0	0.200
Zinc	0.0	0.100

Note: See Exhibit D, Part E, for additional information.

TABLE V. - Example of Analyte Concentration Equivalents (mg/L) Arising from Interferents at the 100 mg/L Level for ICP/AES

Analyte	Wavelength (nm)	Interferent									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Al	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Sb	206.833	0.47	--	2.9	--	0.08	--	--	--	.25	0.45
As	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Ba	455.403	--	--	--	--	--	--	--	--	--	--
Be	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Bo	249.773	0.04	--	--	--	0.32	--	--	--	--	--
Cd	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Ca	317.933	--	--	0.08	--	0.01	*	0.04	--	0.03	0.03
Cr	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Co	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Cu	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Fe	259.940	--	--	--	--	--	--	0.12	--	--	--
Pb	220.353	0.17	--	--	--	--	--	--	--	--	--
Mg	279.079	--	*	0.11	--	0.13	--	0.25	--	0.07	0.12
Mn	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Mo	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Ni	231.604	--	--	--	--	--	--	--	--	--	--
Se	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Si	288.158	--	--	0.07	--	--	--	--	--	--	0.01
Na	588.995	--	--	--	--	--	--	--	--	0.08	--
Tl	190.864	0.30	--	--	--	--	--	--	--	--	--
V	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zn	213.856	--	--	--	0.14	--	--	--	0.29	--	--

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* These reported interferences are probably due to contaminants and not true spectral interference lines. When evaluating interferents, ultrapure reagents should be used. Interferences that are found should be verified by examination of the wavelength tables.

TABLE VI. - Tuning Solution For ICP/MS

(The tuning solution must consist of the following elements at the stated concentrations.)

Element	Concentration (ug/L)
^{7}Li	100
Co	100
In	100
Tl	100

TABLE VII. - Tuning, Response Factor, and Mass Calibration Criteria for ICP/MS

Tuning Criteria

m/z	Ion Abundance Criteria
7Li/59Co	(0.20 - 1.00)
59Co/59Co	(1.00)
115In/59Co	(0.75 - 2.00)
205Tl/59Co	(0.50 - 1.20)

Response Factor Criteria

m/z	Response Factor Criteria
7Li	(>20 counts per ppb)
59Co	(>200 counts per ppb)
115In	(>100 counts per ppb)
102Ru	(<25 counts)
205Tl	(> 10 counts per ppb)

Mass Calibration Criteria

m/z	Exact Mass
7Li	(6.92 - 7.12)
59Co	(58.83 - 59.03)
115In	(114.80 - 115.00)
205Tl	(204.87 - 205.07)

TABLE VIII. - Memory Test Solution for ICP/MS

(The memory solution must consist of the following elements at the stated concentrations)

Element	Concentration (mg/L)
Al	500
Ca	500
Fe	500
Mg	500
Na	500
K	500
C	1000
Cl	3600
Mo	10
P	500
S	500
Ti	10
Sb	10
As	10
Ba	10
Be	10
Cd	10
Cr	10
Co	10
Cu	10
Pb	10
Mn	10
Ni	10
Se	10
Ag	10
T	10
V	10
Zn	10

Note: See Exhibit D, Part E, and Exhibit E for further references to the memory test solution.

TABLE IX. - Internal Standards That May Be Used in ICP/MS

Bismuth
Holmium
Indium
Rhodium
Scandium
^{6}Lithium
Terbium
Yttrium

TABLE X. - Recommended Elemental Expressions for Isobaric Interferences for ICP/MS

Element	Isobaric Correction	Expression Proportional to Elemental Concentration
Al	none	(1.0000) (^{27}M)
Sb	none	(1.0000) (^{121}M)
As	ArCl, Se	(1.0000) (^{75}M) - (3.1278) (^{77}M) + (1.0177) (^{78}M)
Ba	none	(1.0000) (^{135}M)
Be	none	(1.0000) (^{9}M)
Cd	MoO, Sn	(1.0000) (^{114}M) - (0.0149) (^{118}M) - (1.6285) (^{108}M)
Ca	none	(1.0000) (^{44}M)
Cr	none	(1.0000) (^{52}M)
Co	none	(1.0000) (^{59}M)
Cu	none	(1.0000) (^{65}M)
Fe	none	(1.0000) (^{57}M)
Pb	none	(1.0000) (^{208}M) + (1.0000) (^{207}M) + (1.0000) (^{206}M)
Mg	none	(1.0000) (^{25}M)
Mn	none	(1.0000) (^{55}M)
Ni	none	(1.0000) (^{60}M)
Se	Ar_2	(1.0000) (^{78}M) - (0.1869) (^{76}M)
Ag	none	(1.0000) (^{107}M)
Tl	none	(1.0000) (^{205}M)
V	ClO, Cr	(1.0000) (^{51}M) - (3.1081) (^{53}M) + (0.3524) (^{52}M)
Zn	none	(1.0000) (^{66}M)
^{6}Li	Li(natural)	(1.0000) (^{6}M) - (0.0813) (^{7}M)
Sc	none	(1.0000) (^{45}M)
Y	none	(1.0000) (^{89}M)
Rh	none	(1.0000) (^{103}M)
In	Sn	(1.0000) (^{115}M) - (0.0149) (^{118}M)
Tb	none	(1.0000) (^{159}M)
Ho	none	(1.0000) (^{165}M)
Bi	none	(1.0000) (^{209}M)

M = the total ion count rate at the specified mass.

TABLE XI. - Contributions of Concomitant Elements to Nearby Analytes for ICP/MS When Resolution and Measurement Schemes Vary

Concentrations listed are the approximate level (mg/L) measured when the interferant is present at 100 mg/L.

		Peak Width at 10% of the Peak Height			
		1.0 amu		0.8 amu	
	Interferent	Integration Width		Integration Width	
Analyte	Element	0.9 amu	0.3 amu	0.9 amu	0.3 amu
^{121}Sb	^{120}Sn	820	5	10	1
$^{121}Sb^-$	^{122}Te	77	none	1	none
^{75}As	^{74}Se, ^{76}Se	910	4	3	none
^{9}Be	^{10}B	1,200	12	9	1
^{112}Cd	^{113}In	1,700	8	10	none
^{114}Cd	^{115}In	>5,000	150	180	18
^{116}Cd	^{115}In	30	none	5	none
^{52}Cr	^{51}V	1.4	1.5	none	none
^{53}Cr	^{54}Fe	650	7	1	none
^{59}Co	^{58}Ni, ^{60}Ni	>1,500	6	2	none
^{63}Cu	^{62}Ni, ^{64}Ni	190	1	none	none
^{63}Cu	^{64}Zn	4,000	14	9	none
^{65}Cu	^{64}Ni	1	1	none	none
^{65}Cu	^{64}Zn, ^{66}Zn	>4,400	22	15	none
^{208}Pb	^{209}Bi	140	14	57	none
^{55}Mn	^{54}Fe, ^{56}Fe	900	8	4	none
^{58}Ni	^{59}Co	>3,000	96	75	7
^{60}Ni	^{59}Co	9	4	10	5
^{62}Ni	^{63}Cu	>8,500	690	4,500	16
^{107}Ag	^{106}Pd, ^{108}Pd	>2,400	22	80	4
^{107}Ag	^{106}Cd, ^{108}Cd	130	3	5	2
^{109}Ag	^{108}Pd, ^{110}Pd	1,800	12	36	3
^{109}Ag	^{108}Cd, ^{110}Cd	1,600	10	37	3
^{51}V	^{52}Cr	>2,100	45	410	1
^{64}Zn	^{65}Cu, ^{63}Cu	>7,800	57	410	2
^{66}Zn	^{65}Cu	2	none	3	2

TABLE XII. - Isobaric Molecular-Ion Interferences That Could Affect the Analytes

	Interferents						
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	SS, SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN			WC	
^{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^{++}

continued

TABLE XIII (cont'd)

	Interferents						
Analyte	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{61}Ni	ScO	CaOH	TiN	MgCl	SiS	TiC	Sn^{++}
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	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	ClCl, 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	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

Note: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

TABLE XIII. - Mass Choices for Elements That Must Be Monitored Either During the Analytical Run or in a Separate Scan for ICP/MS

Mass	Element of Interest
27	Aluminum
121, 123	Antimony
75	Arsenic
138, 137, 136, **135**, 134, 132, 130	Barium
9	Beryllium
114, 112, 111, 110, 113, 116, 106, 108	Cadmium
42, 43, **44**, 46, 48	Calcium
52,**53**,**50**,54	Chromium
59	Cobalt
63, **65**	Copper
56, **54**, **57**, 58	Iron
208, **207**, **206**, 204	Lead
24, **25**, **26**	Magnesium
55	Manganese
202, **200**, 199, 201	Mercury
58, **60**, 62, **61**, 64	Nickel
39	Potassium
80, **78**, **82**, **76**, **77**, 74	Selenium
107, **109**	Silver
23	Sodium
205, 203	Thallium
51, **50**	Vanadium
64, **66**, **68**, **67**, 70	Zinc
83	Krypton
72	Germanium
139	Lanthanum
140	Cerium
129	Xenon
118	Tin
105	Palladium
47, **49**	Titanium
125	Tellurium
69	Gallium
35, 37	Chlorine
98, 96, 92, **97**, 94	Molybdenum

Note: Although the only masses that must be monitored are underlined, it is strongly recommended that the other elements be monitored to indicate other potential molecular interferences that could affect the data quality.

Boldface and underlined masses indicate the masses that should have the most impact on data quality and the elemental equations used to collect the data. Underlined masses **must** be monitored.

EXHIBIT D: Analytical Methods

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SECTION I: Introduction

Any analytical method specified in Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limits (CRDL) (Exhibit C, Tables I and II). Analytical methods with higher detection limits may be used only if the sample concentration exceeds five times the documented detection limit of the instrument or method.

Samples requiring dissolved metals analysis, will be filtered through a 0.45 μm membrane filter and preserved in the field before the samples are shipped to the laboratory. Instrument calibration standards must be matrix matched (with respect to acid content) to the samples.

All samples must initially be run undiluted (i.e., original sample or final product of sample preparation procedure). When an analyte concentration exceeds the calibration or linear range, re-analysis for that analyte(s) is required after appropriate dilution. Dilutions are prepared using reagent water with the same acid content as the undiluted sample. The Contractor must use the lowest dilution factor necessary to bring each analyte within its valid analytical range (but not below the CRDL). If more than one dilution is made to cover all analytes, the reported value must be from the sample result with the smallest dilution that is in the linear range for the analyte. The Contractor must submit proof that dilution was required to attain valid results by submission of both diluted and undiluted sample measurements in the raw data.

Labware must be acid cleaned according to the U.S. Environmental Protection Agency (EPA) manual "Methods for Chemical Analysis of Water and Wastes" or an equivalent procedure. Samples must be opened and digested in a hood. Stock solutions for standards may be purchased or made up as specified in Section IV, part A of Exhibit D. All sample dilutions shall be made with acidified deionized water to maintain constant acid strength.

Before water sample preparation is initiated, the Contractor must check the pH of all water samples, and note the pH in the sample preparation log and Comments Page.

Unless otherwise instructed by the EPA Administrative Project Officer (APO), all samples must be mixed thoroughly prior to aliquoting for analysis or digestion.

Background corrections are required for all furnace atomic absorption (AA) measurements.

All inductively coupled plasma - atomic emission spectroscopy (ICP-AES), and inductively coupled plasma - mass spectrometry (ICP-MS) measurements shall require a minimum of two complete replicate integrations. Integrations for all samples and quality assurance measurements must be reported in the raw data in concentration units; intensities are not acceptable. The average of the integrations must be used for standardization, sample analysis, and for reporting as specified in Exhibit B.

SECTION II: Sample Preservation and Holding Times

1. Preservation of Water Samples

Measurement Parameter	Container (1,2)	Preservation (3)
Metals (4)	P,G	HNO_3 to pH <2
Cyanide (CN), total and amenable to chlorination	A	0.6 g ascorbic acid (5) NaOH to pH >12 Cool, maintain at 4°C (±2°C)

Footnotes:

(1) Polyethylene (P) or glass (G).

(2) Amber (A) polyethylene or glass.

(3) Sample preservation is performed by the sampler immediately upon sample collection.

(4) Samples are filtered immediately on-site by the sampler before adding preservative for dissolved metals.

(5) Only used in the presence of residual chlorine.

2. Holding Times for Water Samples

Following are the maximum sample holding times allowable under this contract. To be compliant with this contract, the Contractor must analyze samples within these times even if these times are less than the maximum data submission times allowed in this contract.

Analyte	No. of Days Following Sample Receipt by Contractor
Mercury	26
Metals (other than mercury)	180
Cyanide	12

The Contractor must verify that the samples have been preserved properly using wide range pH paper. If the results of such verification do not conform to the requirements stated in item 1 for preservation or in item 2 for holding times, the Contractor must contact the Sample Management Office (SMO) for instructions before proceeding any further.

SECTION III: Sample Preparation

Before collecting samples, a decision must be made by the data user as to the type of data desired, i.e., dissolved or total constituent analysis. This information will be included on the traffic report and the following preparation techniques shall be used for analysis under this contract.

All samples and standards (including Quality Assurance/Quality Control (QA/QC) standards) must be matrix matched before analysis. Matrix matching must be applied without affecting the original sample volume by more than 10 percent.

Part A - Sample Preparation for "Dissolved" Metals Analysis

For the determination of dissolved constituents the sample must be filtered through a 0.45 μm membrane filter and then preserved in the field. This will be performed by the sampling team and recorded on the traffic report form. Analysis performed on a sample so treated shall be reported as "dissolved" concentrations.

Part B - Sample Preparation using Open-Vessel Block or Hot Plate Digestion for "Total" Metals Analysis

1 Scope and Application

This method is an acid digestion procedure using a multi-position block digester or hot plate to prepare water samples for analysis of "total" metals by GFAA, flame AA, ICP, or ICP-MS.

Aluminum	Chromium	Potassium
Antimony	Cobalt	Selenium
Arsenic	Copper	Silver
Barium	Iron	Sodium
Beryllium	Lead	Thallium
Cadmium	Magnesium	Vanadium
Calcium	Manganese	Zinc
	Nickel	

2 Summary of Method

A HNO_3/HCl mixture is added to a representative 100 mL portion of a water sampleand heated for 2 h or until volume is reduced to between 25 and 50 mL. After cooling, the digestate is filtered and brought to a 100 mL final volume.

3 Apparatus and Materials

3.1 MBlock digester (i.e., LABCONCO, AI Scientific, Lachat Questron), or hotplate.

3.2 250 mL volumetric block digestion tubes, or 250 mL beakers equipped with ribbed watchglasses.

3.3 Thermometer that covers a range of 0-200 °C.

3.4 Analytical balance capable of weighing to the nearest mg.

3.5 Whatman No. 42 filter paper or equivalent.

3.6 Polyethylene bottles, 125 mL or 250 mL.

4 Reagents

4.1 Reagent Water: Water used for preparing samples and reagents must meet the specifications for ASTM Type II water (ASTM D1193).

4.2 Concentrated reagent grade nitric acid (HNO_3) (sp gr. 1.41).

4.3 Concentrated reagent grade Hydrochloric Acid (sp gr. 1.19).

4.4 Reagent grade Hydrogen Peroxide (H_2O_2) (30%)

5 Digestion Procedure

Shake and transfer 100 mL of well-mixed sample to a 250 mL block digester tube or a 250 mL beaker. Add 2.0 mL of (1+1) HNO_3 and 1.0 mL (1+1) HCl to the sample. If using beakers, cover with a ribbed watchglass. Reflux at 95 °C for 2 hours or until the volume is reduced to between 25 and 50 mL (see note 1), making certain that the sample does not boil. Cool the sample and filter to remove insoluble material. Adjust the sample volume to 100 mL with reagent water, mix and transfer to a 125 mL plastic bottle (see note 2). The sample is now ready for analysis. Concentrations so determined shall be reported as "total."

6 Notes:

1) Monitor temperature with a thermometer placed in a centrally located digestion vessel that contains 100 mL of the appropriate digestion matrix.

2) Alternatively, the sample may be diluted to volume, mixed, centrifuged or allowed to settle, then decanted into a plastic storage bottle.

Part C - Sample Preparation using Closed-Vessel Microwave Oven Digestion for "Total" Metals Analysis

1 Scope and Application

1.1 This method is an acid digestion procedure using microwave energy to prepare water samples for analysis by GFAA, ICP, and/or ICP-MS for the following metals:

Aluminum	Chromium	Potassium
Antimony	Cobalt	Selenium
Arsenic	Copper	Silver
Barium	Iron	Sodium
Beryllium	Lead	Thallium
Cadmium	Magnesium	Vanadium
Calcium	Manganese	Zinc
	Nickel	

2 Summary of Method

A representative 45 mL water sample is digested with 5 mL of concentrated nitric acid for 20 min using microwave heating with a laboratory microwave digestion oven. The sample is placed in a Teflon® PFA vessel (or Teflon® liquid vessel) with 5 mL of concentrated nitric acid. The vessel is capped and heated in the microwave digestion oven. After cooling, the digestate is filtered if necessary and analyzed.

3 Apparatus and Materials (Microwave)

3.1 Microwave Digestion Oven - Use only laboratory-grade microwave digestion ovens. It must be power programmable with a maximum power rating of at least 600 watts. It must have a rotating sample turntable to ensure uniform exposure to the microwave radiation.

3.2 Digestion Vessels - Use Teflon® or Teflon®-lined closed-system microwave digestion vessels capable of withstanding pressures of at least 100 psi. The vessels must be capable of controlled pressure relief at the vessels maximum pressure rating.

4 Microwave Calibration Procedure

Microwave ovens which are controlled through a % power setting must be calibrated in terms of the microwave energy absorbed by the sample vs. the % power setting. The calibration function is not necessarily linear so that a multi-point calibration must be performed over the range of interest. Also the calibration function can vary from oven-to-oven so that each oven must be calibrated. The calibration procedure is given below. The microwave digestion oven shall be calibrated every six months, and the calibration documented in the laboratory sample preparation log.

4.0.1 Warm-up and equilibrate the microwave oven by heating 1-2L water at 100% power for 5 minutes.

4.0.2 Measure the power absorbed at % power settings of 30, 40, 50, 60, 70, 80, 90, 95, and 100. Perform each measurement in triplicate. For each measurement, pour 1000 ± 2 g of water into a microwave transparent vessel, such as a teflon bottle. Measure and record the initial temperature of the water to 0.1 °C (must be 24 ± 2°C). Place vessel into the microwave, start

carousel, and turn on the exhaust fan. Set the time to 120 sec and the power to the desired power setting (% power), and irradiate vessel. Promptly remove the vessel, add a stir bar, place on magnetic stirrer, and thermally equilibrate the water. Measure and record to 0.1 °C the maximum temperature observed within 30 seconds of removing the vessel from the oven.

Safety Note! Do not irradiate with stir bar in vessel. This can cause electrical arcing.

4.0.3 The absorbed power is determined by the following relationship:

$$P = \frac{(K \cdot Cp \cdot M \cdot \Delta T)}{t}$$

P = the apparent power absorbed by the sample in watts (W)
K = 4.184; the conversion factor for thermochemical cal/sec to W
Cp = the heat capacity of water ($cal \cdot g^{-1} \cdot C^{-1}$)
M = mass of the water sample in grams (g)
ΔT = the final temperature minus the initial temperature (°C)
t = the time in seconds

Using 2 min and 1000 g of distilled water, the calibration equation simplifies to:

$$P = \Delta T \cdot 34.87$$

Plot the measured watts vs. % power setting for each data point. Determine a calibration line for the linear portion of the calibration plot. The line is used to determine the % power setting for the actual watts specified in the protocol. If non-linearity is observed over the range of interest, measure the power at additional power settings to improve the accuracy of the calibration curve.

5 Microwave Vessel Cleaning Procedure

Clean new microwave digestion vessel by rinsing 3 times with reagent water, immersing in a 1:1 HCl cleaning bath for a minimum of 3 hours, rinsing 3 times with reagent water, immersing in a 1:1 HNO_3 cleaning bath for a minimum of 3 hours, and rinsing 3 times with reagent water. Next, heat the vessels for 96 hours at 200°C to anneal them prior to first use. The vessels must be disassembled during annealing. The sealing surfaces (the top of the vessel or its rim) must not be used to support the vessel during annealing. Between digestions wash entire vessel in hot, soapy water (nonphosphate detergent), rinse with 1:1 nitric acid, and rinse 3 times with reagent water. If contamination is detected in the reagent blank, all vessels must be recleaned.

6 Digestion Procedure

6.1 Measure the tare weight of the assembled digestion vessel. Add 45 mL sample and 5.0 mL of concentrated HNO_3 to each digestion vessel. Cap

and weigh the loaded vessel to 0.01 g. Place the loaded vessels into the sample carousel and place the carousel into the microwave oven. For ovens controlled through % power settings, the carousel must contain 5 vessels. If fewer samples are being digested, use additional reagent blanks to make up the difference. For ovens controlled through temperature feedback, install the temperature probe on one of the vessels.

6.2 Program the microwave digestion oven such that the samples are brought to a temperature of 160±4°C in 10 minutes followed by a slow rise to 165-170°C during a second 10 minutes, for a total digestion time of 20 minutes. For microwave digestion ovens programmed in terms of % Power, the correct settings must be determined for each type of digestion vessel. For single wall digestion vessels by CEM Corporation (120 psi limit) this corresponds to 10 minutes at 545 Watts followed by 10 minutes at 344 Watts. For lined, double wall vessels digestion vessels by CEM Corporation (200 psi limit), this corresponds to 10 minutes at 473 Watts followed by 10 minutes at 237 Watts.

6.3 Following the 20 minute digestion, cool the samples 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 them cool until they are no longer hot to the touch. After cooling, weigh the sample vessels to the nearest 0.01 gram and calculate the % digestion loss.

$$\% \text{ digestion loss} = \frac{(I - F)}{(I - T)} \times 100$$

I = Initial weight (vessel + sample + acid)
F = Final weight after digestion (vessel + sample + acid)
T = Tare weight of vessel

6.3.1 For samples with digestion losses less than 1%, shake well to mix any condensate within the digestion vessel. Uncap vessel. Filter or centrifuge to remove any particles. The digestate is now ready for analysis. Results must be corrected for the initial dilution (45 mL diluted to 50 mL).

6.3.2 Samples with digestion losses between 1 and 10%, the final digestate volume must be determined so that the dilution factor can be calculated. Shake well to mix any condensate within the digestion vessel. Quantitatively transfer the digestate to a 50 mL volumetric flask and dilute to volume. Alternatively, the density of the digestate can be measured at the final volume calculated by:

$$FV = \frac{F - T}{D}$$

FV = Final volume
F = final weight after digestion (vessel + sample + acid)
T = vessel weight
D = density of digestate

The digestate is then filtered or centrifuged to remove particulates and is ready for analysis. Results must be corrected for dilution.

6.3.3 For samples with digestion losses greater than 10% The digestion loss indicates that the vessel vented during digestion, which can significantly effect the digestion efficiency.

Part D - Distillation Procedures for CN Analysis in Water

Note: Oxidizing agents such as chlorine decompose most of the cyanide. Test a drop of the sample with potassium iodide-starch test paper (KI-starch paper); a blue color indicates the need for treatment. If a blue color is seen, add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume. Distillation can then proceed.

1 Full-Size Distillation Apparatus (Distillation Option A)

1.1 Place 500 mL of sample, or an aliquot diluted to 500 mL, in the 1 liter boiling flask containing a few boiling chips. Add 50 mL of 1.25N sodium hydroxide (Section 5.2.7 of Part F) to the absorbing tube and dilute if necessary with reagent water to obtain an adequate depth of liquid in the absorber. Add 10 mL of 1.25 N NaOH plus 40 mL of water to the overflow trap (the trap is optional for added safety). Connect the trap, boiling flask, condenser, and absorber in the train and attach to a vacuum source. Note: For added safety, all distillations should be performed in a hood.

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.

1.3 Temporarily disconnect the overflow trap and slowly add 25 mL concentrated sulfuric acid to the sample flask through the air inlet tube. Rinse the tube with reagent water and allow the airflow to mix the flask contents for 3 minutes. Add 20 mL of magnesium chloride solution (510 g $MgCl_2 \cdot H_2O$ in 1 L reagent water) to the sample flask through the air inlet and rinse the inlet tube with a stream of water. Excessive foaming from samples containing surfactants may be quelled by the addition of another 20 mL of magnesium chloride solution or addition of Dow Corning 544 antifoam agent. Reconnect the overflow trap and readjust the vacuum.

1.4 Heat the solution to boiling, taking care to prevent the solution from backing up and overflowing into the trap. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. When the boiling flask is cool, close the vacuum source and disconnect the absorber tube.

1.5 Drain the solution from the absorber into a 250 mL volumetric flask. Rinse the absorber tube using ASTM Type I water and add the washings to the volumetric flask. Bring to volume using ASTM Type I water.

1.6 The samples are now ready for analysis. The samples must be analyzed for cyanide within 12 days of receipt of the original samples, as specified in Section II. If the initial sample volume was less than 500 mL, and had to be diluted to 500 mL prior to distillation, an appropriate dilution factor must be included in the calculations of the original sample concentration. The dilution factor must be reported on Form XVIII.

2 Midi-Distillation Apparatus (Distillation Option B)

2.1 Pipet 50.0 mL of sample, or an aliquot diluted to 50.0 mL into the 100 mL distillation flask. Add 2 or 3 boiling chips.

Add 30 mL of 0.25 N NaOH (Section IV, Part F, 5.2.8) to the gas absorbing tube. Add 50 mL of 0.25 N NaOH to the overflow trap (the overflow trap is optional for added safety). Connect the overflow trap, boiling flask, condenser, absorber and vacuum source, as pictured in Figure 2.

2.2 Turn on the vacuum and adjust the gang (Whitney) valves to give a flow of three bubbles of air per second from the impingers in each reaction vessel.

2.3 After five minutes of vacuum flow, disconnect the overflow trap and inject 5 mL of 1:1 (v/v) H_2SO_4 through the top air inlet tube of the distillation head into the reaction vessel. Allow to mix for 5 minutes. The acid volume must be sufficient to bring the sample/solution pH to below 2.0. Add 2 mL of magnesium chloride solution (Section III, Part D, 1.3) 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 another 2 mL of magnesium chloride solution or addition of Dow Corning 544 antifoam agent. Reconnect the overflow trap.

2.4 Turn on the heating block or heating mantles and set for 123-125 °C. Heat the solution to boiling, taking care to prevent solution backup by periodic adjustment of the vacuum.

2.5 After one and one half hours of refluxing, turn off the heat and continue the vacuum for an additional 15 minutes. The flasks should be cool after this time. After cooling, close off the vacuum at the gang valve and drain the solution from the absorber into a 50.0 mL

volumetric flask and bring to volume with 0.25 N NaOH washings of the absorber tube.

2.6 Seal the receiving solutions and store them at 4 °C until analyzed. The solutions must be analyzed for cyanide within 12 days of receipt of the original samples, as specified in Section II. If the initial sample volume was less than 50 mL, and had to be diluted to 50 mL prior to distillation, an appropriate dilution factor must be included in the calculation of the original sample concentration. The dilution factor must be reported on Form XVIII.

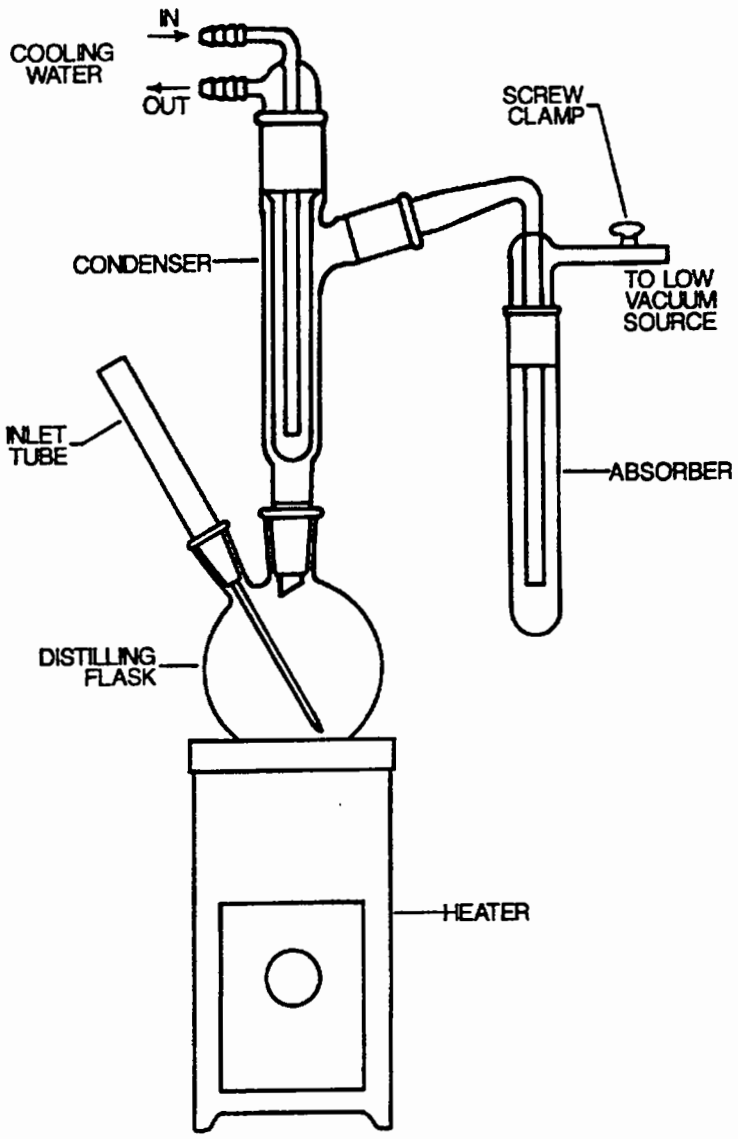

Figure 1. Cyanide distillation apparatus

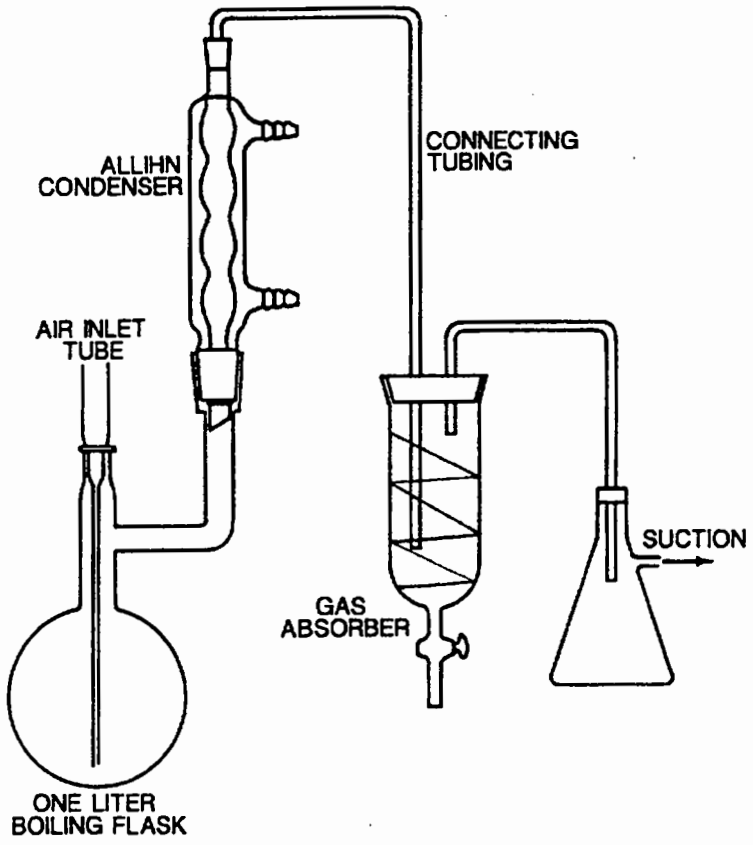

Figure 2. Cyanide distillation apparatus

SECTION IV: Sample Analysis

Part A - Reagents and Standards for Metals Analysis

1 Acid purity must be monitored. Analyte concentrations in the calibration and reagent blanks must be less than the CRDLs.

1.1 Acetic acid, conc. (sp gr 1.06)

1.2 Hydrochloric acid, conc. (sp gr 1.19)

1.3 Hydrochloric acid (1+1): Add 500 mL conc. HCl to 400 mL ASTM Type I water and dilute to 1 liter.

1.4 Nitric acid, conc. (sp gr 1.41)

1.5 Nitric acid (1+1): Add 500 mL conc. HNO_3 to 400 mL ASTM Type I water and dilute to 1 liter.

2 Reagent Water - Water meeting requirements for ASTM Type II Water (ASTM D1193). Prepare by passing distilled water or water purified by reverse osmosis through a mixed bed of cation and anion exchange resins. Use reagent water for the preparation of all reagents, calibration standards and as dilution water. The concentration of each analyte in reagent water must be less than the CRDL for the analyte.

3 Standard stock solutions may be purchased or prepared from ultrahigh-purity grade chemicals or metals (99.99 to 99.999% pure). All salts must be dried for 1 h at 105 °C, unless otherwise specified.

(CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.)

Typical stock solution preparation procedures are given below. Concentrations are calculated based upon the weight of pure element added, or by using the mole fraction and the weight of the metal salt added.

Metal

$$\text{Concentration (mg/L)} = \frac{\text{weight (mg)}}{\text{volume (L)}}$$

Metal salts

$$\text{Concentration (mg/L)} = \frac{\text{weight (mg)} \times \text{mole fraction}}{\text{volume (L)}}$$

NOTE: The recommended amounts of the starting materials specified for the following stock solutions are dependent upon the stoichiometry of the starting materials. Actual assay values of the starting materials should be used and the actual amounts corrected accordingly.

3.1 Aluminum solution, stock, 1 mL = 100 ug Al: Dissolve 1.3903 g $Al(NO_3)_3 \cdot 9H_2O$ in 10 mL reagent water with 10 mL. HNO_3. Dilute to 1000 mL with reagent water.

3.2 Antimony solution, stock, 1 mL = 100 ug Sb: Dissolve 0.1197 g Sb_2O_3 in 5 mL reagent water containing 0.1233 g $C_4O_6H_6$ (tartaric acid). Add 500 mL reagent water and 1 mL conc. HNO_3. Dilute to 1000 mL with reagent water.

3.3 Arsenic solution, stock, 1 mL = 100 ug As: Dissolve 0.1320 g of As_2O_3 in 100 mL of reagent water containing 0.45 g NH_4OH. Acidify the solution with 12 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

3.4 Barium solution, stock, 1 mL = 100 ug Ba: Dissolve 0.1437 g $BaCO_3$ in 10 mL reagent water with 10 mL conc. HNO_3. After dissolution is complete, warm the solution to degas. Dilute to 1000 mL with reagent water.

3.5 Beryllium solution, stock, 1 mL = 100 ug Be: Do not dry. Dissolve 4.5086 g $BeO(C_2H_3O_2)_6$ in reagent water, add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

3.6 Cadmium solution, stock, 1 mL = 100 ug 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.

3.7 Calcium solution, stock, 1 mL = 100 ug Ca: Suspend 0.2498 g $CaCO_3$ (dried at 180 °C for 1 h before weighing) in reagent water and dissolve cautiously with a minimum amount of (1+1) HNO_3. After dissolution is complete, warm the solution to degas. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

3.8 Chromium solution, stock, 1 mL = 100 ug Cr: Dissolve 0.2424 g of $(NH_4)_2Cr_2O_7$ in reagent water. Reduce the chromium with a few drops of hydrazine (NH_2NH_2), exhibited by the color change of the solution from orange to green. When reduction is complete, acidify with 10 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

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

3.10 Copper solution, stock, 1 mL = 100 ug Cu: Dissolve 0.1000 g Cu in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

3.11 Iron solution, stock, 1 mL = 100 ug Fe: Dissolve 0.1000 g Fe in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1000 mL with reagent water.

3.12 Lead solution, stock, 1 mL = 100 ug 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.

3.13 Magnesium solution, stock, 1 mL = 100 ug 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.

3.14 Manganese solution, stock, 1 mL = 100 ug Mn: Dissolve 0.3149 g of $Mn(C_2H_3O_2)_2$ in reagent water. Add 10.0 mL of conc. HNO_3 and dilute to 1000 mL with reagent water.

3.15 Mercury solution, stock, 1 mL = 100 ug Hg: Dissolve 0.1708 g mercury (II) nitrate $Hg(NO_3)_2 \cdot (H_2O)$ in 75 mL of reagent water. Add 10 mL of conc. HNO_3 and dilute to 1000 mL with reagent water.

3.16 Molybdenum solution, stock, 1 mL = 100 ug Mo: Dissolve 0.2043 g $(NH_4)_2MoO_4$ in reagent water. Dilute to 1000 mL with reagent water.

3.17 Nickel solution, stock, 1 mL = 100 ug Ni: Dissolve 0.1000 g of nickel metal in 10 mL hot conc. HNO_3, cool and dilute to 1,000 mL with reagent water.

3.18 Potassium solution, stock, 1mL = 100 ug K: Dissolve 0.1767 g K_2CO_3 in a minimum amount of (1+1)HNO_3. After dissolution is complete, warm the solution to degas. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with reagent water.

3.19 Selenium solution, stock, 1mL = 100 μg Se: Do not dry. Dissolve 0.1727 g H_2SeO_3 (actual assay 94.6%) in reagent water and dilute to 1,000 mL.

3.20 Silver solution, stock, 1 mL = 100 ug Ag: Dissolve 0.1575 g $AgNO_3$ in 100 mL of ASTM Type 1 water and 10 mL conc. HNO_3. Dilute to 1000 mL with reagent water.

3.21 Sodium solution, stock, 1mL = 100 μg Na: Dissolve 0.2305 g Na_2CO_3 in a minimum of (1+1) HNO_3. After dissolution is complete, warm the solution to degas. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL using reagent water.

3.22 Thallium solution, stock, 1 mL = 100 ug 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.

3.23 Titanium solution, stock, 1 mL = 100 ug Ti: Dissolve 0.4133 g $(NH_4)_2TiF_6$ in reagent water. Add 2 drops of conc. HF and dilute to 1000 mL with reagent water.

3.24 Vanadium solution, stock, 1 mL = 100 ug V: Dissolve 0.2296 g 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.

3.25 Zinc solution, stock, 1 mL = 100 ug 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.

PART B - Inductively Coupled Plasma-Atomic Emission Spectrometric Method

1 Scope and Application

Table I (Exhibit C) lists target analytes and their Contract Required Detection Limits (CRDLs). Method detection limits will be sample and matrix dependent. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account.

2 Summary of Method

The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. The aerosol resulting from sample nebulization is transported to a radio-frequency induced inductively coupled argon plasma (ICP) which produces characteristic atomic-line emission spectra. The high temperatures in the plasma atomize, ionize,and excite the analytes in the sample aerosol. As a result, both the atomic and ionic emmision line spectra characteristic of the analytes are produced. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by a detector or detectors capable of responding to the incoming photons. The received signals are processed using a computer system. Background correction measurements are required to compensate for variable background contributions and must be made adjacent to analyte lines during analysis. The position selected for the background intensity measurement may be on either or both sides of the analytical line, and must be determined by the complexity of the spectrum. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. The possibility of additional interferences named in Item 3, below, should also be recognized and appropriate corrections made.

Note: Background correction is not required when line broadening occurs. Background correction under these conditions would actually degrade the analytical result.

3 Interferences

Spectral, physical, and chemical interference effects may contribute to inaccuracies in the determination of trace elements.

3.1 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
- Background contribution from stray light from the line emission of high concentration elements.

The first of these effects can be compensated for by measuring the concentration of the interfering element and correcting for its contribution to analyte. The second effect may require selection of

an alternative wavelength (if available). The third and fourth effects can usually be compensated for by using a background correction point adjacent to the analyte line. Users of simultaneous multielement instrumentation are responsible for verifying the absence of spectral interferences from elements which may be present in a sample but for which there are no analytical channels.

Table VI (Exhibit C) lists potential interferences which may be observed at recommended wavelengths. These data are for information purposes only and do not contain absolute values which would be applicable to a specific laboratory. For the purposes of this contract, linear relations between concentration and intensity of the analytes and the interferents are assumed. The interference information, which was collected at the Ames Laboratory, is expressed as analyte concentration equivalents, i.e., false analyte concentrations arising from aspiration of 100 mg/L of the interfering element.

As an example of using the data in Table VI, assume that arsenic (at 193.696 nm) is to be determined in a sample containing 10 mg/L of aluminum. According to Table VI, Exhibit C, 100 mg/L of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/L. Therefore, 10 mg/L of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/L. The reader is cautioned that individual analytical systems will exhibit different levels of interference from those shown in Table VI, Exhibit C, and that the interference effects must be evaluated on an individual basis. Only those interferents listed were investigated, and the blank spaces in Table VI, Exhibit C, indicate that measurable interferences were not observed from the listed interferent concentrations with the instrumentation used.

3.2 Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Changes 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 lessen these interferences. If these types of interferences occur, they can be reduced by diluting the sample, using internal standards, or by employing the Method of Standard Additions.

3.3 High dissolved solids can result in salt buildup at the tip of the nebulizer. This affects aerosol flow and causes instrumental drift. Wetting the argon prior to nebulization, using a tip washer and sample dilution have been used to control this problem. It has also been reported that the use of mass flow controllers to control the argon flow rate improves instrument performance.

3.4 Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced using ICP-AES. If observed, they can be minimized by careful selection of operating conditions i.e. incident power, observation position, etc., by buffering the sample, matrix matching, or standard addition procedures. These

types of interferences can be highly dependent on matrix and the specific analyte involved.

4 Apparatus

4.1 An Inductively Coupled Plasma-Atomic Emission Spectrometer requires:

- Computer-controlled atomic emission spectrometer with background correction.
- Radio frequency generator.
- Argon gas supply, welding grade or better.
- Use of a mass-flow controller is recommnded.
- Use of a peristaltic pump is recommended.

4.2 Operational Requirements

4.2.1 System configuration

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 instrument's manufacturer. Sensitivity, instrument detection limits (IDLs), precision, linear dynamic range, and interference effects must be investigated and established for each analyte line on that particular instrument. All measurements must be within the instrument's 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 requirement set forth in the SOW. It is also the analyst's responsibility to maintain QC data confirming the instrument performance and analytical results.

The raw data must include hard copies or computer-readable storage media which can be readily examined by an EPA audit team. The raw data must demonstrate the presence or absence of all spectral interferences, including, but not limited to, those listed in Table VI of Exhibit C. The raw data must demonstrate defendable background correction points. This applies to simultaneous and sequential ICP instruments. Sequential ICP data must demonstrate the ability to select the correct peak from a spectrum in which nearby peaks from interferents are present.

5 Reagents and Standards (See Section IV, Part A)

5.1 Matrix matching with the samples is mandatory for all blanks, standards and QC samples. This avoids inaccurate concentrations from being reported due to possible standard curve deviations.

5.2 Prepare mixed calibration standard solutions for ICP by combining appropriate volumes of the stock solutions, (Section IV, Part A), in volumetric flasks. Add 2.0 mL of (1+1) HNO_3 and 1.0 mL of (1+1) HCl

and dilute to 100 mL with ASTM Type I water. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interferences or the presence of impurities. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable. Transfer the mixed standard solutions to a clean teflon or polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change on aging. Calibration standards must be initially verified using a QC sample, and monitored weekly for stability.

Although not specifically required, some typical calibration standard combinations are shown below:

- Mixed standard solution I -- Manganese, beryllium, cadmium, lead, silver, barium, copper, cobalt, nickel and zinc.
- Mixed standard solution II -- Arsenic, selenium, chromium, thallium, aluminum, calcium, magnesium, potassiom, sodium, and mercury.
- Mixed standard solution III -- Antimony, vanadium, and iron.

NOTE: If the addition of silver to the recommended acid combination results in precipitation, add 15 mL of ASTM Type I 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 mg/L. Silver under these conditions is stable in a tap water matrix for 30 days. Higher concentrations of silver require additional HCl.

5.3 Two types of blanks are required for ICP analysis: the calibration blank which is used in establishing the analytical curve, and the preparation blank which is used to evaluate possible contamination resulting from the acids used during sample processing.

5.3.1 The calibration blank is prepared by diluting 2.0 mL of (1+1) HNO_3 and 1.0 mL of (1+1) HCl to 100 mL with reagent water. Prepare sufficient quantity to be used to flush the system between standards and samples.

5.3.2 The preparation blank must contain all of the reagents and at the same volume as used in preparation of the samples.(see Exhibit E).

5.4 The Interference Check Solution (ICS, Exhibit E) is prepared to contain known concentrations of interfering elements. Its analysis verifies that interferences at the levels present in the ICS are corrected for by adequate inter-element and background corrections to within a specified QC limit. The ICS can be prepared by the analyst using certified stock solutions. Alternatively, it can be obtained from the EPA or another certified distributor.

6 Procedure

6.1 Set up instrument with proper operating parameters established in Section 4.2. The instrument must be allowed to become thermally

stable before beginning. This warmup usually requires at least 30 min of operation prior to calibration.

6.2 Initiate appropriate operating configuration of the computer.

6.3 Calibration and Sample Analysis

6.3.1 Profile and calibrate the instrument according to the manufacturer's recommended procedures, using matrix-matched mixed calibration standard solutions, such as those described in 5.1 and 5.2. Calibrate the instrument for the analytes of interest using the calibration blank and at least a single standard. Flush the system with the calibration blank between each standard. Use the average intensity of multiple integrations for both standardization and sample analysis. A minimum of two replicate integrations are required. The raw data must include the concentrations of the analytes in each integration as well as the average.

6.3.2 During the sample run the system should be flushed with the calibration blank solution between each analytical sample.

6.3.3 Dilute and reanalyze samples which exceed the established linear range for an analyte.

7 Calculations

If dilutions were performed, the appropriate dilution factor must be applied to sample concentrations. Appropriate concentration units must be specified on the required forms. The quantitative values shall be reported in units of micrograms per liter (ug/L) for aqueous samples. No other units are acceptable.

8 Quality Control

8.1 QA/QC must be performed as specified in Exhibit E.

8.2 All QA/QC data must be submitted with each data package as specified in Exhibit B.

8.3 The ICS is prepared to contain known concentrations of interfering elements and its analysis will provide an adequate test of any corrections performed. The ICS is used to verify that interferences at the levels present in the ICS are corrected by the data system within specified QC limits.

9 References

9.1 Annual Book of ASTM Standards, Part 31.

9.2 "Carcinogens - Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease

Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.

9.3 Garbarino, J.R. and Taylor, H.E., "An Inductively-Coupled Plasma Atomic Emission Spectrometric Method for Routine Water Quality Testing," Applied Spectroscopy 33, No. 3(1979).

9.4 Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019.

9.5 "Inductively Coupled Plasma-Atomic Emission Spectrometric Method of Trace Elements Analysis of Water and Waste", Method 200.7 modified by CLP Inorganic Data/Protocol Review Committee; original method by Theodore D. Martin, EMSL-Cincinnati.

9.6 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020.

9.7 "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

9.8 "Safety in Academic Chemistry Laboratories, American Chemical Society Publications, Committee on Chemical Safety, 3rd Edition, 1979.

9.9 Winefordner, J.D., "Trace Analysis: Spectroscopic Methods for Elements," Chemical Analysis, Vol. 46, pp. 41-42.

9.10 Winge, R.K., V.J. Peterson, and V.A. Fassel, "Inductively Coupled Plasma-Atomic Emission Spectroscopy Prominent Lines," EPA-600/4-79-017.

Part C - Graphite Furnace and Flame Atomic Absorption Spectroscopy

1 Scope and Application

1.1 Graphite furnace atomic absorption (GFAA) procedures are applicable to the determination of the target analytes in the water samples analyzed under this contract. GFAA is characterized by its low instrument detection limits (IDL), which are necessary to meet the CRDLs for many of the target analytes. IDLs, sensitivity, and optimum ranges for each analyte will vary with the specific instrument and operating conditions. Typically GFAA is used only for the analysis of As, Pb, Sb, Se, Tl, and in some cases Cd for which instrument detection limits are typically less than 2 ppb.

1.2 Flame AAS procedures are applicable to the determination of the target analytes in water samples analyzed under this contract. Flame AAS is characterized by moderate IDLs (mid to high ppb). IDLs, sensitivity, and optimum ranges for each analyte will vary with the specific instrument and operating conditions. Generally, flame AAS analyses are an alternative to ICP or ICP-MS analyses and are only used in special circumstances (eg, catastrophic failure).

2 Summary of Method

2.1 GFAAS - A discrete μL volume sample is placed in an electrically-heated graphite furnace tube, which forms the measurement cell of an atomic absorption spectrometer. Through a series of heating steps, the sample is dried, ashed, and the elements in the sample atomized directly in the furnace tube. A source lamp composed of the element being analyzed directs a light beam through the furnace tube, into a monochromator, and onto a detector, which measures the amount of light passing through the tube. Any ground-state atoms of the element being measured formed during the atomization step absorb some of the light from the source lamp. Consequently, the intensity of the light transmitted is inversely proportional to the concentration of the element in the sample.

2.2 Flame AAS - The principle for flame AAS is the same as that described for GFAAS with the exception that a flame burner replaces the graphite furnace tube and samples are introduced into the flame by aspiration.

3 Interferences

3.1 The composition of the sample matrix can have a major effect on the analysis. By modifying the sample matrix, either to remove interferences or to stabilize the analyte, interferences can be minimized. Examples are the addition of ammonium nitrate to remove alkali chlorides or the addition of ammonium phosphate to prevent cadmium volatilization. Both of these processes occur during the charring step of the temperature program.

Because gases and particulates are generated in the furnace during atomization, they absorb some of the analytes's characteristic radiation. This absorption if uncorrected would lead to an improper quantitation. Therefore, the use of background correction is required for GFAA.

Continuum background correction cannot correct for all types of background interference. When it is not possible to sufficiently compensate for the background interference, choose an alternative wavelength, chemically separate the analyte from the interferant, or use an alternate form of background correction, e.g., Zeeman background correction.

Interferences from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken to prevent loss of analyte.

3.2 Flame AAS - Chemical interferences are the most troublesome type of interference in flame AAS and result from the analyte of interest being bound to another element during atomization, and hence being unavailable for "atomic" absorption. This type of interference can be eliminated or minimized by using a hotter flame (eg, nitrous oxide-acetylene instead of air-acetylene) or by adding a matrix modifier to the sample.

Molecular absorption and light scattering caused by particles in the flame can cause high background absorption, which results in a positive bias in sample values. Background correction can correct for this type of interference. Three common background correction techniques that should provide adequate results are continuum-source, Zeeman, and Smith-Hiefte.

4 Apparatus

4.1 Atomic absorption spectrophotometer: Single or multi-channel, single or double beam instrument equipped with flame burner and/or graphite furnace cells, grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, background correction, and data system.

4.2 4.2.1 Operational Requirements and System Configurations: Due to differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instrument manufacturer's instructions. Sensitivity, IDLs, precision, linear dynamic range, and interference effects must be investigated and established for each analyte on each instrument.

It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements set forth in this SOW. It is also the analyst's

responsibility to maintain QC data confirming instrument performance and analytical results.

The raw data must include hard copies or computer-readable storage media which can be readily examined by an EPA audit team. The raw data must demonstrate defendable choices of furnace temperature programs and matrix modifiers.

5 Reagents and Standards (See Section IV, Part A)

5.1 Preparation of standards: Calibration standards are prepared by diluting stock metal solutions to the appropriate concentration. The acid content of the standards must match the acid content of the samples. Prepare at least four calibration standards for each analyte (blank, at the CRDL, and at two higher conentrations).

Digested samples - Prepare the standards by combining an appropriate volume of stock solution with 2 mL of (1+1) HNO_3 and 1 mL of (1+1) HCl in a 100 mL volumetric flask and dilute to volume with reagent water. If samples are prepared using the microwave digestion procedure, prepare the standards by combining an appropriate volume of stock solution with 10 mL of conc. HNO in a 100 mL volumetric flask and dilute to volume with reagent water.

Undigested samples - Samples being analyzed for dissolved metals do not require digestion. Prepare the standards such that the acid content matches that of the samples.

5.2 Two types of blanks are required for GFAA analysis; the calibration blank is used in establishing the analytical curve while the preparation blank is used to evaluate possible contamination resulting from the acids used in the sample processing.

The calibration blank is prepared as described in section 5.1 above. The preparation blank is prepared as specified in Exhibit E.

6 Procedure

6.1 Set up the instrument following the instrument manufacturer's instructions. The specific operating conditions (flame and furnace parameters, lamp parameters, wavelength, etc) must be determined by the operator to meet the required QA/QC requirements. The optimum conditions will vary with each element and instrument. The specific conditions must be documented in the instrument logbook and raw data. For general conditions and notes for analyses by flame AAS or GFAAS, consult the manufacturer's literature. General information is also available in the references listed below.

"Test Methods for Evaluating Solid Waste", SW-846, U.S. EPA, Series 7000 Methods.

"Standard Methods for the Examination of Water and Wastewater", APHA, AWWA, WEF

"Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S. EPA

"Techniques of Water-Resources Investigations of the United States Geological Survey, Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", Book 5, Chapter A1

6.2 Calibration and Sample Analysis

6.2.1 Calibrate the instrument according to the manufacturer's recommended procedures, using at least 4 calibration standards. One standard must be a blank and one must have a concentration equal to the CRDL. The concentration of the other standards are set by the operator to span the range of interest.

6.2.2 Following calibration, the samples and QC standards are analyzed as described in Exhibit E.

7 Calculations

The measured concentration of an analyte must be corrected for all dilutions performed as part the sample preparation and sample analysis. The required reporting units for concentration are µg/L

8 Quality Control

QA/QC must be performed as specified in Exhibit E. Corrective action for out-of-criteria QC results are also specified in Exhibit E.All QA/QC data must be submitted with each data package as specified in Exhibit B.

Part D - Inductively Coupled Plasma - Mass Spectrometry

1 Scope and Application

1.1 Metals for which this method is applicable are listed in Table I, Exhibit C. Instrument detection limits (IDLs), sensitivities, and linear ranges for these elements will vary with the matrices, instrumentation, and operating conditions. Use of this method is restricted to spectroscopists who are knowledgeable in the recognition and the correction of spectral, chemical, and physical interferences in ICP-MS. Experience requirement is 1 year on a commercially available ICP-MS.

2 Summary of Method

2.1 The method describes the multielemental determination of analytes by ICP-MS. The method measures ions produced by a radiofrequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and by means of a water-cooled interface, introduced into a mass spectrometer, capable of providing a resolution better than or equal to 1 amu peak width at 10% of the peak height. The water-cooled interface, consisting of tandem skimmers, is differentially pumped and leads into the high vacuum chamber of the mass spectrometer. The ions and ion clusters produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a detector. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices.

3 Interferences

3.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z) as the analyte of interest. A data system must be used to evaluate and correct for these interferences when they are present. This correction involves determining the signal for another isotope of the interfering element and subtracting out the appropriate signal from the isotope of interest. Data that is corrected must be noted in the report along with the exact calculations used. Table XI, Exhibit C, shows the analyte concentration measured when an interferent is present at 100 mg/L. Commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height. High ion currents at adjacent masses may also contribute to ion signals at the mass of interest. It should be noted that the information described in Table XI, Exhibit C, was experimentally derived and the interferences which are described occur from several different sources. One interference is the effect of resolution on adjacent peaks. In a quadrupole mass spectrometer, there is a larger effect at 1 amu less than the interferant than at 1 amu greater than the interferant's mass due to

the trapezoidal peak shape of the mass spectra. Another interference which is observed is the formation of a hydride ion. Hydride ion interferences only cause an interference at 1 amu greater than the interferant's mass. These interferences are not necessarily linear, and attempts must not be made to extrapolate the values to a particular data set. The table has been included for its informational content alone and does not contain quantitative values which would be applicable to any particular laboratory.

3.2 Isobaric molecular and doubly charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge. Table XII, Exhibit C, lists isobaric molecular-ion interferences which could affect the analytes. It should be noted that many of these interferences are extremely rare, but adverse effects on data quality could occur if the individual constituents occurred in the sample at sufficiently high concentrations. When the interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections to the data must be applied. Corrections for molecular-ion interferences may either be based upon the natural isotope ratios of the molecular ion or by measuring the interference which occurs when the interferant is present.

3.3 Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Nebulization and transport processes are those in which a matrix component causes a change in surface tension or viscosity in a manner different from the standards used in performing calibration. Internal standards have successfully been used to correct for these interferences. Physical interferences resulting from changes to ion transmission efficiencies are primarily suppressions, and lighter elements are suppressed more than the heavier elements. They also tend to be greater for matrix components with heavier atomic mass than for matrix components with lighter atomic mass. Changes in matrix composition therefore can also cause significant suppressions and enhancements. Dissolved solids can deposit on the tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and therefore changing the ion transmission efficiencies). Total dissolved solid levels below 0.2% (2000 ppm) have been recommended to minimize solid deposition. Internal standards must be affected to the same degree as the analyte to demonstrate that they compensate for these interferences. A minimum of three internal standards, listed in Table IX Exhibit C, bracketing the mass range, must be used. When the intensity level of an internal standard is less than 30% of the intensity of the first standard used during calibration, the sample must be reanalyzed for the affected analytes after performing a fivefold (1+4) dilution. Analyst Note: In the performance of this method, it has been observed the use of new or newly cleaned skimming cones result in large initial changes in the ion transmission efficiencies. These changes result in a large instrumental drift which can cause drift-sensitive quality assurance parameters to exceed control limits. It has been found that by conditioning the skimming cones by exposure to solutions (such as the

ICS) which are similar to the samples analyzed, the changes in ion transmission efficiencies will be mitigated. This conditioning appears to form an oxide layer on the skimming cones which insulates and therefore stabilizes the ion transmission efficiencies.

3.4 Memory interferences are effects which are dependant upon the relative concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer used, affect the extent to which memory interferences are present. To verify that memory effects do not have an adverse impact on data quality, the memory test must be performed on the tuned and calibrated instrument before any analyses are performed. A multielement memory test solution containing levels of analytes as specified in Table VIII, Exhibit C, is aspirated into the system for a normal sample exposure period. A rinse solution is then introduced, noting the time when the uptake tube is switched to the blank solution. After the normal routine rinse time has elapsed, begin a routine analysis of the calibration blank. Inspect the resulting data to see if any analytes are in excess of the CRDL. The memory test must be passed before any samples are analyzed under this contract. If a memory problem does exist (see Exhibit E) for a given analyte, increase the rinse time until the system passes the memory test. If the increased rinse time is not feasible from a sample throughput standpoint, a hardware change may be necessary. An apparent memory problem may in fact be blank contamination. This event may be determined by evaluating a second blank analysis and noting the values.

4 Apparatus and Materials

4.1 Inductively Coupled Plasma - Mass Spectrometer.

4.1.1 System capable of providing resolution, less than or equal to 1.0 amu at 10% peak height from 6-253 amu with a data system that allows corrections for isobaric interferences and the application of the internal standard technique. Use of a mass-flow controller for the nebulizer argon and a peristaltic pump for the sample solution are recommended.

4.1.2 Argon gas supply: Welding grade or better.

4.2 Operational Requirements and System Configuration

Because of the differences between various makes and models of instruments, no detailed operating instruction can be provided. Instead, the analyst should follow instrument manufacturer's instructions. The analyst must maintain QC data confirming instrument performance and analytical results.

It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements set forth in the SOW. It is also the analyst's

responsibility to maintain quality control data confirming instrument performance and analytical results.

The raw data must include hard copies and computer-readable storage media which can be readily examined by an EPA audit team. The raw data must demonstrate defendable choices of instrument operating conditions which minimize interferences such as oxides.

4.3 Precautions must be taken to protect a channel electron multiplier (if present) from high ion currents. Channel electron multipliers suffer from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. During this time period, response factors are constantly changing. This fluctuation invalidates the calibration curve, causes instability, and invalidates sample analyses. Samples run during such periods are required to be rerun at no additional cost to the government.

4.4 Sensitivity, IDL's, precision, linear dynamic range, and interference effects must be established for each analyte on a particular instrument. The analyst must maintain QC data confirming instrument performance and analytical results.

5 Reagents and Standards (See Section IV, Part A.)

5.1 Target analyte levels in acids used in the preparation of standards and for sample processing must be below the CRDLs for the purpose of a study. Redistilled acids or ultrapure acids are recommended for use with ICP-MS because of the high sensitivity of ICP-MS. Many more molecular-ion interferences are observed for analytes when hydrochloric and sulfuric acids are used, as demonstrated in Table XII, Exhibit C. Because HCl is added for stabilization, corrections for the chloride molecular ion interferences must be applied to all data generated.

5.2 Internal standards must be used to monitor and correct for changes that occur from differences between standards and samples. [This information must be clearly reported in the raw data.] The changes for which internal standards correct are primarily physical interferences. A minimum of three internal standards must be present in all standards and samples at identical levels by mixing the internal standard into the solution prior to nebulization. Additionally, if data is collected in a mode designed to extend the linear dynamic range, either by using a different detector or by changing voltages on the mass spectrometer components or detector, then at least one internal standard must be present which is measured in the dame mode that the samples were analyzed. Introduction may be accomplished by using a second channel of the peristaltic pump to add the internal standard to the uptake tube. If on-line addition is not used then internal standard spiking may be performed by adding a constant volume of internal standard concentrate to identical volumes of the standards and prepared samples. One typical example is to

measure out 10.0 mL of all standards and samples into individual containers, then to add 0.100 mL of a 10 mg/L solution of the internal standard to each of the containers. This procedure adds identical amounts of the internal standard to each solution for analysis. The concentrations of the analyte levels in the standards do not have to be corrected for the dilution which occurs, because dilution of the standards and samples is identical. Dilution of the sample by internal standards should be kept to the minimum possible while still maintaining the integrity of the sample analysis.

5.2.1 Bismuth internal standard solution, stock, 1mL = 100 ug Bi: Dissolve 0.1115 g Bi_2O_3 in a minimum amount of dilute HNO_3. Add 10 mL conc. HNO_3 and dilute to 1000 mL with ASTM Type I water.

5.2.2 Holmium internal standard solution, stock, 1 mL = 100 ug Ho: Dissolve 0.1757 g $Ho_2(CO_3)_2 \cdot 5H_2O$ in 10 mL ASTM Type I water and 10 mL HNO_3. After dissolution is complete, warm the solution to degas. Add 10 mL conc. HNO_3 and dilute to 1000 mL with ASTM Type I water.

5.2.3 Indium internal standard solution, stock, 1 mL = 100 ug In: Dissolve 0.1000 g indium metal in 10 mL conc. HNO_3. Dilute to 1000 mL with ASTM Type I water.

5.2.4 Lithium internal standard solution, stock, 1 mL = 100 ug ^6Li: Dissolve 0.6312 g of 95 atom % enriched ^6Li, Li_2CO_3 in 10 ml of ASTM Type I water and 10 mL HNO_3. After dissolution is complete, warm the solution to degas. Add 10 mL HNO_3 and dilute to 1 L with Type I water.

5.2.5 Rhodium internal standard solution, stock, 1 mL = 100 ug Rh: Dissolve 0.3593 g ammonium hexachlororhodate (III) $(NH_4)_3RhCl_6$ in 10 mL ASTM Type I water. Add 100 mL conc. HCl and dilute to 1000 mL with ASTM Type I water.

5.2.6 Scandium internal standard solution, stock, 1 mL = 100 ug Sc: Dissolve 0.15343 g Sc_2O_3 in 10 mL (1+1) hot HNO_3. Add 5 ml conc. HNO_3 and dilute to 1000 mL with ASTM Type I water.

5.2.7 Terbium internal standard solution, stock, 1 mL = 100 ug Tb: Dissolve 0.1828 g $Tb_2(CO_3)_3 \cdot 5H_2O$ in 10 mL (1+1) HNO_3. After dissolution is complete, warm the solution to degas. Add 5 ml conc. HNO_3 and dilute to 1000 mL with ASTM Type I water.

5.2.8 Yttrium internal standard solution, stock, 1 mL = 100 ug Y: Dissolve 0.2316 g $Y_2(CO_3)_3 \cdot 3H_2O$ in 10 mL (1+1) HNO_3. Add 5 ml conc. HNO_3 and dilute to 1000 mL with ASTM Type I water.

5.3 Mixed calibration standard solutions. Dilute the stock-standard solutions to levels in the linear range for the instrument in a solvent consisting of 1 percent (v/v) HNO_3 in ASTM Type I water along with the selected concentration of internal standards such that there

is an appropriate internal standard element for each of the analytes (see Table IX, Exhibit C). Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine possible spectral interferences or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to freshly acid-cleaned teflon or polyethylene bottles for storage. Fresh mixed standards must be prepared as needed with the realization that concentrations can change on aging. Calibration standards must be initially verified using a QC sample and monitored weekly for stability. Although not specifically required, some typical calibration standard combinations follow.

5.3.1 Mixed standard solution I -- manganese, beryllium, cadmium, lead, silver, barium, copper, cobalt, nickel, and zinc.

5.3.2 Mixed standard solution II - arsenic, selenium, chromium, thallium, aluminum, calcium, magnesium, potassium, sodium, and mercury.

5.3.3 Mixed standard solution III -- antimony, vanadium, iron.

5.3.4 Mixed standard solution IV -- bismuth, holmium, indium, lithium, scandium, yttrium, and terbium.

5.3.5 Mixed standard solution V -- rhodium.

Note: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of ASTM Type I water and warm the flask until the solution clears. Cool and dilute to 100 mL with ASTM Type I water. For this acid combination the silver concentration must be limited to 2 mg/L. Silver under these conditions is stable in a tap water matrix for 30 days.

5.4 Three types of blanks are typically required for the analysis. The calibration blank is used in establishing and monitoring the calibration curve The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. And the rinse blank is used to flush the system between all samples and standards.

5.4.1 The calibration blank generally consists of 1 percent HNO_3 plus 0.5 percent HCl (v/v) in ASTM Type I water along with the selected concentration of internal standards such that there is an appropriate internal standard element for each of the analytes (see Table IX, Exhibit C).

5.4.2 The preparation blank must contain all the reagents in the same volumes used for sample preparation. The preparation blank must be carried through the complete procedure and contain the same acid concentration in final solution as the sample solutions. (See Exhibit E).

5.4.3 The rinse blank consists of 2 percent HNO_3 (v/v) in ASTM Type I water. Prepare a sufficient quantity to flush the system between standards and samples.

5.5 The interference check solution(s) (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. For example, the chloride concentration provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^{+}$ on $^{51}V^{+}$ and $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$. Since the natural abundance of ^{35}Cl at 75.8 percent is 3.13 times the ^{37}Cl abundance of 24.2 percent, the ion corrections can be calculated with adjustments for isobaric contributions. Similarly, the iron in the ICS solutions is used to demonstrate adequate resolution of the spectrometer for manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement scheme to correct for various molecular-ion isobaric interferences. The ICS solutions are detailed in Table IV, Exhibit C and are used to verify that the interference levels are corrected by the data system to within QC limits.

5.5.1 Stock solutions for preparing ICS solutions A and AB may be obtained from EPA. Otherwise, refer to Table IV, Exhibit C. The ICS solutions A and AB must be prepared weekly.

5.5.2 Mixed ICS solution I may be prepared by adding 2.781 g $Al(NO_3)_3.9H_2O$, 1.499 g $CaCO_3$ (dried at 180°C for 1 h before weighing), 0.500 g Fe, 0.332 g MgO, 1.153 g Na_2CO_3, and 0.353 g K_2CO_3 to 25 mL of ASTM Type I water. Slowly add 100 mL of (1+1) HNO_3. After dissolution is complete, warm the solution to degas. Cool and dilute to 1000.0 mL with ASTM Type I water.

5.5.3 Mixed ICS solution II may be prepared by slowly adding 1.489 g 85% H_3PO_4, 1.275 g 96% H_2SO_4, 23.589g 37% HCl, and 2.133 g citric acid $C_6O_7H_8$ to 100 mL of ASTM Type I water. Dilute to 1000.0 mL with ASTM Type I water.

5.5.4 Mixed ICS solution III may be prepared by adding 2.500 mL of silver stock solution; 5.00 mL each of arsenic stock solution, cadmium stock solution,selenium stock solution, zinc stock solution; 10.00 mL each of chromium stock solution, cobalt stock solution, copper stock solution, manganese stock solution, nickel stock solution, and vanadium stock solution, and 2.5 mL of cadmium stock solution. Dilute to 100.00 mL with 2% HNO_3.

5.5.5 ICS A may be prepared by adding 50.00 mL of mixed ICS solution I, 2.0 mL each of titanium stock solution, and molybdenum stock solution, and 25.00 mL of mixed ICS solution II. Dilute to 100.00 mL with ASTM Type I water. ICS solution A must be prepared fresh weekly.

5.5.6 ICS AB may be prepared by adding 50.00 mL of mixed ICS solution I, 2.00 mL each of titanium stock solution and molybdenum stock solution, 25.00 mL of mixed ICS solution II, and 2.00 mL of mixed ICS solution III. Dilute to 100.00 mL with ASTM Type I water. ICS solution AB must be prepared fresh weekly.

6 Procedure

6.1 Initiate the appropriate operating configuration of the instrument's computer.

6.2 Set up the instrument with the proper operating parameters. The instrument must be allowed to become thermally stable before beginning. This warmup usually requires at least 30 minutes of operation prior to calibration. This must be verified by running the tuning solution (Table VI, Exhibit C) and obtaining at least four integrations with relative standard deviations of less than 10% for the analytes contained in the tuning solution.

6.3 Conduct mass calibration and resolution checks using the tuning solution (100 ppb of the elements in Table VI, Exhibit C). The recommended intensities and response factor criteria (see Table VII in Exhibit C) are helpful when setting up the instruments but are not required criteria. The mass calibration and resolution parameters must meet the criteria specified in Table VII, Exhibit C.If mass calibration exceeds these criteria, then the mass calibration must be adjusted to the correct values. The resolution must also be verified to be less than 1.0 amu full width at 10 percent peak height. The tuning solution must be analyzed at the beginning of each run prior to calibration and after mass calibration and resolution checks are performed and end of each 8 h shift, or end of the analytical run, whichever is more frequent, and pass the mass calibration and resolution criteria.

6.3.1 Prior to analyzing any samples under this contract, all of the samples must be screened for the presence of internal standards which might be indigenous to the samples. This screen is performed by calibrating the instrument for each of the internal standards using a single point calibration curve at the same level which will be used during the normal analytical run. After the screening calibration has been performed, then each sample to be analyzed during the normal analytical run will be introduced to the instrument without any internal standard added, and all of the masses associated with the internal standards will be scanned. The internal standard calibration standard must be analyzed at the end of the screening run. There is no additional quality assurance criteria associated with the screening run. The data from the screening run must be included in the raw data package and on Form XVII, no further reporting requirements are needed.

6.4 Calibration and sample analysis

6.4.1 Calibrate the instrument for the analytes of interest using the calibration blank and at least a single standard according to the manufacturer's recommended procedure for each detector configuration used in analysis. Flush the system with the rinse blank between each standard solution. Report each integration during the calibration and sample analysis and use the average of the multiple integrations for both standardization and sample analysis. A minimum of two replicate integrations are required for both calibration and sample analysis. The raw data must include the concentrations of elements in each integration as well as the average. Additionally, if different detector configurations are used, the raw data must indicate which detector configuration is being used.

NOTE: Some elements (such as Hg, W, and Mo) require extended flushing times which need to be determined for each instrumental system. Run Memory Test on solution in Table VIII, Exhibit C, to verify that memory problems will not affect the data quality.

6.5 As a minimum, masses which would affect data quality must be monitored to determine potential effects from matrix components on the analyte peaks. This information will be used to assess data quality and, as a minimum, must include the masses which are boldfaced and underlined, listed in Table XIII, Exhibit C, for each element. These masses must be monitored simultaneously in a separate scan, or at the same time that quantification occurs. Failure to provide a scan which includes all of the required masses will result in non-acceptance of the data package, and the samples associated with the incomplete data must be rerun at no cost to the government.

6.6 Flush the system with the rinse blank solution for at least 15 sec prior to the analysis of each sample. Aspirate each sample for at least 15 sec before collecting data. Note: The delay times for the rinse blank and the sample should be determined from the Memory Test.

6.7 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte.

7 Calculations

7.1 If dilutions were performed, the appropriate corrections must be applied to the sample values.

7.2 Appropriate concentration units must be specified on the required forms. The quantitative values shall be reported in units of micrograms per liter (ug/L) for aqueous samples. No other units are acceptable.

8 Quality Control

8.1 Quality control must be performed as specified in Exhibit E.

8.2 All QC data must be submitted with each data package as specified in Exhibit B.

8.3 To obtain analyte data of known quality, it is necessary to measure for more than the analytes of interest in order to know the required interference corrections. If the concentrations of interference sources (such a C, Cl, Mo, Zr, W) are below the levels that show an effect on the analyte level, uncorrected equations may be used provided all QA/QC criteria are met. It should be noted that monitoring the interference sources does not necessarily require monitoring the interference itself, but that a molecular species may be monitored to indicate the presence of the interference. The monitored masses must include those elements whose oxygen, hydroxyl, chlorine, nitrogen, carbon, and sulfur molecular ions which could impact the analytes of interest. When an interference source is present, the sample analytes impacted must be flagged to indicate the presence of an interference on the analyte of interest. These tests will enable the analyst to detect positive or negative interferences which affect the accuracy of the reported values.

8.4 The interference check solution(s) (ICS) is prepared to contain known concentrations of interfering elements and its analysis will provide an adequate test of any corrections performed. The ICS is used to verify that interferences at the levels present in the ICS are corrected by the data system within specified QC limits.

9 References

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Part E - Mercury Analysis in Water

1 Scope and Application

1.1 Organo-mercury compounds will not respond to the cold vapor atomic absorption (AA) technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent 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 percent recovery when used as the oxidant with these compounds. Therefore, a persulfate oxidation step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. Heating is required for methyl mercuric chloride whether indigenous to, or spiked into, a sample. For distilled water, heating is not necessary.

1.2 The range of the method may be changed through adjustment of instrument operating parameters, sample dilution or changes in sample size. Using a 100 mL sample, a detection limit of 0.2 ug Hg/L can be achieved.

2 Summary of Method

2.1 The cold vapor AA procedure is a physical method based on the absorption of radiation by atomic mercury vapor at 253.7 nm. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an AA spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

3 Interferences

3.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 While the possibility of absorption from certain organic substances actually being present in the sample does exist, EMSL-LV has not encountered such samples. This fact is mentioned only to caution the analyst of the possibility.

4 Apparatus

4.1 Atomic Absorption Spectrophotometer: An AA unit equipped with a heated cell suitable for presentation of mercury vapor.

It is the responsibility of the analyst to verify that the instrument configuration and operating conditions satisfy the analytical requirements set forth in this SOW. It is also the analyst's responsibility to maintain QC data confirming instrument performance and analytical result.

4.2 Hollow cathode, electrodeless discharge or low pressure mercury lamp.

4.3 Computer with software for controlling the spectrophotometer and autosampler, for calculating analyte concentration and for applying dilution and background correction factors.

4.4 Absorption cell: Standard spectrophotometer cells having quartz end windows.

4.5 Air pump: Any peristaltic pump capable of delivering 1 liter of air per min may be used.

4.6 Flowmeter: Capable of measuring an air flow of 1 liter per min.

4.7 Aeration tubing: A straight glass frit having a coarse porosity. Additional tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and then return.

4.8 Drying tube: 6 inch long x 3/4 inch diameter tube containing 20 g of magnesium perchlorate.

4.9 Autoanalyzer system including:

- Sampler with provisions for sample mixing.
- Proportioning pump.
- Mercury manifold.
- High temperature heating bath with two distillation coils.
- Vapor-liquid separator.

5 Reagents and Standards (See Part A)

5.1 Sulfuric acid, Conc: Reagent grade.

5.2 Nitric Acid, Conc: Reagent grade, of low mercury content.

5.3 Stannous sulfate or stannous chloride.

5.3.1 Stannous sulfate: Dissolve 11-g $SnSO_4$ in ASTM Type I water containing 7 mL conc. H_2SO_4 and dilute to 100 mL.

5.3.2 Stannous chloride: Dissolve 10-g $SnCl_2$ in ASTM Type I water containing 20 mL conc. HCl and dilute to 100 mL.

5.4 Sodium chloride-hydroxylamine sulfate solution: Dissolve 120-g NaCl and 120-g $(NH_2OH)_2 \cdot H_2SO_4$ in ASTM Type I water and dilute to 1L.

Note: A 10% hydroxylamine hydrochloride solution may be substituted for the hydroxylamine sulfate.

5.5 Potassium permanganate: Dissolve 50-g $KMnO_4$ in ASTM Type I water and dilute to 1 L.

5.6 Potassium persulfate: Dissolve 50-g $K_2S_2O_8$ in ASTM Type I water and dilute to 1 L.

5.7 Mercury solutions

5.7.1 Stock mercury solution: Dissolve 0.1354-g mercuric chloride, $HgCl_2$, in 70 mL ASTM Type I water, add 1 mL conc. HNO_3 and dilute to 100 mL. 1.00 mL = 1.00 mg Hg.

5.7.2 Standard mercury solutions: Prepare a series of standard mercury solutions containing 0 to 5 µg Hg/L by appropriate dilution of stock mercury solution with ASTM Type I water containing 10 mL conc. HNO_3/L. Prepare standards daily.

5.8 Air Scrubber Solution: Mix equal volumes of 0.1 N potassium permanganate and 10% sulfuric acid.

5.9 All blanks, standards, and QC samples must be matrix matched to the samples being analyzed. This avoids inaccurate concentrations from being reported due to possible standard curve deviations.

6 Procedure

6.1 Instrument Operation

6.1.1 Set wavelength to 253.7 nm.

6.1.2 Install absorption cell and align in light path to give maximum transmission.

6.1.3 Connect associated equipment to absorption cell with glass or vinyl plastic tubing. Turn on air and adjust flow rate to 2 L/min. Allow air to flow continuously.

6.2 Standardization

6.2.1 Transfer 100 mL of each of the 1.0, 2.0 and 5.0 µg/L Hg standard solutions and a blank of 100 mL water to 250 mL erlenmeyer flasks or BOD bottles.

6.2.2 Add 5 mL conc. H_2SO_4 and 2.5 mL conc. HNO_3 to each flask.

6.2.3 Add 15 mL $KMnO_4$ solution to each flask and let stand at least 15 min.

6.2.4 Add 8 mL $K_2S_2O_8$ solution to each flask and heat for 2 h in a water bath at 95 °C. Cool to room temperature.

6.2.5 For analysis, add enough NaCl-hydroxylamine sulfate solution to an individual flask to reduce excess $KMnO_4$, then add 5 mL $SnCl_2$ or $SnSO_4$ solution and immediately attach the flask to the aeration apparatus.

6.2.6 As Hg is volatilized and carried into the absorption cell, absorbance will increase to a maximum. When the recorder returns to approximately basline, remove the flask and replace with a flask of water. Flush the system between samples using water until system returns to baseline.

6.2.7 Construct a calibration curve by plotting peak height versus μg Hg. The correlation coefficient of the line should be at least 0.995.

6.3 Analysis of Samples

6.3.1 Transfer 100 mL of sample, or a portion diluted to 100 mL, containing not more than 5.0 μg Hg/L to a reaction flask or BOD bottle.

6.3.2 Treat the samples as in 6.2.2 through 6.2.5.

7 Calculations

7.1 Determine the peak height of the unknown from the chart and read the mercury value from the standard curve.

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

$$ug\ Hg/L = ug\ Hg\ in\ aliquot\ x\ \frac{1000}{volume\ of\ aliquot\ in\ mL}$$

7.3 Appropriate concentration units must be specified on the required forms. The quantitative values shall be reported in units of micrograms per liter (ug/L) for aqueous samples; NO other units are acceptable.

7.4 If dilutions were performed, the appropriate corrections must be applied to the sample values.

8 Quality Control

8.1 QA/QC must be performed as specified in Exhibit E.

8.2 All QA/QC data must be submitted with each data package as specified in Exhibit B.

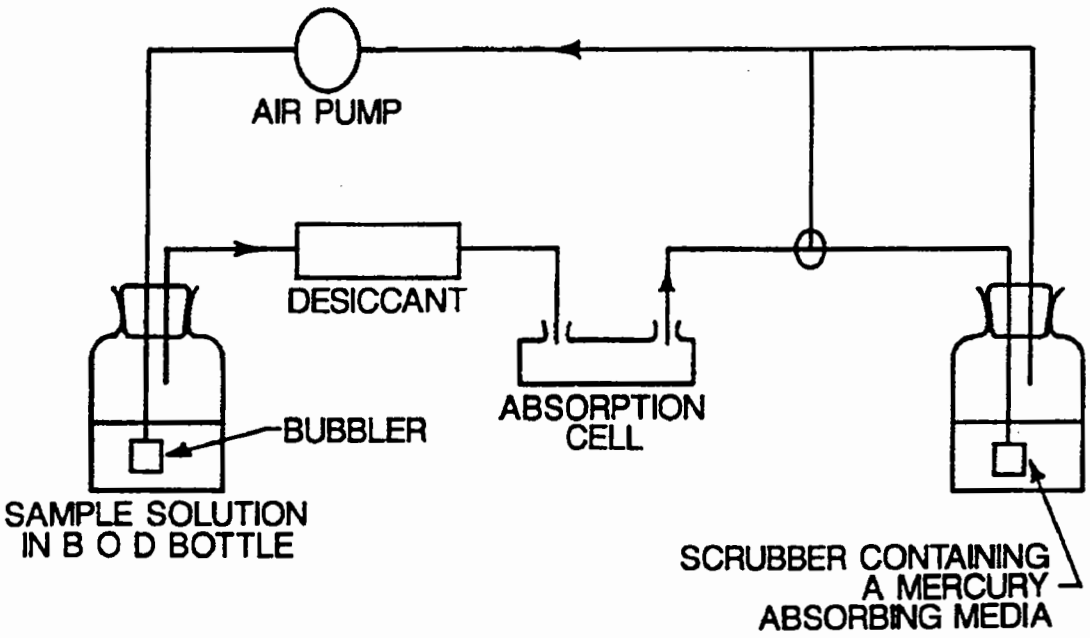

Figure 3. Apparatus for Cold Vapor Mercury Determination

Part F - Method for Total Cyanide Analysis in Water

1 Scope and Application

1.1 This method is applicable to the determination of cyanide in low concentration water samples.

1.2 The manual spectrophotometric procedure is used for concentrations below 1000 µg/L of cyanide and is sensitive to about 5 µg/L.

1.3 The working range of the semi-automated spectrophotometric method is 5 to 200 µg/L. Higher level samples must be diluted to fall within the working range.

2 Summary of Method

2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber solution containing sodium hydroxide. The cyanide ion in the scrubber solution is then determined spectrophotometrically by a manual measurement or semi-automated measurement.

2.2 In the spectrophotometric measurement the cyanide is converted to cyanogen chloride (CNCl) by reaction with chloramine-T at a pH less than 8. Low pH prevents hydrolysis of CNCl to cyanate (CNO). After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridine-barbituric acid reagent. The absorbance is read at 620 nm when using pyridine-pyrazolone or 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

3 Interferences

3.1 Interferences are eliminated or reduced by using the distillation procedure described in Section III, Part D

3.2 Sulfides in a sample will be distilled and collected as H_2S in the scrubber solution and will adversely affect the spectrophotometric procedure via direct competition with cyanide for the colorimetric reagents. Thiocyanates can also decompose during distillation to give sulfides. A test for sulfides in the scrubber solution must be performed before proceeding with the analysis. After distillation and dilution of the solution to volume, a drop of the solution is placed on lead acetate test paper. A dark color indicates the presence of sulfides. If darkening is seen, treat 1½ times the aliquot needed for analysis with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the scrubber solution contains sulfide. Repeat this operation until a drop of the treated 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 aliquot of sample to be used for analysis. Avoid a large excess of cadmium

carbonate and a long contact time in order to minimize cyanide losses by complexation or co-precipitation.

4 Apparatus

4.1 For the manual spectrophotometric method, use a spectrophotometer suitable for measurements at 578 nm or 620 nm with a 1.0 cm cell or larger.

4.2 For semi-automated spectrophotometric method:

4.2.1 Sampler

4.2.2 Pump

4.2.3 Cyanide Manifold

4.2.4 SCIC Spectrophotometer (or equivalent) with 15 mm flow cells and 570 nm filters

4.2.5 Data System

4.2.6 Glass or plastic tubes for the sampler

5 Reagents and Standards

5.1 Matrix matching with the samples is mandatory for all blanks, standards, and QC samples to avoid inaccurate concentration values due to differences in salt content and pH.

5.2 Preparation Reagents:

5.2.1 Cadmium carbonate, powdered

5.2.2 Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 1 L of reagent water (1 mL ≈ 1000 µg CN). Standardize by titrating with 0.0192 N $AgNO_3$ using a few drops of Rhodanine indicator. Note: The analyst should become familiar with the yellow to brownish-pink end point of the titration and the amount of indicator to be used before actually titrating the samples. A 5 or 10 mL microburet may be conveniently used to obtain a more precise titration.

5.2.3 Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 g $AgNO_3$ crystals and drying to constant weight at 40 °C. Weigh 3.2647 g of dried $AgNO_3$, dissolve in distilled water, and dilute to 1 L using reagent water. 1 mL of this solution will titrate approximately 1000 µg of CN.

5.2.4 Rhodanine indicator: Dissolve 20 mg of p-dimethyl-aminobenzalrhodanine in 100 mL of acetone.

5.2.5 Standard cyanide solution, intermediate: Dilute 50.0 mL of stock (5.2.2) to 1 L with reagent water (1 mL = 50 μg CN).

5.2.6 Standard cyanide solution, working: Prepare fresh daily by diluting 100.0 mL of intermediate cyanide solution (5.2.5) to 1 L with reagent water and store in a glass stoppered bottle (1 mL = 5.0 ug CN).

5.2.7 Sodium hydroxide solution, 1.25 N: Dissolve 50 g of NaOH in reagent water and dilute to 1 L with reagent water.

5.2.8 Sodium hydroxide solution, 0.25 N: Dilute 200 ml of 1.25 N NaOH (5.3.6) to 1 L with reagent water or dissolve 10 g of NaOH in reagent water and dilute to 1 L with reagent water.

Note: All cyanide solutions should be stored in amber bottles in the dark at 4 °C to prevent loss of cyanide through photodissociation.

5.2.9 Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $NaH_2PO_4 \cdot H_2O$ in 1 L of reagent water. Refrigerate this solution until ready to use. Bring to room temperature before using.

5.2.10 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. Bring to room temperature before using. Prepare fresh weekly.

5.2.11 Color Reagent. One of the following may be used:

5.2.11.1 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 concentrated HCl (sp gr 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.

5.2.11.2 Pyridine-pyrazolin-5-one solution:

5.2.11.2.1 3-Methyl-1-phenyl-2-pyrazolin-5-one reagent, saturated solution: Add 0.25 g of 3-methyl-1-phenyl-2-pyrazolin-5-one to 50 mL of reagent water, heat to 60°C with stirring. Cool to room temperature.

5.2.11.2.2 3,3'Dimethyl-1,1'-diphenyl [4,4'-bi-2 pyrazolin]-5,5'dione (bispyrazolone): Dissolve 0.01 g of bispyrazolone in 10 mL of pyridine.

5.2.11.2.3 Pour solution (5.2.11.2.1) through nonacid-washed filter paper. Collect the filtrate. Through the same filter paper, pour solution (5.2.11.2.2) collecting the filtrate in the same container as filtrate from (5.2.11.2.1). Mix until the filtrates are homogeneous. The mixed reagent develops a pink color that does not affect the color production with cyanide if used within 24 hours of preparation.

5.2.12 Phosphate buffer: Dissolve 138 g of $NaH_2PO_4.H_2O$ in reagent water and dilute to 1 liter. Add 0.5 mL of Brij-35 (available from Technicon). Store at 4°C (±2°C).

6 Procedure

6.1 Standards and samples may be analyzed manually or by using a semi-automated spectro-photometer. The semi-automated analysis should be performed in accordance with manufacturer's guidelines.

6.1.1 Calibration: Prepare a minimum of three standards and a blank by pipetting suitable volumes of standard solution into 100 mL volumetric flasks. NOTE: One calibration standard must be at the Contract Required Detection Limit (CRDL) of 10 µg/L.

To each undistilled standard, add 50 mL of 0.25 N sodium hydroxide, then add the appropriate amount of working standard as suggested from the table below. At the time of calibration, add the sodium phosphate, chloramine-T, and color reagent, then dilute to 100 mL with reagent water. Record the absorbance of each standard within the recommended development time for the color reagent used. Standards must bracket the concentration of the samples. If dilution of a high sample is required, use the blank solution as a diluent. As an example, standard solutions could be prepared as follows:

mL of Working Standard (1.0 mL = 5 ug CN)	Conc. µg/L CN in 100 mL
0	Blank
0.1	5
0.2	10
0.5	25
1.0	50
2.0	100*
2.0	100

* standard suggested for distillation. Sodium phosphate, chloramine-T, and color reagent are not added prior to distillation. After distillation, the standard is analyzed as if it were a sample.

6.1.1.1 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 should find and correct the cause of the apparent error before proceeding.

6.1.1.2 Perform a linear regression of standard absorbance vs. cyanide concentration in µg/L.

6.1.2 For sample analysis, withdraw 50.0 mL or less of the diluted solution (treated for sulfides, if necessary) from the flask and transfer to a 100.0 mL volumetric flask. Add 15.0 mL of sodium phosphate solution and mix.

6.1.2.1 Pyridine-barbituric acid method: Add 2 mL of chloramine-T solution and mix. After 1 to 2 min, add 5 mL of pyridine-barbituric acid solution and mix. Dilute to mark with reagent water and mix again. Allow 8 min for color development, then read absorbance at 578 nm in a 1 cm cell within the next 7 minutes.

6.1.2.2 Pyridine-pyrazolone method: Add 0.5 mL of chloramine-T solution and mix. After 1 to 2 min, add 5 mL of pyridine-pyrazolone solution and mix. Dilute to mark with reagent water and mix again. After 40 min, read absorbance at 620 nm in a 1 cm cell. NOTE: More than 0.5 mL of chloramine-T will prevent the color from developing with pyridine-pyrazolone.

7 Calculations

7.1 When the manual method is used in conjunction with a full-sized distillation or midi-distillation, calculate the cyanide, in µg/L, in the original sample as follows:

$$C_s = C_m \, x \, \frac{V_m}{V_s} \, x \, \frac{V_{da}}{V_{aa}}$$

where: C_s = µg/L CN in the original sample,
C_m = µg/L CN in the measured sample as read from the standard curve,
V_m = measurement solution volume after final dilution (100 mL),
V_s = volume of original sample taken for distillation (500 mL for full-size and 50 mL for midi-distillation),
V_{da} = final volume of diluted scrubber solution (250 mL for full-size and 50 mL for midi-distillation),
V_{aa} = aliquot of diluted scrubber solution taken for analysis (50 mL),

Note: If recommended volumes are used without dilution, all of the volume terms for the full-size distillation will cancel and the original sample concentration will be the same as the measured sample concentration. The volume terms for the midi-distillation will reduce to a factor of two and the original sample concentration will be twice the measured sample concentration. If other than the recommended volumes are used, actual values will have to be substituted in the above equation.

7.2 When the semi-automated method is used in conjunction with a full-sized distillation or midi-distillation, calculate the cyanide, in µg/L, in the original sample as follows:

$$C_s = C_m \times \frac{V_{da}}{V_s} \times D$$

where: C_s = µg/L CN in the original sample,
C_m = µg/L CN in the measured sample as read from the standard curve,
V_s = volume of original sample taken for distillation (500 mL for full-size and 50 mL for midi-distillation),
V_{da} = final volume of diluted scrubber solution (250 mL for full-size and 50 mL for midi-distillation),
D = any dilution factor necessary to bracket a sample value within standard values.

Note: If recommended volumes are used without dilution, the volume terms for the full-size distillation will reduce to a factor of 0.5 and the original sample concentration will be one-half the measured sample concentration. The volume terms for the midi-distillation

will cancel and the original sample concentration will be the same as the measured sample concentration. If other than the recommended volumes are used, actual values will have to be substituted in the above equation.

7.2.1 If dilutions were performed, the appropriate corrections must be applied to the sample values. The dilution factor used is reported on Form XIV.

7.3 Appropriate concentration units must be specified on the required forms. The quantitative values shall be reported in units of micrograms per liter (μg/L) for aqueous samples, no other units are acceptable.

8 Quality Control

8.1 Quality control must be performed as specified in Exhibit E.

8.2 All QC data must be submitted with each data package as specified in Exhibit B.

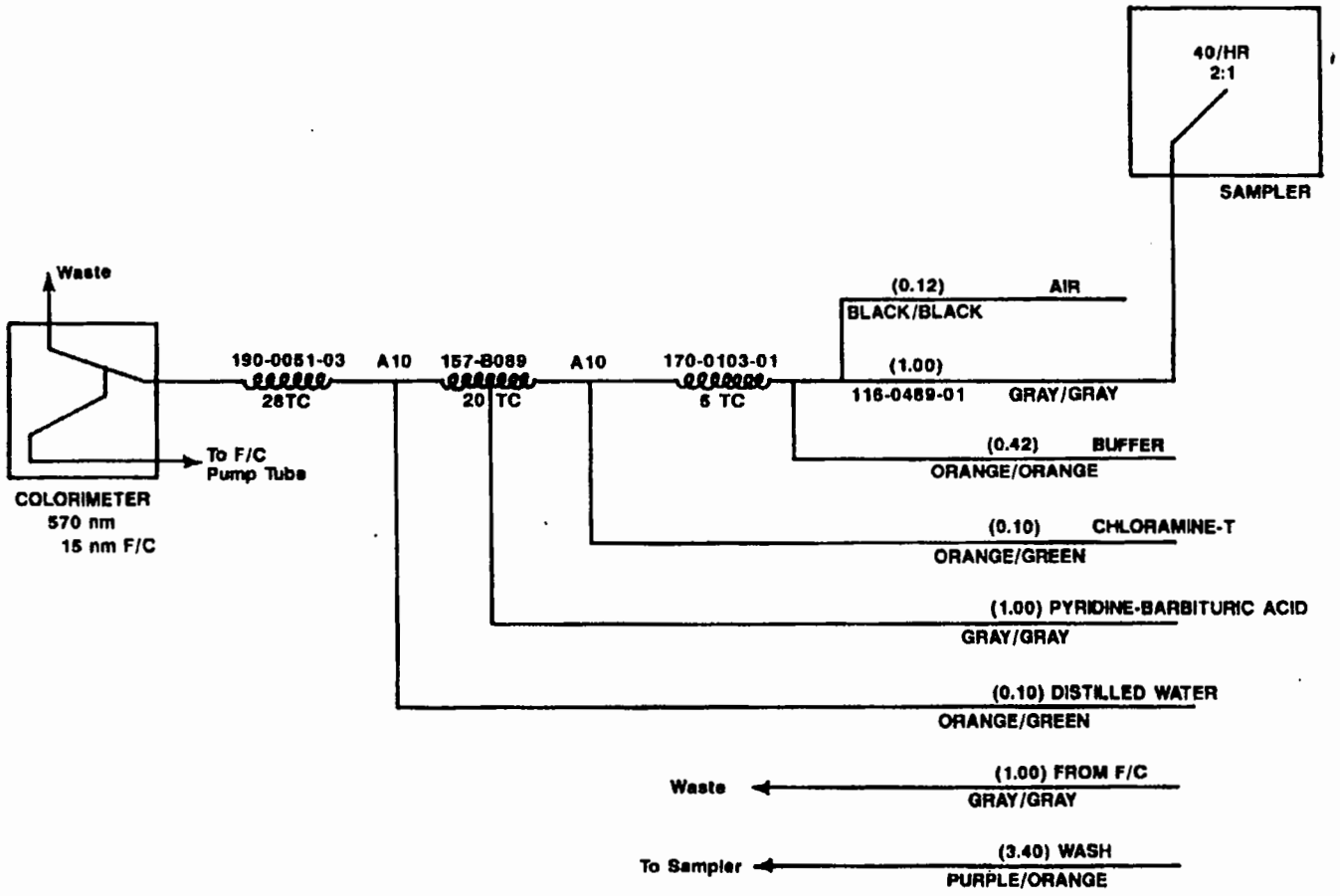

Figure 4. Cyanide Manifold

EXHIBIT E: Quality Assurance/Quality Control

SECTION I - General QA/QC Procedures and Definitions

1. The purpose of this document is to specify a uniform set of QA procedures for the analysis of inorganic constituents of samples, documentation of the methods used and its performance, and verification of the sample data generated. The program will also assist laboratory personnel in recalling and defending their actions under cross examination if required to present court testimony in enforcement case litigation.

2. The primary function of the QA/QC program is to define procedures for evaluating and documenting sampling and analytical methodologies, and for data reduction and reporting. The objective is to provide a uniform basis for sample handling, analysis, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. Although it is impossible to address all analytical situations in one document, the approach taken here is to define minimum requirements for all major steps relevant to any inorganic analysis. In many instances where methodologies are available, specific QC procedures are incorporated into the method documentation (Exhibit D). Ideally, samples involved in enforcement actions are analyzed only after the methods have met the minimum performance and documentation requirements described in this document.

3. The quality assurance/quality control (QA/QC) procedures defined herein must be used by the Contractor when performing the methods specified in Exhibit D. When additional QA/QC procedures are specified by the methods in Exhibit D, the Contractor must also follow these procedures.

   3.1 The cost of performing all QA/QC procedures specified in this Statement of Work (SOW) is included in the price of performing the bid lot. An exception is the analysis of duplicate, spike, and performance evaluation samples (PES) which shall be considered separate sample analyses.

4. The Contractor is required to participate in the Laboratory Audit Study Program conducted by Environmental Protection Agency (EPA). The Contractor must perform and report to Sample Management Office (SMO) and the appropriate Region, as specified in Exhibit B, quarterly verification of instrument detection limits (IDLs) by the method specified in Exhibit D, for each instrument used to perform under this contract. All IDLs must meet the Contract Required Detection Limits (CRDLs) specified in Exhibit C (Table I). For inductively coupled plasma (ICP) methods, the Contractor must also report, as specified in Exhibit B, all elemental expressions used.

5. In this Exhibit, as well as other places within this SOW, the term "analytical sample" is used when discussing the required frequency or placement of certain QA/QC measurements. The term "analytical sample" is defined in the glossary, Exhibit G. As the term is used, analytical samples include all field samples and performance evaluation samples. Matrix spikes, analytical/post-digestion spikes, duplicates, serial dilutions, laboratory check samples (LCS), interference check solutions (ICS), CRDL standards (CRI), preparation blanks, linear-range determination standards (LRS), memory test solutions, and tuning solutions are also included under this definition. Calibration verification standards, i.e., initial calibration verification (ICV), initial calibration blank (ICB), continuing calibration verification (CCV), and continuing calibration blank (CCB) solutions are the only analyses in a run that are not considered to be analytical samples. A "frequency of 10%" means once every 10 analytical samples. Note: Calibration verification samples (ICVs and CCVs) and calibration verification blanks (ICBs and CCBs) are not counted as analytical samples when determining 10%

frequency.

6. In order for the QA/QC information to reflect the status of the samples analyzed, all samples and their QA/QC analysis must be analyzed under the same operating and procedural conditions.

   6.1 If any QC measurement fails to meet contract criteria, the analytical measurement may not be repeated prior to taking the appropriate corrective action as specified in Exhibit E.

   6.2 The Contractor must report all QC data in the exact format specified in Exhibits B.

7. Standard laboratory practices for cleaning glassware and apparatus and for using reagents, solvents, and gases must be adhered to by laboratory personnel. For additional guidelines regarding these general laboratory procedures, see Sections 4 and 5 of the Handbook for Analytical Quality Control in Water and Wastewater Laboratories EPA-600/4-79-019, U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory, Cincinnati, Ohio, September 1982.

SECTION II: Quality Assurance Program and Plan

1. Quality Assurance Program

The Contractor shall establish a Quality Assurance (QA) program with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, all of the documentation required during data collections, and the quality assessment measures performed by management to ensure acceptable data production.

2. Quality Assurance Plan

2.1 As part of the QA program, the Contractor shall prepare a written Quality Assurance Plan (QAP) which describes the procedures that are implemented to achieve the following:

- Maintain data integrity, validity, and useability.
- 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.
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

2.2 QAP Format - The QAP must present, in specific terms, the policies, organization, objectives, functional guidelines, and specific quality assurance (QA) and quality control (QC) activities designed to achieve the data quality requirements of this contract. Where applicable, Standard Operating Procedures (SOPs) pertaining to each element shall be included or referenced as part of the QAP. The QAP must be available during on-site laboratory evaluations and upon written request by the Administration Project Officer (APO) or the Technical Project Officer (TPO). The suggested format and contents of the QAP are given in Figure E-1.

2.3 QAP Submission - During the contract solicitation process, the Contractor is required to submit a QAP to the APO. Within sixty (60) days after contract award, the Contractor shall mainatin on file a revised QAP, fully compliant with the requirements of this contract. The revised QAP is the official QAP under the contract. The revised QAP must include changes resulting from:

- The Contractor's internal review of their organization, personnel, facility, equipment, policy and procedures.
- The Contractor's implementation of the requirements of the contract.
- The Agency's review of the laboratory evaluation sample data, bidder-supplied documentation.
- Recommendations made during the preaward on-site laboratory evaluation.

2.4 QAP Amendments - During the term of contract, the Contractor shall revise and maintain on file, with all previous revision, an amended QAP within 30 days of the following circumstances:

- The Agency modifies the contract
- The Agency notifies the Contractor of deficiencies in the QAP document
- The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance
- The Contractor identifies deficiencies resulting from their

internal review of the QAP document
- The Contractor's organization, personnel, facility, equipment, policy or procedures change
- The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy or procedures changes.

2.5 When the QAP is amended, all changes must be clearly marked with a bar in the margin indicating where the change is found in the document, or by highlighting the change by underlining, bold printing, or using a different print font. The amended section pages must have the date on which the changes were implemented.

2.6 QAP Archival - The Contractor shall maintain a master QAP which incorporates the original QAP and all subsequent amendments. The Contractor shall provide a copy of the master QAP or any of its amendments to the designated recipients within 14 days of a request by the TPO or APO.

3. Corrective Action - If a Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but the Agency is not limited to the following actions:

- reduction in the number of samples sent under this contract
- suspension of sample shipment to the Contractor
- ICP-MS tape audit
- data package audit
- on-site laboratory evaluation
- remedial performance evaluation sample
- contract sanctions, such as a Cure Notice

1. Organization and Personnel
1.1 QA Policy and Objectives
1.2 QA Management
    1.2.1 Organization
    1.2.2 Assignment of QA and QC Responsibilities
    1.2.3 Reporting Relationships
    1.2.4 QA Document Control Procedures
    1.2.5 QA Program Assessment Procedures
1.3 Personnel
    1.3.1 Resumes
    1.3.2 Education and Experience Pertinent to this Contract
    1.3.3 Training Program Completed

2. Facilities and Equipment
2.1 Instrumentation and Backup Alternatives
2.2 Maintenance Activities and Schedules

3. Document Control
3.1 Laboratory Notebook Policy
3.2 Sample Tracking/Custody Procedures
3.3 Logbook Maintenance and Archiving Procedures
3.4 Sample Delivery Group (SDG) File Organization, Preparation, and Review Procedures
3.5 Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs
3.6 Process for Revision of Technical or Documentation Procedures

4. Analytical Methodology
4.1 Calibration Procedures and Frequency
4.2 Sample Preparation Procedures
4.3 Sample Analysis Procedures
4.4 Standards Preparation Procedures
4.5 Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

5. Data Generation
5.1 Data Collection Procedures
5.2 Data Reduction Procedures
5.3 Data Validation Procedures
5.4 Data Reporting and Authorization Procedures

6. QA
6.1 Data QA
6.2 Systems/Internal Audits
6.3 Performance/External Audits
6.4 Corrective Action Procedures
6.5 QA Reporting Procedures
6.6 Responsibility Designation

7. QC
7.1 Solvent, Reagent, and Adsorbent Check Analysis
7.2 Reference Material Analysis
7.3 Internal QC Checks
7.4 Corrective Action and Determination of QC- Limit Procedures
7.5 Responsibility Designation

Figure E-1. Format and Content of QAP

SECTION III - Standard Operating Procedures

To obtain reliable results, adherence to prescribed analytical methodology is imperative. When an operation is performed on a repetitive basis, reproducibility is best accomplished through the use of SOPs. As defined by the EPA, an SOP is a 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.

SOPs prepared by the Contractor must be functional, i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts. They must be available at workstations, as appropriate. SOPs must be reviewed regularly and updated when contract, facility, or Contractor procedural modifications are made. Old revisions of SOPs must be archived for future reference in usability or evidentiary situations. Finally, SOPs must be subject to document control procedures which preclude the use of outdated or inappropriate SOPs.

A complete set of SOPs must be available for inspection by the EPA during on-site laboratory evaluations. All SOPs, as presented to the Agency, must reflect activities as they are currently performed in the laboratory. During on-site laboratory evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs.

The format for SOPs and a list of required SOPs are given in Parts A and B of this Section. Part C of this Section describes the submission requirements for SOPs.

PART A - SOP Format

The format for SOPs may vary depending upon the procedure for which they are written. A general format and minimum requirements for SOPs is given in Figure E-2. Overall, SOPs must be consistent with current EPA regulations, guidelines, CLP contract requirements, and instrument manufacturers's operating manuals. They also must describe the corrective measures and feedback mechanisms used when analytical results do not meet the CLP protocol QC requirement. Finally, they must require that sufficient data be recorded (including operator observations and actions and instrument data) to document the performance of all tasks required by CLP protocol and to validate all results reported by the contractor.

1. Title page
2. Scope and application
3. Definitions
4. Procedures
5. Quality control (QC) limits
6. Corrective action procedures, including procedures for secondary review of information being generated
7. Documentation description and example forms
8. Miscellaneous notes and precautions
9. References

Figure E-2. General SOP Format

PART B - SOPS Required

The following SOPs are required by the Agency. Included with each are a list of elements which should be included in the SOP.

1. Evidentiary SOP. A more detailed discussion of evidentiary SOPs required chain-of-custody and document control are discussed in Exhibit F

2. Sample Receipt and Storage

   - Sample receipt and identification logbooks
   - Refrigerator temperature logbooks
   - Security precautions

3. Sample preparation

4. Glassware cleaning

5. Calibration of balances, etc.

   - Procedures
   - Frequency requirements
   - Preventative maintenance schedule and procedures
   - Acceptance criteria and corrective actions
   - Logbook maintenance authorization

6. Analytical procedures (for each analytical system)

   - Instrument performance specifications
   - Instrument operating procedures
   - Data acquisition system operation
   - Procedures when automatic quantitation algorithms are overridden
   - QC-required parameters
   - Analytical run/injection logbooks
   - Instrument error and editing flag descriptions and resulting corrective actions

7. Maintenance activities (for each analytical system)

   - Preventative maintenance schedule and procedures
   - Corrective maintenance determinants and procedures
   - Maintenance authorization

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
   - QC and corrective action measures

9. Data reduction procedures

   - Data processing systems operation
   - Outlier identification methods
   - Identification of data requiring corrective action
   - Procedures for format and/or forms for each operation

10. Documentation policy/procedures

- Laboratory/analyst notebook policy, including review policy
- Complete SDG file contents
- Complete SDG File organization and assembly procedures, including review policy
- Document inventory procedures, including review policy

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 ensure that hard copy and diskette deliverables are complete and compliant with the requirements in SOW Exhibits B and H.
- Procedures to ensure that hard copy deliverables are in agreement with their comparable diskette deliverables.
- Demonstration of internal 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).
- Documentation of audit reports, (internal and external), response, corrective action, etc.

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.
- Data base 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.
- Specifications for staff training procedures.

PART C - SOPs Delivery Requirements

1. SOP Submission - During the contract solicitation process, the Contractor is required to submit SOPs to the APO. Within sixty (60) days after contract award, the Contractor shall maintain on file a revised SOP, fully compliant with the requirements of this contract. The revised SOP is the official SOP under the contract. The revised SOPs must include changes resulting from:

   - The Contractor's internal review of their procedures.
   - The Contractor's implementation of the requirements of the contract.
   - The Agency's review of the laboratory evaluation sample data, bidder-supplied documentation.
   - Recommendations made during the preaward on-site laboratory evaluation.

2. SOP Revisions - During the term of contract, the Contractor must revise SOPs within 30 days of the following circumstances:

   - The Agency modifies the contract and the modification affects an SOP
   - The Agency notifies the Contractor of deficiencies in the SOP documentation
   - The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance
   - The Contractor identifies deficiencies resulting from their internal review of their SOP documentation
   - The Contractor's procedures change
   - The Contractor identifies deficiencies resulting from the internal review of their procedures

   2.1 When an SOP is amended, all changes must be clearly marked with a bar in the margin indicating where the change is found in the document, or by highlighting the change by underlining, bold printing, or using a different print font. The amended section pages must have the date on which the changes were implemented.

   2.2 When the SOPs are amended or new SOPs are written, the Contractor shall document in a letter to the TPO the reasons for the changes. An alternate delivery schedule for the submittal of the letter may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the TPO or APO, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the TPO, APO, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The TPO or APO will not grant an extension of more than 30 days for amending or writing new SOPs. The TPO or APO will not grant an extension of more than 14 days for submission of the letter documenting the reasons for the changes. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO. The Contractor shall send a complete set of current SOPs to the designated recipients within 14 days of a request by the TPO or APO.

3. Corrective Action - If a Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but the Agency is not limited to the following actions:

- reduction in the number of samples sent under this contract
- suspension of sample shipment to the Contractor
- data package audit
- on-site laboratory evaluation
- remedial performance evaluation sample
- contract sanctions, such as a Cure Notice

SECTION IV - Required QA/QC Operations

This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements of the contract. The following QA/QC operations must be performed as described in this Exhibit:

- Sample Analysis
- Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) Tuning and Mass Calibration
- Instrument Calibration
- ICV and CCV analysis
- CRDL Standards (CRI/CRA) analysis
- Linear Range Standard (LRS) Analysis
- ICB, CCB, and PB Analyses
- ICP and ICP-MS ICS Analyses
- Matrix Spike Sample Analysis (S)
- Duplicate Sample Analysis (D)
- LCS Analysis
- PES
- Serial Dilution Analysis (L)
- Internal Standards Evaluation for ICP-MS
- Instrument Detection Limit (IDL) Determination
- Elemental Expression for ICP and ICP-MS
- Graphite Furnace Atomic Absorption (GFAA) QC Analysis

If a Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but the Agency is not limited to the following actions:

reduction in the number of samples sent under this contract,
suspension of sample shipment to the Contractor,
ICP-MS data audit,
data package audit,
an on-site laboratory evaluation,
remedial performance evaluation sample, and/or
contract sanctions, such as a Cure Notice.

1. Sample Analysis

After sample preparation all samples must be analyzed initially without any further dilution. If ICP or ICP-MS results are outside of the linear range for the analyte, the sample must be diluted so that the concentration is within the linear range. For results obtained by other methods, if the concentration exceeds the calibrated range, the sample must be diluted so that the concentration is within the calibrated range of the instrument.

2. ICP-MS Tuning and Mass Calibration

2.1 Guidelines - Guidelines for mass calibration and tuning are given in Exhibit D. Resolution and mass calibration must be performed for each ICP-MS system, each time the instrument is set up. The resolution and mass calibration must be verified immediately prior to instrument calibration. The resolution and mass calibration must also be verified at the end of each analytical run, or every 8 h, whichever is more frequent. The tuning solution must be analyzed after the final CCV/CCB in the run. The mass calibration and tuning times as well as their verification times must be included in the raw data.

2.2 Tuning Verification Solution - A 100-ppb solution of Li, Co, In, and Tl must be used as a tuning verification solution. The intensities and relative response ratios of the tuning criteria are recommended

in Table VII, Exhibit C.

2.3 Resolution and Mass Calibration Criteria - The resolution and mass calibration criteria must be within the control limits in Table VII, Exhibit C. If not, the analysis must be terminated, the problem must be corrected, and all instrument results since the last compliant mass calibration and resolution check must be rerun in a new analytical run.

2.4 Mass Calibration and Resolution Check Reporting - The values for the initial and subsequent mass calibrations and resolution check shall be recorded on Form XV - LCIN for ICP-MS analyses, as indicated in Exhibit B.

3. Instrument Calibration

3.1 Guidelines - Guidelines for instrumental calibration are given in EPA 600/4-79-0201 and Exhibit D. Calibrate all instruments according to the instrument manufacturer's instructions or as specified in Exhibit D. At least one blank and one standard must be used for ICP and ICP-MS systems. All other systems must have a calibration standard at the CRDL, a blank, and at least two other standards. Systems that use nonlinear calibration curves must use at least three additional standards that cover the upper and lower ranges of the curve. Instruments must be calibrated daily or once every 24 h and each time the instrument is set up. The date and time of instrument calibration must be reported in the raw data.

3.2 Standards - Calibration standards must be prepared by diluting stock solutions at the time of analysis, and must be discarded after use. The date and time of the preparation and analysis of the standards must be reported in the raw data. Calibration standards must be prepared using the same matrix as the preparation blank.

3.3 Correlation Coefficient - Calibration curves with three or more points must have a correlation coefficient of 0.995 or greater before any analysis may be started. The correlation coefficient for each calibration curve must be clearly documented and must be submitted with the raw data.

4. Initial Calibration Verification (ICV)

4.1 Analysis - Immediately after calibration and prior to sample analysis, an ICV must be analyzed to verify the accuracy of the calibration. An ICV analysis is required for every analyte reported. The analysis conditions for the ICV must be the same as those for the analytical samples. If multiple sets of analytical conditions are used to measure and report results for an analyte, the ICV must also be measured and reported for each set of analytical conditions. For each ICV analysis, a recovery is calculated by

$$\%R = \frac{ICV_{measured}}{ICV_{true}} \times 100$$

When the ICV recovery for an analyte exceeds the control limits listed in Table III of Exhibit C, the analysis must be terminated, the problem must be corrected, the instrument must be recalibrated, and the calibration must be verified.

4.2 Source - ICV solution(s) can be obtained from the EPA, a commercial vendor, or prepared in-house. The source of the analytes in an ICV solution must be independent of the source used for the calibration standards, and the concentration of the analyte in the source material must be certified. Finally, the concentration of an analyte in the ICV should be in the mid-calibration range and different than the calibration standard concentrations.

4.3 Cyanide - The ICV solution for cyanide also serves as the LCS and is distilled prior to analysis. Furthermore, it must be distilled with the batch of samples to be analyzed (i.e., for which it serves as the LCS).

4.4 Reporting - The true and measured concentrations for the ICV as well as the percent recoveries must be recorded on Form II - LCIN for all analytical systems, as indicated in Exhibit B.

5. Continuing Calibration Verification (CCV)

5.1 Analysis - A CCV solution must be analyzed periodically throughout an analytical run to verify the stability of the calibration. The CCV solution(s) must be analyzed at the beginning of an analytical run (within 2 hours of the first analytical sample in the run), at the end of the run (within 2 hours of the last analytical sample), and at a minimum frequency of 10% or 2 hours during the run (whichever is more frequent). A frequency of 10% corresponds to 10 analytical samples between CCVs. A CCV analysis is required for every analyte reported. The analysis conditions for the CCV must be the same as those for the analytical samples. If multiple sets of analytical conditions are used to measure and report results for an analyte, the CCV must also be measured and reported for each set of analytical conditions. For each CCV analysis, a recovery is calculated by

$$\%R = \frac{CCV_{measured}}{CCV_{true}} \times 100$$

5.2 When the CCV recovery for an analyte exceeds the control limits listed in Table II of Exhibit C, the analysis must be terminated, the problem must be corrected, and the CCV must be reanalyzed. If the reanalysis yields a CCV value within the specified control limits, then the preceding 10 analytical samples or all analytical samples analyzed since the last compliant CCV must be reanalyzed for the analytes affected. Otherwise the instrument must be recalibrated, the calibration must be verified, and the affected samples must be reanalyzed in a new analytical run. If an affected analytical sample is an instrument-related QC sample such as an ICS, CRI, or LRS, then the problem must be corrected and the standards must be reanalyzed within the 8-h limit for those standards. If not, all samples and QC samples in the run must be reanalyzed.

5.3 Source - CCV solutions can be obtained from a commercial vendor or prepared in-house. The concentration of the analyte in the source material must be certified. The analyte concentrations in the CCV standard must be at a concentration near the mid-point of the calibrated range. Also, the same CCV standard must be used throughout an analytical run.

5.4 Reporting - The true and measured concentrations for the CCV as well

as the percent recoveries must be recorded on Form II - LCIN for all analytical systems, as indicated in Exhibit B.

6. CRDL Standard (CRI/CRA)

6.1 Analysis - A standard with a concentration at or near the CRDL is analyzed as part of an analytical run to verify the analysis accuracy near the CRDL for all analytical systems. For ICP and ICP-MS analyses, the standard is referred to as the CRI standard. For other analyses, it is referred to as the CRA standard. The CRI and CRA standards must be analyzed at the beginning (after the ICV) and end of each analytical run, or a minimum of two times every 8 consecutive hours, whichever is more frequent. A CRI or CRA analysis is required for every analyte reported. The analysis conditions for the CRI/CRA must be the same as those for the analytical samples. If multiple sets of analytical conditions are used to measure and report results for an analyte, the CRI/CRA must also be measured and reported for each set of analytical conditions. For each CRI/CRA analysis, a recovery is calculated by

$$\%R = \frac{CRI/CRA_{measured}}{CRI/CRA_{true}} \times 100$$

6.2 The CRI/CRA recovery for an analyte must be within the control limits listed in Table II of Exhibit C. If the CRI/CRA at the beginning of the analytical run is not within the control limits, the analysis must be terminated, the problem must be corrected, and the instrument must be recalibrated. All samples associated with non-compliant CRI/CRA must be reanalyzed in a new analytical run. If the CRI/CRA at the end of the run is not within the control limits, then all sample results between the CRI/CRA standards must be flagged with a "C" on Form I-LCIN in the Q qualifier column.

6.3 Source - The CRDL standard may be obtained from a commercial vendor or prepared in-house. The concentration of the analyte in the source material must be certified. The analyte concentrations in the CRI standard must be twice the CRDL concentration while the analyte concentrations in the CRA standard must be equal to the CRDL concentration.

6.4 Reporting - The true values, measured concentrations, and percent recovery for the initial and subsequent CRDL standards for all analysis systems must be recorded on Form III - LCIN, as indicated in Exhibit B.

7. Linear Range Determination Standard (LRS)

7.1 Analysis - For ICP and ICP-MS analyses, the accuracy of analyses can extend above the calibrated range. To verify the accuracy of analyses above the calibrated range, a LRS standard can be analyzed at the beginning (after the ICV) and end of each analytical run, or a minimum of two times every 8 consecutive hours, whichever is more frequent. A LRS analysis is required for every analyte reported with a concentration above the calibrated range. The analysis conditions for the LRS must be the same as those for the analytical samples. If multiple sets of analytical conditions are used to measure and report results for an analyte, the LRS must also be measured and reported for each set of analytical conditions. For each LRS analysis, a

recovery is calculated by

$$\%R = \frac{LRS_{measured}}{LRS_{true}} \times 100$$

7.2 If the LRS recovery for an analyte is within 90-110%, sample results up to the LRS concentration can be reported. If the recovery is not within the control limits, sample results above the calibrated range cannot be reported. Such samples would require dilution and reanalysis.

7.3 Source - The LRS standard may be obtained from a commercial vendor or prepared in-house. The concentration of the analyte in the source material must be certified. The analyte concentrations in the LRS standard is determined by the operator and is near the upper linear range of the instrument (under the analytical conditions used).

7.4 Reporting - The true values, measured concentrations, and percent recovery for the initial and subsequent LRS standards for all analysis systems must be recorded on Form IV - LCIN, as indicated in Exhibit B.

8. Calibration Blanks (ICV and CCV)

8.1 Analysis - Calibration blanks are analyzed as samples periodically throughout an analytical run in order to verify the accuracy of the calibration at a concentration of "0", to monitor the stability of the calibration at a "0" concentration, and to check for carryover between samples. The initial calibration blank (ICB) is analyzed immediately after analyzing the ICV solution(s). Continuing calibration blanks (CCB) must be analyzed after the LRS solution(s), memory test solution (MTS), and every CCV solution. ICB and CCB analyses are required for every analyte reported. The analysis conditions for the ICB and CCBs must be the same as those for the analytical samples. If multiple sets of analytical conditions are used to measure and report results for an analyte, the ICB and CCBs must also be measured and reported for each set of analytical conditions. Analytical conditions include steps performed between analyses, such as flushing, rinsing, and cleaning.

8.2 If the magnitude (absolute value) of an ICB or CCB for an analyte exceeds its IDL, the result must be reported on Form V - LCIN. If the absolute value of an ICB or CCB result exceeds the CRDL, analysis must be terminated, the problem must be corrected, the instrument must be recalibrated, and all analytical samples analyzed since the last compliant ICB or CCB must be reanalyzed in a new analytical run. If any of the affected analytical samples are instrument related QC samples, such as ICS, CRI, LRS, or MTS, then the problem must be corrected and the standards must be reanalyzed within the 8-hour limit for those standards. If not, all samples and QC in the run must be reanalyzed.

8.3 Source - Calibration blanks are prepared using the matrix solution of the calibration standards.

8.4 Reporting - Results for the ICB and CCBs must be recorded on Form IV - LCIN for all analytical systems, as indicated in Exhibit B.

9. Preparation Blank (PB) Analyses

9.1 Analysis - A PB is prepared and analyzed with every SDG or batch of samples prepared, whichever is more frequent. The results are used to detect significant contamination from sample preparation.

9.2 If the PB concentration for an analyte is greater than the CRDL, corrective action may be necessary. For samples associated with the PB, results for the analyte may be reported if the concentration is greater than 10 times the concentration measured in the PB or less than the CRDL. Otherwise, the samples must be redigested and reanalyzed for that analyte.

9.3 Source - At least one PB, consisting of reagent water (See Exhibit D, Part IV Section 5.2), must be prepared and analyzed with every SDG, or with each batch of samples prepared, whichever is more frequent.

9.4 If more than one PB is required for the same method, then the first batch of samples is to be associated with PB 1, the second batch of samples with PB 2, etc. Each data package must contain the results of all PB analyses that are associated with the samples in that SDG.

9.5 Reporting - The values for PBs for all analysis systems must be recorded on Form V - LCIN, as indicated in Exhibit B.

10. ICP and ICP-MS Interference Check Sample Analysis

10.1 Analysis - Inherent in ICP analyses are interelement correction factors and spectral background corrections. Inherent in ICP-MS analyses are interelement correction equations (also known as elemental expressions). For ICP, to verify the accuracy of the background correction points and interelement correction factors and equations, two interference check samples (ICS) are analyzed at the beginning and end of each analytical run, or every 8 hours, whichever is more frequent. For ICP-MS, the ICS solutions must be analyzed once every 8 hours. The ICS samples are identified as ICS-A, which contains only the common interferants, and ICS-AB, which contains both the common interferants and analytes. An ICS analysis is performed by analyzing the ICS-A and ICS-AB solutions in sequence. The analysis conditions for the ICS-A and ICS-AB must be the same as those for the analytical samples. If multiple sets of analytical conditions are used to measure and report results for an analyte, the ICS-A and ICS-AB must also be measured and reported for each set of analytical conditions.

Dilution of the ICS-A or ICS-AB solution prior to analysis is only permitted if an analyte or interferant concentration exceeds the linear range of the instrument. When dilution is necessary, the dilution factor must be kept to a minimum. Results from the diluted ICS analysis are only reported for the specific analytes/interferants which required dilution. Results for the other analytes/interferants must be reported from the undiluted ICS analysis. When dilution is required, the ICS analysis sequence must be undiluted ICS-A, undiluted ICS-AB, diluted ICS-A, diluted ICS-AB.

For the analytes in the ICS-AB solution, a percent recovery is calculated by

$$\%R = \frac{ICS\text{-}AB_{measured}}{ICS\text{-}AB_{true}} \times 100$$

10.2 The absolute value of the analyte concentrations measured in the ICS-A solution must not exceed the CRDL and the recovery of the analytes in the ICS-AB solution must be within the limits of 80-120%, inclusive. If the results are not within the control limits, the analysis must be terminated, the problem must be corrected, the instrument must be recalibrated, and the analytical samples that followed the last compliant ICS analysis (ICS-A and ICS-AB) must be reanalyzed.

10.3 Source - ICS solutions can be obtained from a commercial vendor, or prepared in-house. The interferant and analyte concentrations for ICS-A and ICS-AB are specified in Exhibit C, Table IV.

10.4 Reporting - The true values, measured concentrations, and percent recoveries (ICS-AB only) for the initial and subsequent ICS analyses must be recorded on Form VI - LCIN, as indicated in Exhibit B.

11. Matrix Spike Sample Analysis

11.1 Preparation and Analysis - One matrix spike sample is prepared and analyzed per method with each SDG or batch of samples prepared, whichever is greater. If two or more methods are used to determine and report a given analyte, then the matrix spike sample must be analyzed by each method. A separate preparation is only required if dictated by the analytical method. The matrix spike sample analysis provides information about the effect of the sample matrix on the preparation and measurement methodology. To prepare a matrix spike sample, an aliquot of the field sample is spiked, prior to digestion, with the analytes to measured by the chosen analytical method. After spiking, the sample is prepared and analyzed. The analyte concentrations levels in the spike are specified in Exhibit C, Table III.

Unless otherwise specified by the documents shipped with the samples, use the same field sample for both the matrix spike and duplicate sample analysis. Samples identified as field blanks must not be used for the matrix spike sample.

11.2 Recovery Calculation and Interpretation - A recovery is calculated for each analyte added to the sample in the matrix spike as follows;

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

Where SSR = analyte concentration in the matrix spike sample
SR = analyte concentration in the original sample

SA = true analyte concentration added by the matrix spike

11.3 For SSR or SR which are less than the IDL, a value of "0" is used to calculate the recovery. When the value of SR is less than 4 times the value of SA, the acceptance criteria for the matrix spike recovery are 75-125%, inclusive. When the value of SR is greater than 4 times the value of SA, there are currently no defined acceptance criteria for the matrix spike recovery. If the matrix spike recovery for an analyte is outside of the acceptance criteria, the results for that analyte in all samples associated with that matrix spike sample and determined by the same analytical method must be flagged with the letter "N" on Form I- LCIN and VII - LCIN. If there is more than one matrix spike sample per method per SDG and the recovery for one of them is not within the control limits, all samples analyzed by the same method in the SDG must be flagged.

11.4 Reporting - The results for the sample, matrix spike sample, matrix spike added, and recovery shall be recorded on Form VII - LCIN for all analysis systems, as indicated in Exhibit B.

12. Duplicate Sample Analysis

12.1 Preparation and Analysis - One duplicate sample is prepared and analyzed with each SDG or batch of samples prepared, whichever is greater. The duplicate sample analysis provides information regarding the precision of the preparation and analysis procedures. To prepare a duplicate sample, a duplicate aliquot of the field sample is prepared and analyzed. If analyte concentrations are determined and reported by more than one method, duplicate results for that analyte must also be determined and reported by both methods.

Unless otherwise specified by the documents shipped with the samples, use the same field sample for both the matrix spike and duplicate sample analysis. Samples identified as field blanks must not be used for the duplicate sample.

12.2 Duplicate Precision Calculation and Interpretation - The duplicate precision results are evaluated using either a simple difference (delta) or relative percent difference (RPD), depending upon the analyte concentration. The evaluation process and calculations are summarized below.

SR concentration	DR concentration	Precision Estimator	Control Limit
SR > 5*CRDL	DR > 5*CRDL	RPD	20%
SR > 5*CRDL SR ≤ 5*CRDL 5*CRDL ≥ SR > CRDL SR ≤ 5*CRDL	DR ≤ 5*CRDL DR > 5*CRDL DR ≤ 5*CRDL 5*CRDL ≥ DR > CRDL	delta	± CRDL
SR ≤ CRDL	DR ≤ CRDL	none	none

$$delta = SR - DR$$

$$\% \ RPD = \frac{|SR - DR|}{\left(\frac{SR + SD}{2}\right)} \times 100$$

Where SR = analyte concentration in the original sample
SD = analyte concentration in the duplicate sample

If the duplicate sample results for an analyte are outside the control limits, the results for that analyte in samples associated with that duplicate sample and analyzed by the same method must be flagged with an "*" on Form I - LCIN and Form VIII - LCIN. In the instance where there is more than one duplicate sample per SDG, if one duplicate result is not within the control limits, flag all samples of the same method in the SDG.

12.3 Reporting - The results for the sample, duplicate sample, and RPD (when appropriate) shall be recorded on Form VIII - LCIN for all analysis systems, as indicated in Exhibit B.

13. Laboratory Control Sample (LCS) Analysis

13.1 Source - The LCS sample can be obtained from a commercial vendor, or prepared in-house. If obtained commercially, the vendor must certify the analyte concentrations against NIST-traceable standards. If prepared in-house, the concentration of the analyte in the source material must be certified against NIST-traceable standards. The concentration of analytes in the LCS must be between the CRDL and the upper linear range for the method of analysis.

13.2 Preparation and Analysis - One LCS sample is prepared and analyzed with each SDG or batch of samples prepared, whichever is greater. The LCS sample analysis provides information regarding the accuracy of the preparation and analysis procedures. The LCS is prepared the same as a field sample. If analyte concentrations are determined and reported by more than one method, LCS results for that analyte must also be determined and reported by both methods.

13.3 LCS Recovery Calculation and Interpretation - A recovery is calculated for each analyte in the LCS as follows;

$$\% \ Recovery = \frac{LCS_{measured}}{LCS_{true}} \times 100$$

The default control limits for the LCS are 80-120%, inclusive. These limits are used unless other limits are provided by the LCS supplier. If the LCS results for an analyte are outside the control limits, the problem must be corrected, and the samples

associated with that LCS must be reprepared and reanalyzed for the analyte in question.

13.4 Reporting - The true values, measured values, and percent recoveries for the LCS must be recorded for all analytes on Form IX - LCIN, as indicated in Exhibit B, Section II.

14. Performance Evaluation (PE) Sample Analysis

14.1 Source - PE samples are used by the EPA to monitor contractor performance and are provided either single or double blind samples. Single blind PE samples are provided to a contract lab as a PE sample with unknown analytes and concentrations. Double blind PE samples may be submitted unknown to a contract lab along with routine field samples.

14.2 Preparation and Analysis - Single blind PE samples are prepared according to the directions supplied with the samples. They are analyzed as normal field samples. The Agency will notify the Contractor of unacceptable performance. Unacceptable performance for identification and quantification of analytes in the PES is defined as a score of less than 75%. (See Exhibit E, Section VIII, for additional details).

14.3 Reporting - Results for PE samples are reported the same as field samples.

15. Serial Dilution Analysis

15.1 Preparation and Analysis - In order to check for the presence of matrix interferences during ICP and ICP-MS analyses, a serial dilution analysis is performed. A serial dilution sample is prepared by diluting an aliquot of prepared sample by a factor of five (eg., diluting 10 mL of prepared sample to 50.0 mL). The diluent is reagent watr with the same acid content as the calibration standards. One serial dilution sample is prepared and analyzed with each SDG for both ICP and ICP-MS methods. If analyte concentrations are determined and reported by both methods, then results for serial dilution results must also be determined and reported by both methods.

Unless otherwise specified by the documents shipped with the samples, use the same field sample for the matrix spike, duplicate, and serial dilution sample analysis. Samples identified as field blanks must not be used for the duplicate sample.

15.2 Serial Dilution Recovery Calculation and Interpretation - For each analyte in the serial dilution sample with a concentration at least 20 times the IDL, a serial dilution recovery is calculated by

$$\%\ Recovery = \frac{SDR}{SR} \times 100$$

where

SDR = concentration of the serial dilution sample (corrected for dilution)
SR = concentration of the original sample

The control limits for the serial dilution recovery are 90-110%, inclusive. If the serial dilution recovery is outside the control limits for an analyte, a chemical or physical interference effect must be suspected and the results for that analyte in all samples associated with that serial dilution sample must be flagged with an "E" on Form X - LCIN and Form I - LCIN.

15.3 Reporting - The values for the original sample result, serial dilution sample result, and percent recovery for all serial dilution analyses must be recorded on Form X - LCIN, as indicated in Exhibit B.

16. Internal Standards for ICP-MS

16.1 Preparation and Analysis - In order to correct for physical interferences during ICP-MS analyses, a series of internal standards are added to all samples (QC and field samples) prior to analysis. A minimum of three internal standards, listed in Table IX of Exhibit C, must be used, bracketing the mass range being analyzed. Internal standards may be added manually to aliquots of the samples being analyzed or automatically during analysis by combining the sample stream and an internal standard stream prior to aspiration.

16.2 Relative Intensity Calculation and Interpretation - The corrected intensities for the internal standards in each sample are compared to the corrected intensities for the internal standards in the "0" concentration calibration standard and a relative intensity (%RI) calculated by

$$\%\ RI = \frac{I_{sample}}{I_{STD\text{-}0}} \times 100$$

If the %RI for a given internal standard in a given sample is less than 30%, a physical interference may be affecting the results. If the %RI values for the surrounding QC samples (CCVs and CCBs) are similar, the low %RI values are probably the result of a drop in instrument sensitivity and may indicate that retuning and/or cleaning is necessary. As long as the QC results are acceptable, sample results are acceptable. If the %RI values for the surrounding QC samples is significantly higher, the results for the sample with a low %RI value are probably affected by matrix. The sample must be reanalyzed after performing a 5-fold dilution. If the %RI remains less than 30%, the sample results associated with the low %RI value must be flagged with an "E" on Form XV and on Form I - LCIN. If the affected sample is a matrix spike or duplicate sample, the analytes affected must also be listed in the Comment Section on the appropriate Forms VII - LCIN and VIII - LCIN.

16.3 Reporting - The values for %RI must be reported for each ICP-MS analysis on Form XVI - LCIN as indicated in Exhibit B.

17. Instrument Detection Limit (IDL) Determination

17.1 Frequency - IDLs must be determined for all analytes determined under this contract and the IDL for each analyte must be determined separately for each individual instrument. The IDLs for an

instrument must be determined prior to its use on this contract as described in Section 18.3 and must be updated quarterly thereafter as described in Section 18.4. IDLs must also be determined as described in Section 18.3 any time a change is made to an instrument that will affect its IDLs.

17.2 Requirements - The CRDLs are specified in Table I of Exhibit C. IDLs must be less than or equal to the CRDLs. If the IDL for a given instrument exceeds the CRDL, only results from that instrument greater than 5 times its IDL can be reported.

17.3 Initial Determination

17.3.1 Metals - Calibrate the instrument as performed on a routine basis. Analyze, as unknown samples, 7 separate aliquots of the calibration blank on three different days. The analysis procedure must match that used for routine sample analysis. Calculate the standard deviation for the 7 replicate values on each day and a pooled standard deviation for all three days.

$$S_p = \sqrt{\frac{S_1^2 + S_2^2 + S_3^2}{3}}$$

where S_p = pooled standard deviation
S_n = standard deviations for day 1, day 2, and day 3

The IDL is defined as 3 times the pooled standard deviation, in units of μg/L.

$$IDL = 2.61 \times S_p$$

An IDL must be determined for each set of operating conditions used to analyze samples. (This definition is based upon the 99% prediction interval for the measured concentration of a blank sample. The multiplier has been rounded to one significant figure).

17.3.2 Non-metals - The sample analyzed to estimate the IDL must provide a realistic estimate of measurement variability at concentrations near the detection limit. For the non-metal analytes, depending upon the instrumentation and detection system, this may or may not be possible with a calibration blank. If possible, then the IDL for non-metal analytes is determined as described for metals in Section 18.3.1. If not possible, prepare a standard containing the analytes at concentrations that are 1-3 times the estimated IDL. If an estimated IDL is not known, use the CRDL as an estimate. Substituting this standard for the calibration blank, determine the IDL as described in Section 18.3.1.

17.4 Quarterly Update - Repeat the procedure described in 18.3 (Initial Determination).

17.5 Reporting - The current IDLs must be reported on Form XII - LCIN, and submitted with each data package, for each instrument used (and each set of operating conditions) to produce data in the SDG, as indicated in Exhibit B.

18. Elemental Expressions for ICP-AES and ICP-MS

18.1 The ICP-AES and ICP-MS elemental expression factors must have been determined within 3 months prior to beginning sample analyses under this contract, and at least annually thereafter. Correction factors for spectral and isobaric interferences must be determined at all wavelengths and elemental expressions used for each analyte reported using ICP-AES and ICP-MS. The correction factors must be determined under the same instrument conditions used for sample analysis. If the instrument was adjusted in any way that may affect the interelement correction factors, the factors must be redetermined and the results submitted for use.

18.2 Elemental expression factors must be determined annually. The results of that determination must be reported on Form XIII - LCIN, and submitted with each data package, for all ICP-AES and ICP-MS parameters, for every instrument used to generate data in the SDG, as indicated in Exhibit B.

18.3 Elemental expression factors for internal standards must be reported on Form XIV - LCIN, and submitted with each data package for all ICP-AES and ICP-MS parameters, for every instrument used to generate data in the SDG, as indicated in Exhibit B.

19. GFAA QC Analyses

19.1 Because of the nature of GFAA techniques, the procedures summarized in flowchart pictured in Figure E-3 are required for quantitation. (These procedures do not replace those in Exhibit D of this SOW, but supplement the guidance provided therein). Each step in the flowchart is described below;

[1] Prepare a post-digest analytical spike for every sample except the calibration QC samples (ICV, ICB, CCV,CCB,CRA) and analyze along with the unspiked sample. The required spiking concentrations are listed below. The spiked sample can be prepared by the furnace AAS instrument directly in the furnace tube or manually by the operator. To prepare in an automated fashion using the instrument, consult the operator's manual. To prepare manually, add a known quantity of the analyte to an aliquot of the digested sample and the same quantity of deionized water to another aliquot of the digested sample. The volume of spiking solution must not exceed 10% of the sample aliquot volume.

Post-digest analytical spike concentration (ppb)			
As	Pb	Se	Tl
40	40	40	20

[2] The concentration of analyte in the spiked and unspiked samples must fall within the calibrated range of the furnace AAS instrument. If not, the sample must be diluted, respiked, and reanalyzed (refer to Step 8). If the concentration is within the calibrated range, the analytical spike recovery is calculated by

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

The recovery is interpreted and corresponding action taken, as described in Steps 3-8, with one exception. For the preparation blank, the recovery must be within the window of 85-115%. If the PB analytical spike recovery is out of control, the spiking solution must be verified by respiking and rerunning the PB once. If the PB analytical spike recovery is still out of the control limits, the problem must be corrected, the PB respiked and all analytical samples associated with that blank must be reanalyzed.

[3] If the analytical spike recovery is less than 40%, the sample must be diluted, respiked, and reanalyzed once. If after dilution and reanalysis, the analytical spike recovery is still less than 40%, the result is reported down to the IDL and flagged with an "E" to indicate matrix interference problems (refer to Step 8).

[4] If the analytical spike recovery is within the windows of 85-115%, the results by direct quantification are "acceptable" and are reported down to the IDL. If the recovery is not within the acceptance limits, the analyst has the option of reanalyzing the sample and spike if this is the first analysis.

[5] If the analytical spike recovery is outside of the windows 85-115% and the sample concentration is less than half of the spike concentration, the results are reported down to the IDL and flagged with a "W". The "W" flag indicates that the analytical spike recovery is out-of-control for unspecified reasons (eg, slight matrix problems or poor spiking procedure). Because of the sample concentration, additional effort to resolve the problem is not expected to result in a better number.

[6] If the analytical spike recovery is outside of the windows 85-115% and the sample concentration is greater than half of the spike concentration and greater than 20 times the CRDL, the sample is diluted, respiked, and reanalyzed (refer to Step 8).

[7] If the analytical spike recovery is outside of the windows 85-115% and the sample concentration is greater than half of the spike concentration but less than 20 times the CRDL, the sample is quantified by the method of standard additions (MSA). Samples for MSA analysis are prepared manually by the operator. Alternatively, the MSA aliquots can be prepared in an automated fashion by the furnace AAS instrument if it has the capability. In either case, the following steps must be incorporated into the MSA analysis.

[8] If called for by steps 2, 3, or 6, samples are diluted, respiked, and reanalyzed. Generally, dilutions of 5-10 are acceptable. However, analyst judgement may be used to perform other dilutions. However, the sample must not be diluted so that the analyte is less than the CRDL. If the sample is diluted below the CRDL, it must be reanalyzed using a lower dilution factor, if possible.

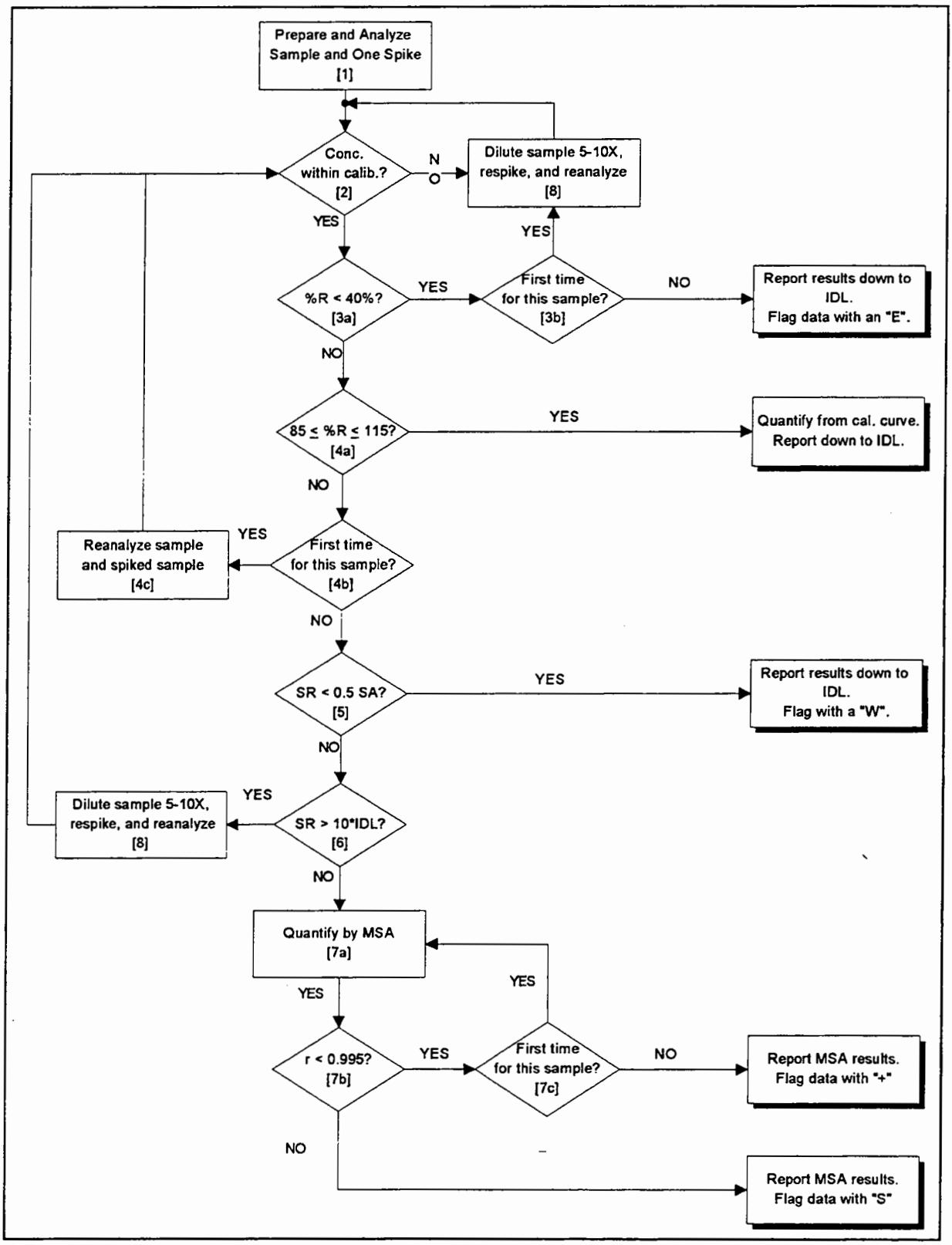

Figure E-3. GFAA QC Flow Diagram

SECTION V - Contract Compliance Screening

1. Purpose

Contract Compliance Screening (CCS) is a contractual way for the Government to inspect analytical data packages for adherence to contract requirements. CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies.

2. Description

CCS is performed by the SMO under the direction of EPA. Using a set of standardized procedures sample data packages submitted by the Contractor are evaluated against the technical and completeness requirements of the contract. Copies of CCS results are mailed to contractor, regional client, and QATS. The Agency may also generate a CCS trend report which summarizes CCS results over a given period of time. The Agency may send the CCS trend report to the lab or discuss it during an on-site laboratory evaluation. Both the individual CCS reports and the trend report will identify any contractor deficiencies. In either case, the Contractor must respond in writing within 14 days of receiving the report(s) or the on-site laboratory evaluation. The response must address the deficiencies and describe the corresponding corrective action(s). An extension for responding of up to 14 days may be requested by the Contractor, but it is the sole decision of the Agency, represented by the TPO or APO to approve or disapprove request. If an extension is requested, the request must include a justification for the delay. Any corrections to a data package must be sent to the regional client, and SMO.

If required by the corrective action, any new or amended SOPs must be submitted as required in Section IV of Exhibit E.

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions:

- reduction in the number of samples sent under this contract,
- suspension of sample shipment to the Contractor,
- data package audit,
- an on-site laboratory evaluation,
- remedial performance evaluation sample, and/or
- contract sanctions, such as a Cure Notice.

SECTION VI - Analytical Standard Requirements

1. Source of Standards

The U.S. Environmental Protection Agency (EPA) does not supply analytical reference standards for direct analytical measurements or for the purpose of traceability. The contract laboratory is required to prepare in-house or obtain commercially the standards necessary to successfully and accurately perform the analyses required in this protocol.

1.1 In-house Preparation of Stock Standard Solutions - Instructions for preparing stock standards from high-purity reagent chemicals are given in Exhibit D. After preparation, it is the responsibility of the contract lab to verify the concentration of the stock standard solutions. Verification can be performed using classical wet chemistry or by analyzing against a certified standard from another source. If a classical technique is used to set the standard concentration, the relative precision of triplicate determinations must be less than 2%. If analysis against a certified standard is used, the measured concentration and theoretical concentration must agree within 2%. The standard cannot be used until an acceptable verification is obtained

1.2 Commercially Obtained Stock Standard Solutions - Stock standard solutions with certified analyte concentrations are readily available from a number of private vendors. These standards can be used without additional verification. However, the Contractor retains responsibility for the quality of the commercially-obtained standards used for analyses under this contract.

2. Documentation

It is the responsibility the contractor to maintain the documentation demonstrating that the standards used for analysis conform to the requirements previously listed. All data, whether produced by the laboratory or supplied by a commercial vendor, must be maintained by the laboratory and may be subject to review during on-site inspection visits. The documentation which relates to the analytical results for EPA data packages must be kept on file by the laboratory for 1 year.

2.1 Stock Standards - A standards logbook must be maintained to document the preparation or receipt of stock standard solutions. For stock standards prepared in-house, the logbook must fully document how and when the standard was prepared and verified. For stock standards obtained commercially, the logbook must include the standard source, date received, date opened, and a copy of the certification (or reference to its location). Stock standard solutions must be clearly labeled with the analyte(s), concentration, dates (preparation or receipt/opened dates), matrix (eg., acid type and concentration), logbook reference, and initials of the preparer.

2.2 Working Standards - The preparation of working standards from the stock standards must be documented in a logbook. It must contain how and when each working standard was prepared (eg., stock standard identification, volume of stock standard, final volume after dilution, diluent, resultant concentration, etc.). The calculations for determining the working standard concentration must be verified by a second person (as indicated by their initials).

3. Stability of Standards

It is the responsibility of the contractor to maintain the quality of their standards over time. Stock standards are generally stable for at

least 6 months when stored properly. The concentration of an analyte in commercially obtained stock standard is certified for a fixed time period and the standard often has an "expiration date". All stock standards older than 6 months or their expiration date must be reverified prior to use.

4. EPA Auditing/Deficiency Reporting/Corrective Action

Upon request by the TPO or APO, the Contractor must submit the standards documentation from the previous year (12 months). Documentation for stock standards or working standards may be requested and must be submitted within 14 days of the request.

The Agency may notify the Contractor of deficiencies in documentation either by letter report or by discussing the deficiencies during an on-site laboratory evaluation. In either case, the Contractor must respond in writing within 14 days of receiving the report or the on-site laboratory evaluation. The response must address the deficiencies and describe the corresponding corrective action(s). An extension for responding of up to 14 days may be requested by the Contractor, but it is the sole decision of the Agency, represented by the TPO or APO to approve or disapprove request. If an extension is requested, the request must include a justification for the delay.

If required by the corrective action, any new or amended SOPs must be submitted as required in Section IV of Exhibit E.

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions:

- reduction in the number of samples sent under this contract,
- suspension of sample shipment to the Contractor,
- data package audit,
- an on-site laboratory evaluation,
- remedial performance evaluation sample, and/or
- contract sanctions, such as a Cure Notice.

SECTION VII - Data Package Audits

1. Purpose of Audits

Data packages are audited by the Agency (or by a contractor under its direction) for program and regional concerns. The audits are used to assess the technical quality of the data, evaluate overall and individual laboratory performance, and provide an in-depth review of data packages with regard to QA/QC criteria. Additionally, this independent monitoring of the data packages (outside of the CCS screening) provides external review of the program QC requirements.

2. Description of Audits

Data packages are periodically selected from recently received cases and evaluated for the technical quality of raw data, QC acceptability, and adherence to contractual requirements. Audits are performed according to established SOPs to ensure uniformity of the process. An audit includes reviewing and comparing form data and raw data, verifying that raw data is complete, checking calculations, and ensuring that all contractual requirements are met. The data package is also compared to the general lab information for contract compliance. For example, the qualifications of the laboratory personnel involved with the case are compared to the contract requirements and the raw data is reviewed to and compared to what is expected from the current SOPs on file.

3. Audit Reports/Corrective Action

Upon completion of the data package audit, the Agency may send a copy of the audit report to the Contractor or may discuss it with the Contractor during an on-site laboratory evaluation. In either case, if deficiencies are noted, the Contractor must respond in writing within 14 days of receiving the report or the on-site laboratory evaluation. The response must address the deficiencies and describe the corresponding corrective action(s). Copies of the response must be submitted to the TPO, APO, and QATS. An extension for responding of up to 14 days may be requested by the Contractor, but it is the sole decision of the Agency, represented by the TPO or APO to approve or disapprove request. If an extension is requested, the request must include a justification for the delay.

If required by the corrective action, any new or amended SOPs must be submitted as required in Section IV of Exhibit E.

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions:

- reduction in the number of samples sent under this contract,
- suspension of sample shipment to the Contractor,
- data package audit,
- an on-site laboratory evaluation,
- remedial performance evaluation sample, and/or
- contract sanctions, such as a Cure Notice.

SECTION VIII - Performance Evaluation Samples

1. Purpose of PE Samples

As a means of measuring Contractor and method performance, Contractors participate in Performance Evaluation (PE) studies conducted by the EPA. As part of a PE study, a contractor analyzes a set of PE samples following the same procedures used for routine samples. Results from PE studies are used by the EPA to verify a Contractor's continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

2. Description of PE Samples

Quarterly, a Contractor will receive PE sample set consisting of up to three water samples. The samples are generally shipped as concentrates and must be diluted prior to use. Instructions are included with the sample sets regarding any dilutions necessary. The laboratory is not informed of the analytes in the PE samples nor their concentrations. After any initial dilution, the PE samples are treated as field samples. They must be prepared and analyzed and the results reported as specified in this SOW, including the contract required turnaround time. All contract-required QC must be met. The PE samples will be scored following an established procedure. Analyte identification and quantification as well as duplicate precision and matrix spike recoveries are critical in calculating a performance score (0 to 100%). The Agency will notify the Contractor of their performance score.

3. Performance Score

A Contractor's performance is evaluated based upon the score received for the PE sample analyses. Depending upon the score, corrective action must be taken.

3.1 Score > 90% - Most or all of the analytes in the PE samples have been acceptably identified and quantified. The Contractor's performance is acceptable. No corrective action is necessary.

3.2 90% > Score ≥ 75 - Some of the analytes in the PE samples have been misidentified or misquantified. The Contractor's performance is acceptable but deficiencies exist. The Contractor must respond in writing within 14 days of receiving the PE scoring report. The response must address the deficiencies and describe the corresponding corrective action(s). Copies of the response must be submitted to the TPO, APO, and QATS. An extension for responding of up to 14 days may be requested by the Contractor, but it is the sole decision of the Agency, represented by the TPO or APO to approve or disapprove request. A request for an extension must include a justification.

If required by the corrective action, any new or amended SOPs must be submitted as required in Section IV of Exhibit E.

3.3 Score < 75% - Many of the analytes in the PE samples have been misidentified or misquantified. The Contractor's performance is unacceptable and major deficiencies exist. The National Program Office considers that the Contractor has not demonstrated the capability of meeting the contract requirements. The Contractor must respond in writing within 14 days of receiving the PE scoring report. The response must address the deficiencies and describe the corresponding corrective action(s). Copies of the response must be submitted to the TPO, APO, and QATS. An extension for responding of up to 14 days may be requested

by the Contractor, but it is the sole decision of the Agency, represented by the TPO or APO to approve or disapprove the request. A request for an extension must include a justification.

If required by the corrective action, any new or amended SOPs must be submitted as required in Section IV of Exhibit E.

Upon receiving an unacceptable score, the Contractor shall be notified by the TPO or APO concerning the consequence for their unacceptable performance. A Contractor may expect, but the Agency is not limited to, the following actions:

- reduction in the number of samples sent under this contract
- suspension of sample shipment to the Contractor
- data package audit
- an on-site laboratory evaluation
- remedial performance evaluation sample
- contract sanctions, such as a Cure Notice

A Contractor's prompt response demonstrating that corrective actions have been taken to ensure the Contractor's capability to meet contract requirements may facilitate continuation of full sample delivery.

4. Corrective Action

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions:

- reduction in the number of samples sent under this contract,
- suspension of sample shipment to the Contractor,
- data package audit,
- an on-site laboratory evaluation,
- remedial performance evaluation sample, and/or
- contract sanctions, such as a Cure Notice.

SECTION IX - On-site Laboratory Evaluations

1. Purpose and Frequency of On-site Laboratory Evaluations

   On-site laboratory evaluations are carried out to monitor the Contractor's ability to meet selected terms and conditions specified in the contract. There are two separate categories of on-site evaluations; Quality Assurance (QA) Evaluations and Evidentiary Audit Evaluations. The frequency of on-site evaluations is dictated by a contract laboratory's performance. The evaluations are performed by the Administrative Project Officer (APO), Technical Project Officer (TPO), or their authorized representative.

2. Description of On-site Laboratory Evaluations

   2.1 QA Laboratory Evaluation - Prior to a QA evaluation, documentation pertaining to performance of the Contractor is integrated into a profile package for discussion during the evaluation. Items that may be included are previous on-site evaluation reports, performance evaluation sample scores, Regional reviews of data, Regional QA materials, data audit reports, CCS reports, and data trend reports. During the on-site evaluation, the audits will evaluate the entire operation used by the Contractor to analyze samples under the contract, from sample receipt to final sample disposition. As a minimum, the following items will be inspected and evaluated:

   - Size and appearance of the facility
   - Quantity, age, availability, scheduled maintenance, and performance of instrumentation
   - Availability, appropriateness, and utilization of the quality assurance plan (QAP) and standard operating procedures (SOPs)
   - Staff qualifications, experience, and personnel training programs
   - Reagents, standards, and sample storage facilities
   - Standard preparation logbooks and raw data
   - Bench sheets and analytical logbook maintenance and review
   - Review of the Contractor's sample analysis/data package inspection/data management procedures

   2.2 Evidentiary Audit Evaluation - Agency auditors conduct on-site laboratory evaluations to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements as stated in Exhibit F. SOPs for sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis), and analytical project file organization and assembly are reviewed for adequacy and completeness. Actual laboratory records are examined to determine the accuracy of the SOPs (i.e., are the SOPs being followed as written). Analytical project files are reviewed to determine the accuracy of the document inventory, completeness of the files, adequacy and accuracy of document numbering system, traceability of sample activity, identification of activity recorded on the documents, and error correction methods.

3. Audit Debriefing/Reporting/Contractor Response

   The QA and evidentiary auditors discuss their findings with the APO and/or TPO, then debrief the Contractor. During the debriefing, the auditors present their findings to the Contractor and make recommendations for any corrective actions necessary. Additionally, a written audit reports that describes deficiencies found during the on-site evaluation may be sent to the Contractor. The Contractor must respond in writing within 14 days of receiving notification of deficiencies, either during the on-site evaluation or in an on-site

evaluation report. The response must address the deficiencies and describe the corresponding corrective action(s). Copies of the response must be submitted to the TPO, APO, and QATS. An extension for responding of up to 14 days may be requested by the Contractor, but it is the sole decision of the Agency, represented by the TPO or APO to approve or disapprove the request. A request for an extension must include a justification.

If required by the corrective action, any new or amended SOPs must be submitted as required in Section IV of Exhibit E.

4. Corrective Action

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions:

- reduction in the number of samples sent under this contract,
- suspension of sample shipment to the Contractor,
- data package audit,
- an on-site laboratory evaluation,
- remedial performance evaluation sample, and/or
- contract sanctions, such as a Cure Notice.

EXHIBIT F - CHAIN-OF-CUSTODY, DOCUMENT CONTROL, AND STANDARD OPERATING PROCEDURES

1. Sample Chain-of-Custody

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 accomplish this, Contractors are required to develop and implement the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures.

1.1 Sample Identification

To assure traceability of the samples while in the Contractor's possession, the Contractor shall have procedures for maintaining identification of samples throughout the laboratory. Each sample and sample preparation container shall be labeled with the U.S. Environmental Protection Agency (EPA) number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the EPA number.

1.2 Chain-of-Custody Procedures

Because of the nature of the data being collected, the custody of EPA samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. The Contractor shall have procedures ensuring that EPA sample custody is maintained and documented. A sample is under custody if:

- It is in your possession, or
- It is in your view after being in your possession, or
- It was in your possession and you locked it up, or
- It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel.)

1.3 Sample Receiving Procedures

1.3.1 The Contractor shall designate a sample custodian responsible for receiving all samples.

1.3.2 The Contractor shall designate a representative to receive samples in the event that the sample custodian is not available.

1.3.3 The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.4 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.5 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:

- Airbills or airbill stickers
- Custody seals
- EPA custody records
- EPA traffic reports or Special Analytical Services (SAS) packing lists

- Sample tags

1.3.6 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records, traffic reports or packing lists, and airbills) accompanying the samples at the time of sample receipt.

1.3.7 The Contractor shall contact the Sample Management Office (SMO) to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).

1.3.8 The Contractor shall record the resolution of discrepancies and problems on Telephone Contact Logs.

1.3.9 The following information shall be recorded on Form DC-1 (See Exhibit B) by the sample custodian or his/her representative as samples are received and inspected:

- Condition of the shipping container
- Presence or absence and condition of custody seals on shipping and/or sample containers
- Custody seal numbers, when present
- Condition of the sample bottles
- Presence or absence of airbills or airbill stickers
- Airbill or airbill sticker numbers
- Presence or absence of EPA custody records
- Presence or absence of EPA traffic reports or SAS packing lists
- Presence or absence of sample tags
- Sample tag identification numbers cross-referenced to the EPA sample numbers
- Verification of agreement or non-agreement of information recorded on shipping documents and sample containers
- Problems or discrepancies

1.4 Sample Tracking Procedures

The Contractor shall maintain records documenting all phases of sample handling from receipt to final analysis.

2. Document Control Procedures

The goal of the laboratory document control program is to assure that all documents for a specified Sample Delivery Group (SDG) will be accounted for when the project is completed. Accountable documents used by contract laboratories shall include, but not be limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored for delivery to EPA or are available upon request from EPA prior to the delivery schedule.

2.1 Preprinted Laboratory Forms and Logbooks

2.1.1 All documents produced by the Contractor which are directly related to the preparation and analysis of EPA samples shall become the property of the EPA and shall be placed in the complete SDG file (CSF). All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a SDG is compiled, all original laboratory forms and copies of all SDG-related logbook entries shall be included in the documentation package.

2.1.2 The Contractor shall identify the activity recorded on all laboratory documents which are directly related to the preparation and analysis of EPA samples.

2.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.4 Logbook entries shall be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed.

2.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one SDG per page.

2.1.6 Pages in both bound and unbound logbooks shall be sequentially numbered.

2.1.7 Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments. Because the laboratory must provide copies of the instrument run logs to EPA, the laboratory may exercise the option of using only laboratory or EPA sample identification numbers in the logs for sample ID rather than government agency or commercial client names to preserve the confidentiality of commercial clients.

2.1.8 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable. All notations shall be recorded in ink. "Zs" shall be placed in unused portions of documents.

2.2 Consistency of Documentation

The Contractor shall assign a document control officer responsible for the organization and assembly of the CSF. All copies of laboratory documents shall be complete and legible.

2.2.1 Original documents which include information relating to more than one SDG shall be filed in the CSF of the lowest SDG number. The copy(s) shall be placed in the other CSF(s) and the Contractor shall record the following information on the copy(s) in red ink:

"COPY ORIGINAL IS FILED IN CSF _____"

The Contractor shall sign and date this addition to the copy(s).

2.2.2 Before releasing analytical results, the document control officer shall assemble and cross-check the information on samples tags, custody records, lab bench sheets, personal and instrument logs, and other relevant deliverables to ensure that data pertaining to each particular

sample or sample delivery group is consistent throughout the CSF.

2.3 Document Numbering and Inventory Procedure

2.3.1 In order to provide document accountability of the completed analysis records, each item in the CSF shall be inventoried and assigned a serialized number as described in Exhibit B).

2.3.2 All documents relevant to each sample delivery group, including logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc. shall be inventoried.

2.3.3 The Document Control Officer (DCO) shall be responsible for ensuring that all documents generated are placed in the CSF for inventory and are delivered to the appropriate EPA region or other receiver as designated by EPA. The DCO shall place the sample tags in plastic bags in the file.

2.3.4 Storage of EPA Files
The Contractor shall maintain EPA laboratory documents in a secure location.

2.4 Shipment of Deliverables
The Contractor shall document shipment of deliverables packages to the recipients. These shipments require custody seals on the containers placed such that they cannot be opened without damaging or breaking the seal. The Contractor shall document which deliverables were sent, to whom, the date, and the method (carrier) used.

A copy of the transmittal letter for the CSF shall be sent to the NEIC/CEAT and the SMO.

3. Specifications for Written Standard Operating Procedures
The Contractor shall have written standard operating procedures (SOPs) for receipt of samples, maintenance of custody, sample identification, sample storage, sample tracking, and assembly of completed data. An SOP is defined as a written narrative stepwise description of laboratory operating procedures including examples of laboratory documents. The SOPs shall accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. These procedures are necessary to ensure that analytical data produced under this contract are acceptable for use in EPA enforcement case preparation and litigation. The Contractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications and shall be used by EPA as the basis for laboratory evidence audits.

3.1 The Contractor shall have written SOPs describing the sample custodian's duties and responsibilities.

3.2 The Contractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:

3.2.1 Presence or absence of EPA chain-of-custody forms

3.2.2 Presence or absence of airbills or airbill stickers

3.2.3 Presence or absence of traffic reports or SAS packing lists

3.2.4 Presence or absence of custody seals on shipping and/or sample containers and their condition

3.2.5 Custody seal numbers, when present

3.2.6 Airbill or airbill sticker numbers

3.2.7 Presence or absence of sample tags

3.2.8 Sample tag ID numbers

3.2.9 Condition of the shipping container

3.2.10 Condition of the sample bottles

3.2.11 Verification of agreement or non-agreement of information on receiving documents and sample containers

3.2.12 Resolution of problems or discrepancies with the SMO

3.2.13 An explanation of any terms used by the laboratory to describe sample condition upon receipt (e.g., good, fine, OK)

3.3 The Contractor shall have written SOPs for maintaining identification of EPA samples throughout the laboratory.

3.3.1 If the Contractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and shall include a description of the document used to cross-reference the unique laboratory identifier to the EPA sample number.

3.3.2 If the Contractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions.

3.4 The Contractor shall have written SOPs describing all storage areas for samples in the laboratory. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.

3.5 The Contractor shall have written SOPs describing the method by which the laboratory maintains samples under custody.

3.6 The Contractor shall have written SOPs describing the method by which the laboratory maintains the security of any areas identified as secure.

3.7 The Contractor shall have written SOPs for tracking the work performed on any particular samples. The tracking SOP shall include:

- A description of the documents used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.
- A description of the documents used to record calibration and QA/QC laboratory work.

- Examples of document formats and laboratory documents used in the sample receipt, sample storage, sample transfer, and sample analyses.

- A narrative step-wise description of how documents are used to track samples.

3.8 The Contractor shall have written SOPs for organization and assembly of all documents relating to each SDG. Documents shall be filed on a SDG-specific basis. The procedures shall ensure that all documents including logbook pages, sample tracking records, chromatographic charts, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the SDG are compiled in one location for submission to EPA. The written SOPs shall include:

- A description of the numbering and inventory method.

- A description of the method used by the laboratory to verify consistency and completeness of the CSF.

- Procedures for the shipment of deliverables packages using custody seals.

4. Handling of Confidential Information

A Contractor conducting work under this contract may receive EPA-designated confidential information from the agency. Confidential information must be handled separately from other documentation developed under this contract. To accomplish this, the following procedures for the handling of confidential information have been established.

4.1 All confidential documents shall be under the supervision of a designated document control officer (DCO).

4.2 Confidential Information
Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the DCO will log these documents into a Confidential Inventory Log. The information will then be available to authorized personnel but only after it has been signed out to that person by the DCO. The documents shall be returned to the locked file at the conclusion of each working day. Confidential information may not be reproduced except upon approval by the EPA Administrative or Technical Project Officer. The DCO will enter all copies into the document control system described above. In addition, this information may not be disposed of except upon approval by the EPA Administrative or Technical Project Officer. The DCO shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record on the disposition in the Confidential Inventory Log.

EXHIBIT G - GLOSSARY OF TERMS

ABSORBANCE A detector measurement of the decrease in incident light after it passes through a sample.

ALIQUOT A measured portion of a field sample taken for analysis.

ANALYSIS DATE/TIME The date and military time (24-h clock) of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE The element or ion of interest.

ANALYTICAL SAMPLE Any solution or media, introduced into an instrument, on which an analysis is performed (excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification, and continuing calibration blank). Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post digestion spike samples, interference check samples (ICS), Contract Required Detection Limit (CRDL) standard for atomic absorption (CRA), CRDL standard for Inductively Coupled Plasma (CRI), laboratory control sample (LCS), preparation blank (PB), and linear range analysis sample (LRS).

ANALYTICAL SPIKE The post digestion spike. The addition of a known amount of standard after digestion.

AUTOZERO Zeroing the instrument at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

AVERAGE INTENSITY The average of two different injections (exposures).

BACKGROUND CORRECTION A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

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 acid or concentration of acids as used in the sample preparation.

CALIBRATION BLANK A volume of ASTM Type I water acidified with the same acid concentrations as is present in the samples.

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 checked by the sampler on the Traffic Report (TR).

CONTINUING CALIBRATION Analytical standard run every 10 analytical samples or every 2 h, whichever is more frequent, to verify the calibration of the analytical system.

CONTRACT REQUIRED DETECTION LIMIT (CRDL) Minimum level of detection acceptable under the contract Statement of Work.

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.

CORRELATION COEFFICIENT A number (r) which indicates the degree of dependence between two variables (e.g., concentration and absorbance). Two variables with a high degree of dependence would produce a value for "r" which approaches ±1.

DAY - unless otherwise specified, day shall mean calendar day.

DIGESTION LOG An official record of the sample preparation (digestion).

DISSOLVED METALS Analyte elements which have not been digested prior to analysis.

DUPLICATE A second aliquot of a sample which has been treated in the same manner as the original sample for the purpose of determining the precision of the method.

FIELD BLANK Any sample submitted from the field identified as a blank.

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) Atomic absorption which utilizes a graphite cell for atomization and excitation.

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)

HYDRIDE MANIFOLD The area in which the acid borhydride solution and/or potassium iodine solution mix before entering the nebulizer.

INDEPENDENT STANDARD A contractor-prepared standard solution that is composed of analytes from a different source than those used in the standards for the initial calibration.

INDUCTIVELY COUPLED PLASMA (ICP) A technique for the simultaneous or sequential multielement 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.

IN-HOUSE At the Contractor's facility.

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.

INSTRUMENT 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.

INSTRUMENT DETECTION LIMIT (IDL) Determined by summing the standard deviations obtained on 3 nonconsecutive days of 7 consecutive measurements of a standard containing the analyte in reagent water at a concentration that is 3-5 times the IDL.

INTERFERENCE CHECK STANDARD (ICS) A solution containing both interfering and analyte elements of known concentrations that can be used to verify background and interelement correction factors.

INTERFERENTS Substances which affect the analysis for the element of interest.

INTERNAL STANDARDS In-house compounds added at a known concentration.

LABORATORY Synonymous with Contractor as used herein.

LABORATORY CONTROL SAMPLE (LCS) A control sample of known composition. Aqueous laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the EPA 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. Also referred to as VTSR (validated time of sample receipt).

LINEAR RANGE, LINEAR DYNAMIC RANGE The concentration range over which an analytical curve remains linear as determined by the analysis of a standard analyzed during an analytical run for which the standard is $\pm$ 5% of the true value.

MATRIX The predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is water. Matrix is not synonymous with phase (liquid or solid).

MATRIX MODIFIER Salts used in GFAA to lessen the effects of chemical interferents, viscosity, and surface tension.

MATRIX SPIKE Aliquot of a sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure Calculated spike recoveries indicate the appropriateness of the method for the matrix.

METHOD OF STANDARD ADDITIONS (MSA) The addition of three increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept, and y-intercept are determined by least-square analysis. The analyte concentration is determined by the absolute value of the x- intercept. Ideally, the spike volume is low relative to the sample volume (approximately 10% of the volume). Standard addition may counteract matrix interferences; it will not counteract spectral interferences. Also referred to as Standard Addition.

PERFORMANCE EVALUATION (PE) SAMPLE A sample of unknown composition provided by EPA for Contractor analysis. Used by EPA to evaluate Contractor performance.

PREPARATION BLANK (reagent blank, method blank) An analytical quality control sample which is composed of distilled, deionized water and reagents. It is carried through the entire analytical procedure (digested and analyzed).

PROTOCOL A compilation of the procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control. Used synonymously with Statement of Work (SOW).

RELATIVE STANDARD DEVIATION (RSD) The standard deviation as a percent of the arithmetic mean.

ROUNDING RULES If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded to 11.44.

- If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded to 11.45.

- If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As

an example, 11.435 is rounded to 11.44, while 11.425 is rounded to 11.42.

- 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 measurements as required by the contract Statement of Work.

SAMPLE DELIVERY GROUP (SDG) A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer samples within a Case, received over a period of up to 7 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, whichever occurs first:

- Case; or
- Each 20 samples within a Case; or
- Each 7-day calendar period during which samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

SAMPLE NUMBER (EPA SAMPLE NUMBER) A unique identification number designated by EPA for each sample. The EPA Sample Number is six digits in length and appears on the sample Traffic Report 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 5. 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.

STOCK SOLUTION A standard solution which can be diluted to derive other standards.

SUSPENDED ELEMENTAL CONCENTRATION The fraction of elements in an untreated sample which is retained by a 0.45 μm membrane filter.

TEN PERCENT 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 Statement of Work.

TOTAL METALS Analyte elements which have been digested prior to analysis.

TRAFFIC REPORT (TR) An EPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which is used for documenting sample condition and receipt by the laboratory.