

EPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

INORGANIC SUPERFUND METHODS

Multi-Media, Multi-Concentration

ISM02.4
October 2016

STATEMENT OF WORK

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INORGANIC ABBREVIATIONS/ACRONYM LIST

ABBREVIATION/ACRONYM	DEFINITION
AA	Atomic Absorption
ASB	Analytical Services Branch
ASB CLP COR	Analytical Services Branch Contract Laboratory Program Contracting Officer's Representative
°C	Degrees Celsius (unit of measurement)
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
CLP	EPA Contract Laboratory Program
CO	Contracting Officer
COC	Chain of Custody
COR	Contracting Officer's Representative
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
CVAA	Cold Vapor Atomic Absorption Spectroscopy
%D	Percent Difference
DF	Dilution Factor
DRD	Data Receipt Date
DTD	Document Type Definition
Dup	Duplicate Sample
EDD	Electronic Data Deliverable
EPA	United States Environmental Protection Agency
EXES	Electronic Data Exchange and Evaluation System
FCC	Federal Communications Commission
FEP	Fluorinated Ethylene Propylene
g	Gram (unit of measurement)
HRS	Hazard Ranking System
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectrometer or Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometer or Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Sample
ICSA	Interference Check Sample Solution A
ICSAB	Interference Check Sample Solution AB
ID	Identifier
IEC	Interelement Correction
ICV	Initial Calibration Verification
IPC	Instrument Performance Check
IR	Infrared
kg	Kilogram (unit of measurement)
L	Liter (unit of measurement)
Lab	Laboratory
LCS	Laboratory Control Sample
LEB	Leachate Extraction Blank
LRD	Laboratory Receipt Date

INORGANIC ABBREVIATIONS/ACRONYM LIST

ABBREVIATION/ACRONYM	DEFINITION
MA	Modified Analysis
MDL	Method Detection Limit
mg	Milligram (unit of measurement)
mL	Milliliter (unit of measurement)
mm	Millimeter (unit of measurement)
MS	Matrix Spike
MSDS	Material Safety Data Sheet
NCS	Non-Client Sample
NERL	National Exposure Research Laboratory
NIST	National Institute of Standards and Technology
nm	Nanometer (unit of measurement)
NSCEP	National Service Center for Environmental Publications
OSHA	Occupational Safety and Health Administration
OSRTI	EPA Office of Superfund Remediation and Technology Innovation
PB	Preparation Blank
PDF	Portable Document Format
PDS	Post-Digestion/Distillation Spike
PE	Performance Evaluation
PRPs	Potentially Responsible Parties
PT	Proficiency Testing
PTFE	Polytetrafluoroethylene
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QATS	Quality Assurance Technical Support
QC	Quality Control
QMP	Quality Management Plan
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
RPD	Relative Percent Difference
%S	Percent Solids
SA	Spike Added
SARA	Superfund Amendments and Reauthorization Act of 1986
SD	Serial Dilution
SD	Standard Deviation
SDG	Sample Delivery Group
SEDD	Staged Electronic Data Deliverable
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SPLP	Synthetic Precipitation Leaching Procedure
SR	Sample Result
SSR	Spiked Sample Result
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
TR	Traffic Report
TR/COC	Traffic Report/Chain of Custody
µg	Microgram (unit of measurement)
UTF-8	Unicode Transformation Format - 8 bit
VTSR	Validated Time of Sample Receipt
W3C	World Wide Web Consortium
XML	eXtensible Markup Language

EXHIBIT A
SUMMARY OF REQUIREMENTS

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Exhibit A - Summary of Requirements

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1.0 PURPOSE

The purpose of this analytical service is to provide analytical data for use by the U.S. Environmental Protection Agency (EPA), in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). Other EPA Program Offices, as well as customers outside the Agency, that have similar analytical data needs also use this service.

2.0 DESCRIPTION OF SERVICE

This Statement of Work (SOW) provides a contractual framework for laboratories to perform analytical services. This framework applies EPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 23 metals and cyanide in aqueous/water and soil/sediment samples, and total metals analysis in wipes. The SOW also includes Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) leachate extraction procedures. The analytical service contract provides the methods to be used and the specific contractual requirements by which the EPA will evaluate the data.

3.0 DATA USES

This analytical service provides data used for a variety of purposes, such as: determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate clean-up actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including site inspections, Hazard Ranking System (HRS) scoring, remedial investigation/feasibility studies, remedial design, treatability studies, and removal actions.

In addition, the Contractor must be aware of the importance of maintaining the integrity of data generated under the contract, since it is used to make major decisions regarding public health and environmental welfare. The data may also be used in litigation against Potentially Responsible Parties (PRPs) in the enforcement of Superfund legislation.

4.0 SUMMARY OF REQUIREMENTS

The SOW is comprised of eight exhibits:

- Exhibit A - Summary of Requirements
- Exhibit B - Reporting and Deliverables Requirements
- Exhibit C - Inorganic Target Analyte List and Contract Required Quantitation Limits
- Exhibit D - Analytical Methods
- Exhibit E - Quality Systems
- Exhibit F - Programmatic Quality Assurance/Quality Control Elements
- Exhibit G - Glossary of Terms
- Exhibit H - Format for Electronic Data Deliverables

Exhibit A - Section 4

4.1 Major Task Areas

For each sample, the Contractor shall perform the tasks described in each section. Specific requirements for each task are detailed in the exhibits referenced.

4.1.1 Sample Receiving, Storage, and Disposal

The Contractor will receive samples from potential hazardous waste sites and shall store and maintain these samples under proper chain of custody (COC) procedures. The Contractor shall follow procedures outlined in Section 5.0 of this Exhibit for proper sample receipt and handling as well as each Exhibit D - Analytical Methods for proper storage and disposal of unused portion of samples. All anomalies and identified issues shall be communicated to the EPA via the CLP Sample Management Office (SMO) Contractor.

4.1.2 Sample Preparation and Analysis

The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high levels of organic and inorganic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the analysis with appropriate caution. It is the Contractor's responsibility to take all necessary measures to ensure laboratory safety.

4.1.2.1 The Contractor shall prepare samples as described in the respective Exhibit D - Analytical Methods for the requested analysis type. Sample preparation methods shall remain consistent for all samples analyzed within a Sample Delivery Group (SDG).

4.1.3 Sample Reporting and Resubmission of Data

4.1.3.1 Required formats for the reporting of data are found in Exhibit B - Reporting and Deliverables Requirements and Exhibit H - Format for Electronic Data Deliverables. The Contractor shall be responsible for completing and submitting analysis data sheets and electronic data as requested in a format specified in this SOW and within the time specified in Exhibit B - Reporting and Deliverables Requirements, Section 1.1.

4.1.3.2 Use of formats other than those approved will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format will be required at no additional cost to the Government.

4.1.4 Quality Assurance/Quality Control

The Contractor shall maintain a Quality Assurance Project Plan (QAPP) with the objective of providing sound analytical chemical measurements. This program shall incorporate the Quality Control (QC) procedures, any necessary corrective action, and all documentation required during data collection, as well as the Quality Assurance (QA) measures performed by management to ensure acceptable data production.

4.1.4.1 The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D - Analytical Methods and F - Programmatic Quality Assurance/Quality Control Elements. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit E - Quality Systems, and shall be reported in accordance with Exhibit B - Reporting and Deliverables Requirements and Exhibit H - Format for Electronic Data Deliverables.

4.1.4.2 Additional QC shall be conducted in the form of the analysis of Performance Evaluation (PE) samples submitted to the laboratory by the EPA. Unacceptable results of all such QC or PE samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to the EPA or rejection of the data for specific analyte(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values as determined by the EPA, as well as meeting the contract requirements for analysis (Exhibit D - Analytical Methods); QA/QC (Exhibit F - Programmatic Quality Assurance/Quality Control Elements); data reporting and other deliverables (Exhibits B - Reporting and Deliverables Requirements and H - Format for Electronic Data Deliverables); and sample custody, sample documentation, and Standard Operating Procedure (SOP) documentation (Exhibit E - Quality Systems). As an alternative to data rejection, the EPA may require reanalysis of noncompliant samples. Reanalysis will be performed by the Contractor at no additional cost to the EPA.

4.1.5 Modified Analysis

The Contractor may be requested by the EPA to perform a Modified Analysis (MA). The modifications may include, but are not limited to: modified preparation or analysis procedures; additional analytes; sample matrices other than those present in the SOW; and/or lower quantitation limits. The requests will be made in writing, prior to sample scheduling. All contract requirements specified in the SOW/Specifications will remain in effect unless specifically modified.

5.0 SAMPLE RECEIPT AND HANDLING

5.1 Chain of Custody

The Contractor shall receive and maintain samples under proper COC procedures. All associated document control and inventory procedures shall be developed and followed. Documentation described herein shall be required to show that all procedures are strictly followed. This documentation shall be reported as the Complete SDG File (CSF) (See Exhibit B - Reporting and Deliverables Requirements). The Contractor shall establish and use appropriate procedures to handle confidential information received from the EPA.

Exhibit A - Section 5

5.2 Sample Scheduling

- 5.2.1 Sample shipments to the Contractor's facility will be scheduled and coordinated by the CLP SMO. The EPA may request analyses that include all or a subset of the Target Analytes listed in Exhibit C - Inorganic Target Analyte List and Contract Required Quantitation Limits. The EPA may also request modified analyses due to the nature of the samples or project requirements. The Contractor shall communicate with SMO personnel as necessary, throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.
- 5.2.2 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 defined 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.

5.3 Sample Shipments

- 5.3.1 Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing of the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier within the Contractor's geographical area. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including weekends.
- 5.3.2 Unless otherwise instructed by the EPA Region or originating sampler, the Contractor shall be required to routinely return sample shipping containers to the appropriate sampling office within 14 calendar days following shipment receipt. This shipment must be done via ground transportation only pending receipt of a valid return authorization, unless specifically instructed to do otherwise. The Contractor will be provided a shipping mechanism by the EPA Region or originating sampler (e.g., field sampler). The Contractor shall ensure that the account numbers provided are used only for the return of Government-owned shipping containers.
- 5.3.2.1 The Contractor shall remove packing and other materials from the shipping containers before each pick-up and shall ensure that the shipping containers are clean. The Contractor can determine from visual inspection whether the shipping container is clean.

5.4 Sample Receipt

- 5.4.1 If insufficient sample amount (less than the required amount) is received to perform the analyses, the Contractor shall notify SMO and proceed with the analysis of the sample at reduced volume. The Contractor shall document this issue in the SDG Narrative.
- 5.4.2 If the Contractor receives broken sample containers, with enough remaining sample to perform sample analysis, but potentially not enough volume to analyze any possible re-extractions/reanalyses, the Contractor shall note the issue in the SDG Narrative, proceed with analysis of the samples, and notify SMO. If re-extraction/reanalyses are necessary, the Contractor shall contact SMO. The Contractor shall document the provided resolution in the SDG Narrative.

- 5.4.3 If the Contractor encounters other problems with samples or related documentation [e.g., mixed media, sample pH, sample documentation and paperwork such as Traffic Report/Chain of Custody (TR/COC) Records not with shipment, sample and TR/COC Record do not correspond], the Contractor shall immediately contact SMO for resolution.
- 5.4.4 Shipping Container Temperature Monitoring
- 5.4.4.1 To monitor the temperature of the sample shipping container more effectively, a sample shipping container temperature indicator bottle may be included with each shipping container shipped. The applicable temperature blank will be clearly labeled.
- 5.4.4.2 When a shipping container temperature indicator bottle is included in the sample shipping container, the Contractor shall use the supplied shipping container temperature indicator bottle to determine the shipping container temperature. The temperature of the sample shipping container shall be measured and recorded immediately upon opening the shipping container, and prior to unpacking the samples or removing the packing material.
- 5.4.4.3 To determine the temperature of the shipping container, the Contractor shall locate the shipping container temperature indicator bottle in the sample shipping container, invert it several times, remove the cap, and insert a calibrated [National Institute of Standards and Technology (NIST)-traceable] thermometer into the shipping container 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 thermometer used shall be capable of measuring and registering the temperature of the shipping container with an accuracy of $\pm 1^{\circ}\text{C}$.
- 5.4.4.4 If a temperature indicator bottle is not present in the shipping container, an alternative means of determining shipping container temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining shipping container temperature. Other devices (e.g., infrared thermometer) which can measure temperature may be used if they can be calibrated to $\pm 1^{\circ}\text{C}$.
- 5.4.4.5 If a temperature indicator bottle is not present in the shipping container, and the temperature of the shipping container is not less than or equal to 6°C , the Contractor shall note the issue, and the method used to determine the temperature, in the SDG Narrative and proceed with analysis of the samples. If the temperature exceeds 10°C and the samples are soil/sediment samples for any analytical method or aqueous/water samples for cyanide analysis, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the EPA for instructions on how to proceed. SMO will in turn notify the Contractor of the EPA's decision. The Contractor shall document the EPA's decision and the EPA Sample Numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.

5.4.4.6 Liquid bearing thermometers such as mercury or alcohol thermometers shall be traceable to NIST calibration and verified at least annually, and whenever the thermometer has been exposed to temperature extremes. The correction factor shall be indicated on the thermometer, and the date the thermometer was calibrated and the calibration factor shall be kept as prescribed in the laboratory's QA documents and be available for inspection. The NIST thermometer shall be recalibrated at least every five years or whenever the thermometer has been exposed to temperature extremes.

Digital thermometers, thermocouples, and other similar electronic temperature measuring devices shall be calibrated at least quarterly. The date the thermometer was calibrated and the calibration factor shall be kept as prescribed in the laboratory's QA documents and be available for inspection.

When an infrared (IR) detection device is used to measure the temperature of samples, the device shall be verified at least every six months using an NIST certified thermometer over the full temperature range that the IR thermometer will be used. This would include ambient (20-30°C), iced (4°C), and frozen (0 to -5°C). Each day of use, a single check of the IR shall be made by measuring the temperature of a bottle of water, that contains a calibrated thermometer, at the temperature of interest. Agreement between the two readings should be within 0.5°C, or the device shall be recalibrated. The daily checks of the IR shall be documented and the records maintained on file.

5.4.5 Recording Sample pH

5.4.5.1 The pH for all aqueous/water samples received by the Contractor shall be measured, using a method capable of demonstrating that proper preservation was performed (e.g., pH test strips, electronic hand-held pen, pH meter), and recorded. The pH shall be determined using a small aliquot of the sample to prevent contamination. Under no circumstances shall a strip or any device be inserted into a sample bottle for the purpose of determining pH.

5.4.5.2 All pens and pH meter electrodes shall be rinsed with reagent water between sample readings.

5.5 Sample Case

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.

5.5.1 A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received; or
- Each 20 samples (excluding PE samples) within a Case; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with receipt of the first sample in the SDG).

- In addition, all samples assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining an SDG.
 - All samples scheduled with the same level of deliverables.
- 5.5.2 Samples may be assigned to SDGs by matrix (i.e., all soil/sediment in one SDG, all aqueous/water in another), at the discretion of the laboratory. If PE samples are received within a Case, they shall be assigned to an SDG containing field samples for that Case. Such assignment shall be made at the time the samples are received and shall not be made retroactively. The SDG may exceed the 20 samples limit since the limitation excludes PE samples.
- 5.5.3 Each sample received by the Contractor will be labeled with an EPA Sample Number and accompanied by a TR/COC Record bearing the Sample Number and descriptive information regarding the sample. The EPA Sample Numbers are continuous, without spaces or hyphens. If the sample numbers do not conform to this requirement, contact SMO. The Contractor shall complete and sign the TR/COC Record, recording the date of sample receipt and sample condition on receipt for each sample container.
- 5.5.3.1 The Contractor shall follow the instructions given on the TR/COC Record in choosing the QC samples, when such information is provided. If no QC sample is designated on the TR/COC Record, the Contractor shall select a sample and notify SMO for EPA Regional acceptance. SMO shall contact the EPA Region for confirmation immediately after notification.
- 5.5.3.2 If the Sampler designated two (or more) samples as QC for the same matrix, and the QC samples are not specifically labeled with the analysis they are to be used for (dissolved metals and total metals), then the Contractor is to contact SMO to report the issue. SMO shall then contact the EPA Region and notify the Contractor of the EPA Regional decision. If the Sampler did not designate QC samples, then the Contractor is to select a sample for QC and to contact SMO to report the issue.
- 5.5.4 The date of delivery of the SDG, or any samples within the SDG, is the date that the last sample in the SDG is received. Validated Time of Sample Receipt (VTSR) is the date of sample receipt at the Contractor's facility, as recorded on the shipper's delivery receipt and sample TR/COC Record.
- 5.5.5 The Contractor shall submit electronic copy(ies) of signed TR/COC Record as Portable Document Format (PDF) file(s) for all samples in an SDG to SMO via the Superfund Analytical Services SMO Portal at <https://epasmoweb.fedcsc.com> within 3 working days following the receipt of the last sample in the SDG. TR/COC Records shall be submitted with their SDG information as specified in Exhibit B - Reporting and Deliverables Requirements.
- 5.5.6 The EPA Case Numbers, SDG Numbers, and EPA Sample Numbers shall be used by the Contractor in identifying samples received under this contract, both verbally and in reports/correspondence.
- 5.5.7 The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

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EXHIBIT B
REPORTING AND DELIVERABLES REQUIREMENTS

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Exhibit B - Reporting and Deliverables Requirements

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1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 Report Deliverable Schedule

The following table identifies the contract reporting and deliverables requirements, and specifies the distribution that is required for each deliverable.

TABLE 1. DELIVERABLE SCHEDULE

Item		No. of Copies ¹	Delivery Schedule	Distribution		
				SMO	Region	QATS
A.	Sample Traffic Reports/Chain of Custody (TR/COC) Records	1	3 working days after receipt of last sample in Sample Delivery Group (SDG).	X		
B. ^{2,3}	Complete SDG File (CSF)	1	XX ⁴ days after Validated Time of Sample Receipt (VTSR) of last sample in SDG.		X	
C. ^{2,5,8}	Copy of CSF and Hardcopy Data in Portable Document Format (PDF) Format	1	XX ⁴ days after VTSR of last sample in SDG.	X		
D. ^{2,6}	Preliminary Results	1	Within 48 hours after receipt of each sample at laboratory, if requested.	X	X	
E. ^{2,8}	Electronic Data Deliverable (EDD)	1	XX ⁴ days after VTSR of last sample in SDG.	X		
F. ²	Proficiency Testing (PT) Audits	1	XX ⁴ days after VTSR of last sample in SDG.	X		
G. ^{7,8}	Determination of Method Detection Limits (MDL) And Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) Interelement Correction (IEC) Factors	1	MDL values in spreadsheet format specified in Appendix A of Exhibit H prior to analysis of field samples, annually thereafter, and after major instrument adjustments to SMO and QATS. MDL and IEC study data prior to analysis of field samples, annually thereafter, and after major instrument adjustments to QATS only. Submission of all deliverables within 7 days of determinations.	X		X

TABLE 1. DELIVERABLE SCHEDULE (CON'T)

Item		No. of Copies ¹	Delivery Schedule	Distribution		
				SMO	Region	QATS
H.	Standard Operating Procedures (SOPs)	1	Submit within 60 days after contract award. Submit the latest version within 7 days of receipt of written request, to recipients as directed. (See Exhibit E, Section 4.0) Submit amended documents within 14 days of amended SOP(s) as directed in Exhibit E, Section 4.4.			X
I.	Quality Assurance Project Plan (QAPP)	1	Submit within XX ⁴ days after contract award. Submit the latest version within 7 days of receipt of written request, to recipients as directed. (See Exhibit E, Section 3.0) Submit amended documents within 14 days of amended QAPP as directed in Exhibit E, Section 3.3.			X
J.	Instrument Electronic Data	Lot	Retain for 3 years after data submission of the reconciled CSF. Submit within 7 days of receipt of written request, to recipients as directed. (See Exhibit F, Section 8.3)	As Directed		X
K.	Digestates	Lot	Retain for total metals (excluding mercury) 180 days after data submission. Submit within 7 days after receipt of written request, to recipients as directed.	As Directed		
L.	Samples	Lot	Retain for 60 days after data submission. Submit within 7 days after receipt of written request, to recipients as directed.	As Directed		

Footnotes:

- ¹ The number of copies specified is the number of copies required to be delivered to each recipient.
- ² **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE.** Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. This includes resubmission of both the hardcopy and electronic deliverable. The date of delivery of the SDG, or any sample within the SDG, is the date that all samples have been delivered. **If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables received after this time will be considered late.**
- ³ CSF will contain the original Sample Data for Level 2a, 2b, and 3 deliverables, plus all of the original documents described in Exhibit B, Section 2.4.
- ⁴ The number of days associated with these elements will be provided in the associated laboratory contract document and will also be provided at the time of sample scheduling by the Sample Management Office (SMO) Contractor.
- ⁵ Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the U.S. Environmental Protection Agency's Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR) and Analytical Services Branch CLP COR (ASB CLP COR). Supplemental data (i.e., logbooks) may be requested in writing from the EPA Regional staff or the ASB CLP COR. All written communication sent by the EPA must include the EPA Regional CLP COR in the distribution list. If the EPA Regional CLP COR has not been included in the distribution list, contact the ASB CLP COR.
- ⁶ If requested at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of Form 1-IN sample analyses and field Quality Control (QC) analyses. The Contractor shall provide the SMO copy via the EPA Electronic Data Exchange and Evaluation System (EXES) at <https://epasmoweb.fedcsc.com> as a PDF file as preliminary results. The PDF file name should be PR_Case Number_SDG Number_Contract Number_Method. Sample TR/COC Records and SDG Cover Page (per Exhibit B, Section 2.7.1) shall be submitted with the Preliminary Results. The designated Regional recipient shall receive the Preliminary Results as a PDF file or in alternative electronic formats (e.g., Microsoft® Word) via email. The Contractor will be notified of the email address and format at the time of sample scheduling.
- NOTE: Preliminary Results Delivery Schedule:
- If a sample requiring Preliminary Results arrives at the laboratory before 5 p.m., the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received at the laboratory after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day.
- ⁷ Results required in each CSF.
- ⁸ The Contractor shall provide SMO the electronic files via EXES at <https://epasmoweb.fedcsc.com>.

Exhibit B - Sections 1-2

1.2 Distribution

The following addresses correspond to the "Distribution" column in Exhibit B, Section 1.1, Table 1 - Deliverable Schedule.

Sample Management Office (SMO)¹:

Delivery instructions shall be provided upon contract award.

EPA Region:

SMO will provide the Contractor with the list of addressees for data delivery for the 10 EPA Regions. SMO will provide the Contractor with updated EPA Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

EPA Regional CLP Contracting Officer's Representative:

SMO will provide the Contractor with the list of addresses for the EPA Regional CLP CORs. SMO will provide the Contractor with updated name/address lists as necessary throughout the period of the contract.

Quality Assurance Technical Support (QATS)²:

Delivery instructions shall be provided upon contract award.

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1 Introduction

The Contractor shall provide reports and other deliverables as specified in Exhibit B, Section 1.1 (for hardcopy) and Exhibit H (for electronic). The required content and form of each deliverable are described in this Exhibit. All reports and documentation **shall be**:

- Legible;
- Clearly labeled and completed in accordance with instructions in this Exhibit;
- Arranged in the order specified in this Exhibit;
- Paginated sequentially according to instructions in this Exhibit; and
- Double-sided.
- Information reported on the forms listed in this Exhibit [excluding the Sample Log-In Sheet (DC-1) and the Complete SDG File (CSF) Inventory Sheet (DC-2)] must be computer-generated.

¹ SMO is a Contractor-operated facility operating under the SMO contract awarded and administered by the EPA.

² QATS is a Contractor-operated facility operating under the QATS contract awarded and administered by the EPA.

- The Contractor shall use EPA Case Numbers, SDG Numbers, and EPA Sample Numbers to identify samples received under this contract, verbally, electronically, and in reports and correspondence. The Contract Number and the Statement of Work (SOW) Number shall be specified in all correspondence. The Modification Analysis Number (MA No.) shall also be included for all Modified Analyses.
- 2.1.1 The Contractor shall submit Staged Electronic Data Deliverable (SEDD) Level 2a, Level 2b, or Level 3 deliverables as specified at the time of scheduling.
- Level 2a deliverables consist of a specified limited subset of the data reporting forms as specified in this Exhibit.
 - Level 2b deliverables include all data reporting forms as specified in this Exhibit.
 - Level 3 deliverables include all data reporting forms and supporting raw data as specified in this Exhibit.
- 2.1.2 Section 3.0 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide the EPA with all required data. Section 4.0 of this Exhibit contains the required Data Reporting Forms in Agency-specified format. Data elements and instructions for electronically reporting data are contained in Exhibit H - Format for Electronic Data Deliverables.
- 2.2 Resubmission of Data
- If submitted documentation does not conform to the above criteria, the Contractor is required to resubmit such documentation with deficiency(ies) corrected, at no additional cost to the EPA.
- 2.2.1 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, through an EPA Regional CLP COR action, or through an EPA Regional data reviewer's request, the data shall be clearly marked as "Additional Data" and shall be sent to both contractual data recipients (SMO and EPA Region) and to the EPA's designated recipient when a written request for a copy of the CSF has been made within 5 business days (3 business days for a 7-day turnaround) of receipt of the request. A cover letter shall be included which describes what data are being delivered, to which EPA Case Number(s) and SDG Number(s) the data pertains, and who requested the data. Corrected data submitted as "Additional Data" at the request of an EPA Regional data reviewer shall only include the affected pages and be accompanied by a revised SDG Narrative (described in Section 2.4.5 of this Exhibit) documenting the reason(s) for the resubmittal.
- 2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data shall be sent to both contractual data recipients (SMO and EPA Region), and to the EPA's designated recipient when a written request for a copy of the CSF has been made, within 6 business days of receipt of the request. In all instances, the Contractor shall include a cover sheet (Laboratory Response to Results of Contract Compliance Screening). Electronic deliverables shall be submitted or resubmitted to SMO only. Revised DC-1 and DC-2 forms shall be resubmitted to SMO and the EPA Region.

2.3 Sample Traffic Report/Chain of Custody Records

2.3.1 Each sample received by the Contractor shall be labeled with an EPA Sample Number and will be accompanied by a TR/COC Record bearing the Sample Number and descriptive information regarding the sample. The Contractor shall complete the TR/COC Record, recording the date of sample receipt, verifying the number of samples, and signing the TR/COC Record.

2.3.1.1 Upon receipt, the Contractor shall sign for the receipt of samples in the COC Record section. The laboratory Sample Custodian or designated recipient opening and verifying the contents of the shipping container shall then verify receipt of all samples identified within the CLP Traffic Report section and sign and date the signature box located in the CLP Traffic Report section. If a non-CLP TR/COC Record is submitted with the samples (e.g., a Regional TR/COC Record), then the Contractor shall: (1) record the receipt date of the samples and sign the TR/COC Record to maintain the chain-of-custody, and (2) the Sample Custodian or designated recipient shall sign and date the TR/COC Record to verify sample information.

NOTE: If the laboratory is requested to transfer samples to another facility, the Contractor shall date and enter the name of the facility to where the samples will be transferred on the CLP TR/COC Record and document in the SDG Narrative.

2.3.1.2 The Contractor shall also enter the SDG Number, Case Number, and the Laboratory Contract Number on the CLP TR/COC Record. The EPA Sample Number of the first sample received in the SDG is the SDG Number. When several samples are received together in the first SDG shipment, the SDG Number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. Under no circumstances should any SDG Number be replicated within a Case. If necessary, select an alternative sample number for the SDG Number. The SDG Number is also reported on all data reporting forms (see Exhibit B, Section 3.0 - Form Instructions).

2.3.2 The Contractor shall submit TR/COC Records in SDG sets (i.e., TR/COC Records for all samples in an SDG), with an SDG Definition Sheet attached. The SDG Definition Sheet shall contain the following items:

- Laboratory Name;
- Contract Number;
- Modified Analysis Number (if applicable);
- Case Number;
- List of the method/analysis for each sample; and
- List of EPA Sample Numbers of all samples in the SDG, identifying the first and last samples received, and their Laboratory Receipt Dates (LRDs).

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

- 2.3.3 EPA Sample Numbers are continuous, without spaces or hyphens. The original Sample TR/COC Record page, with laboratory receipt information and signed with an original Contractor signature, shall be submitted for each sample in the SDG.
- 2.3.4 If samples are received at the laboratory with multi-sample TR/COC Records, all the samples on one multi-sample TR/COC Record may not necessarily be in the same SDG. In this instance, the Contractor must make the appropriate number of photocopies of the TR/COC Record and submit one copy with each SDG Definition Sheet.

2.4 Complete Sample Delivery Group File

The CSF is described in this section. Sections 2.4.7 through 2.4.10 are specific to the individual analytical methods. If analysis by one or more of the analytical methods is not required, then those method sections are not required as a deliverable. Each method section shall include data for analysis of all samples in one SDG, including field samples, calibrations, QC samples, and supporting documentation. The CSF shall be complete before submission. The CSF shall be consecutively paginated (starting with page number one and ending with the number of all pages in the package).

- 2.4.1 The CSF shall contain all original documents where possible. No photocopies of original documents shall be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to the EPA with another Case/SDG. The CSF shall contain all original documents and be numbered according to the specifications in Exhibit B, Sections 3.0 and 4.0; and organized according to Form DC-2.

NOTE: The Contractor shall retain a legible electronic (PDF) or hardcopy of the CSF for 365 days after submission of the reconciled data package to the Government. After this time, the Contractor may dispose of the package.

- 2.4.2 The CSF shall consist of the following original documents:
- Completed SDG Cover Page with signature and date
 - EPA Sample TR/COC Record
 - Completed and signed Sample Log-In Sheet [Form DC-1]
 - Completed and signed Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]
 - SDG Narrative
 - All original shipping documents, including, but not limited to, the following documents:
 - Airbills (if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information);
 - Sample Tags (if present) sealed in plastic bags; and

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- o All original receiving documents, including, but not limited to, other receiving forms or copies of receiving logbooks.

NOTE: All Case-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other Case-specific documents generated after the CSF is sent to the EPA, as well as copies that are altered in any fashion, are also deliverables to the EPA. Send the original to the EPA Region and a copy to SMO. Send to the EPA's designated recipient only upon written request.

2.4.3 For Level 3 deliverables, all original laboratory records of sample transfer, preparation, and analysis, including, but not limited to, the following documents:

- Percent Solids Log;
- Original preparation and analysis forms, or copies of preparation and analysis logbook pages;
- Internal sample and sample digestate and distillate transfer Chain of Custody Records; and
- Performance Evaluation (PE) Instruction forms.

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

- Communication logs;
- Copies of personal logbook pages;
- All handwritten SDG-specific notes; and
- Any other SDG-specific documents not covered by the above.

If the Contractor does submit SDG-specific documents to the EPA after the submission of the CSF, the documents shall be identified with submission codes. For example, if a page or pages were submitted with errors, the corrected pages would be identified with the Case and SDG Number, and the code R#, where the "#" is incremented for any subsequent resubmissions. If a page has been left out of a CSF, it must be submitted with the code A#. If the entire CSF is to be resubmitted, it must be designated with the code RS#. A revised Form DC-2 should be submitted, and the submission codes and locations of the documents in the CSF shall be recorded in the "Other Records" section on the revised Form DC-2.

2.4.5 SDG Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: Laboratory Name, SOW Number, Contract Number, Case Number, SDG Number, Modified Analysis Number (if applicable), and detailed documentation of any QC, sample, shipment, and/or analytical problems encountered in processing the samples reported in the CSF.

2.4.5.1 The Contractor shall list the target analytes for the SDG.

2.4.5.2 The Contractor shall include any technical and administrative problems encountered, and the resolution or corrective actions taken. These problems may include, but are not limited to interference problems encountered during analysis, listing results from raw results less than the negative Contract Required Quantitation Limit (CRQL), and any problems with the analysis of samples.

- 2.4.5.3 Document the alternative temperature technique used, if applicable, to determine shipping container temperature if a temperature indicator bottle is not present in the shipping container.
- 2.4.5.4 The Contractor shall also provide equations for calibration curves with its fit expression (at least one equation or calibration curve per method), to allow the recalculation of sample results from raw instrument output.
- 2.4.5.5 The Contractor shall also include a discussion of any SOW Modified Analyses. This includes attaching a copy of the approved modification form to the SDG Narrative.
- 2.4.5.6 The Contractor shall also identify and explain any differences which exist between the Form(s) 1-IN and supporting documentation provided in the data package and those previously provided as Preliminary Results.
- 2.4.5.7 When submitting corrected data as "Additional Data" at the request of an EPA Regional data reviewer, the Contractor shall include a revised SDG Narrative documenting the reason(s) for the resubmittal.
- 2.4.5.8 The Contractor shall indicate if IEC Factors were applied during the ICP-AES analysis and if background corrections were applied, during the ICP-AES and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) analyses. If background corrections were applied, the Contractor shall indicate if raw data was generated prior to the application of the background corrections.
- 2.4.5.9 The Contractor shall document the use of collision or reaction cells for reducing ICP-MS interferences. The Contractor shall document: the type of cell and cell mode; the gas(es) used; any additional manufacturer-recommended setup or QC applied to establish analytical conditions (e.g., oxide ratios); list the analysis conditions applied to each analyte and internal standard (e.g., mass), along with any changes in the course of the analytical sequence; and any deliberate use of molecular species to avoid isobaric interferences (e.g., $^{75}\text{As}^{16}\text{O}$ at mass 91 to avoid $^{40}\text{Ar}^{35}\text{Cl}$ at mass 75).
- 2.4.5.10 The Contractor shall report the pH value for soil/sediment samples, if the measurement is requested.
- 2.4.6 SDG Cover Page
- Cover Page for the inorganic analyses data shall include: Laboratory Name; Laboratory Code; Contract Number; Case Number; SDG Number; Modified Analysis Number (MA No.) (if appropriate); SOW Number; EPA Sample Numbers in alphanumeric order cross-referenced with Laboratory Sample ID numbers; and Analytical Method.
- 2.4.6.1 The SDG Cover Page shall contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed in the SDG Narrative. Release of the data contained in this hardcopy Complete SDG File and in the electronic data submitted has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature." This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

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2.4.7 ICP-AES Sample Data Forms and Raw Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. All reporting forms shall be arranged in sequential order in increasing alphanumeric EPA Sample Number order, where applicable. The reporting forms shall be followed by the raw data, including sample, calibration, and QC data. This shall be followed by supporting documentation, including but not limited to: Digestion Logs, Standard and Reagent Preparation Logs, Analysis Logs, and Extraction Logs for Toxicity Characteristic Leaching Procedure/Synthetic Precipitation Leaching Procedure (TCLP/SPLP), where applicable.

2.4.7.1 Inorganic Analysis Data Sheet [Form 1-IN]. Tabulated analytical results of the requested analytes shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

2.4.7.2 Quality Control and Calibration Data

The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are not required for Level 2a deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

2.4.7.2.1 Initial and Continuing Calibration Verification [Form 2-IN]. Not required for Level 2a deliverables.

2.4.7.2.2 Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.

2.4.7.2.3 ICP Interference Check Sample [Form 4-IN]. Not required for Level 2a deliverables.

2.4.7.2.4 Matrix Spike Sample Recovery [Form 5A-IN]

2.4.7.2.5 Post-Digestion/Distillation Spike Sample Recovery [Form 5B-IN]

2.4.7.2.6 Duplicates [Form 6-IN]

2.4.7.2.7 Laboratory Control Sample [Form 7-IN]

2.4.7.2.8 ICP-AES and ICP-MS Serial Dilutions [Form 8-IN]

2.4.7.2.9 Method Detection Limit [Form 9-IN]. Not required for Level 2a deliverables.

2.4.7.2.10 ICP-AES Interelement Correction Factors [Form 10A-IN]. Not required for Level 2a deliverables.

2.4.7.2.11 ICP-AES Interelement Correction Factors [Form 10B-IN]. Not required for Level 2a deliverables.

2.4.7.2.12 Analysis Log [Form 12-IN]. Not required for Level 2a deliverables.

2.4.7.2.13 Initial Calibration [Form 15-IN]. Not required for Level 2a deliverables.

2.4.7.2.14 Initial Calibration Summary [Form 16-IN]. Not required for Level 2a deliverables.

2.4.7.3 Raw Data - Only required for Level 3 deliverables.

For each reported value, the Contractor shall include in the CSF all raw data used to obtain that value. This applies to all required Quality Assurance/Quality Control (QA/QC) measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the verification of method and instrument parameters submitted as a part of each CSF. The raw data for all samples shall include not only the results for the requested analyte(s), but also those for all the interferences [Exhibit D - Inductively Coupled Plasma - Atomic Emission Spectroscopy, Table 1 - Interferent and Analyte Concentrations Used for ICP-AES Interference Check Sample (ICS)].

The raw data shall also contain the results of any other element(s) which have been determined to interfere with the requested analytes(s).

- 2.4.7.3.1 Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. For example, if the instrument is applying an interelement correction for a reduced analyte list, the data used to calculate the correction must be present in the raw data. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.
- 2.4.7.3.2 All raw data shall include concentration units.
- 2.4.7.3.3 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.
- 2.4.7.3.4 Raw data shall be labeled with EPA Sample Numbers and appropriate codes, shown in Exhibit B, Table 5 - Codes for Labeling Data, to unequivocally identify:
- Calibration standards;
 - Initial and Continuing Calibration Blanks and Preparation Blanks;
 - Initial and Continuing Calibration Verification standards, Interference Check Samples (ICs), serial dilution samples, and Laboratory Control Samples (LCSs);
 - Diluted and undiluted field samples;
 - Duplicates;
 - Spikes (matrix and post-digestion); and
 - Instrument used.

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- 2.4.7.4 Digestion Logs (only required for the Level 3 deliverables). The digestion logs shall be submitted for each preparation procedure for ICP-AES. These logs shall include: date; sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; sufficient information to unequivocally identify which QC samples (i.e., LCS, Preparation Blank) correspond to each batch digested; comments describing any significant sample changes or reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; indication of pH less than or equal to 2; PE preparation information (e.g., as-received PEs to final digestate); identification of the sample preparer(s) [signature(s)]; and sufficient information to identify the concentrations and volumes of reagents added to the samples.
- 2.4.7.5 Analysis Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic form shall be maintained for all analytical sequences to enable their reconstruction in time. The analysis logs shall record at a minimum: the date and time of analysis of each analysis within the sequence; identification that includes electronic data file identifiers (IDs), Lab Sample IDs or EPA Sample IDs; analyst identification; notation of QC failures and reasons; and sample dilutions.
- 2.4.7.6 Standard and Reagent Preparation Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic format shall be maintained for the preparation of all standards, reagents, and extraction fluids. Standards shall be clearly labeled as to the identity of: the analyte or analytes, the standard ID, concentration, date prepared, expiration date of the solution, special storage requirements if any, and the preparer's signature. Standards and reagents must be traceable. Dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person.
- 2.4.7.7 Extraction Logs for TCLP and SPLP (only required for Level 3 deliverables). Logbooks shall be submitted for any extraction performed by the Contractor. These shall include: the amount of aqueous and solid phases, percent solids determination, sample weight extracted, extraction fluid used, and start and end time of extraction. For TCLP, include log for determination of extraction fluid, including sample weights and the initial and final pH determination.
- 2.4.7.8 Performance Evaluation (PE) Sample Instructions (only required for the Level 3 deliverable). If PE or PT audit samples are provided to the Contractor and analyzed for ICP-AES as part of the SDG, the Contractor shall submit a copy of the instructions that accompanied the sample(s) in the CSF.
- 2.4.8 ICP-MS Sample Data Forms and Raw Data
- Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. All reporting forms shall be arranged in sequential order in increasing alphanumeric EPA Sample Number order, where applicable. The reporting forms shall be followed by the raw data, including sample, calibration, and QC data. This shall be followed by supporting documentation, including but not limited to: Digestion Logs, Standard and Reagent Preparation Logs, and Analysis Logs, where applicable.

2.4.8.1 Inorganic Analysis Data Sheet [Form 1-IN]. Tabulated analytical results of the requested analytes shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

2.4.8.2 Quality Control and Calibration Data

The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are not required for Level 2a deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

2.4.8.2.1 Initial and Continuing Calibration Verification [Form 2-IN]. Not required for Level 2a deliverables.

2.4.8.2.2 Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.

2.4.8.2.3 ICP Interference Check Sample [Form 4-IN]. Not required for Level 2a deliverables.

2.4.8.2.4 Matrix Spike Sample Recovery [Form 5A-IN]

2.4.8.2.5 Post-Digestion/Distillation Spike Sample Recovery [Form 5B-IN]

2.4.8.2.6 Duplicates [Form 6-IN]

2.4.8.2.7 Laboratory Control Sample [Form 7-IN]

2.4.8.2.8 ICP-AES and ICP-MS Serial Dilutions [Form 8-IN]

2.4.8.2.9 Method Detection Limit [Form 9-IN]. Not required for Level 2a deliverables.

2.4.8.2.10 ICP-MS Internal Standard Association [Form 11-IN]. Not required for Level 2a deliverables.

2.4.8.2.11 Analysis Log [Form 12-IN]. Not required for Level 2a deliverables.

2.4.8.2.12 ICP-MS Tune [Form 13-IN]. Not required for Level 2a deliverables.

2.4.8.2.13 ICP-MS Internal Standards Relative Intensity Summary [Form 14-IN]. Not required for Level 2a deliverables.

2.4.8.2.14 Initial Calibration [Form 15-IN]. Not required for Level 2a deliverables.

2.4.8.2.15 Initial Calibration Summary [Form 16-IN]. Not required for Level 2a deliverables.

2.4.8.3 Raw Data - Only required for Level 3 deliverables.

For each reported value, the Contractor shall include in the CSF all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the verification of method and instrument parameters submitted as a part of each CSF. The raw data for all samples shall include not only the results for the requested analyte(s), but also those for all the interferences [Exhibit D - Inductively Coupled Plasma - Mass Spectrometry, Table 1 - Interferent and Analyte Concentrations Used for ICP-MS Interference Check Sample (ICS)].

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The raw data shall also contain the results of any other element(s) or masses which have been determined to interfere with the requested analytes(s).

- 2.4.8.3.1 Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. For example, if the instrument is applying a correction for a reduced analyte list, the data used to calculate the correction must be present in the raw data. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.
- 2.4.8.3.2 All raw data shall include concentration units.
- 2.4.8.3.3 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.
- 2.4.8.3.4 Raw data shall be labeled with EPA Sample Numbers and appropriate codes, shown in Exhibit B, Table 5 - Codes for Labeling Data, to unequivocally identify:
- Calibration standards;
 - Initial and Continuing Calibration Blanks and Preparation Blanks;
 - Initial and Continuing Calibration Verification standards, ICSS, serial dilution samples, and LCSs;
 - Diluted and undiluted field samples;
 - Duplicates;
 - Spikes (matrix and post-digestion); and
 - Instrument used.
- 2.4.8.4 Digestion Logs (only required for the Level 3 deliverables). The digestion logs shall be submitted for each preparation procedure for ICP-MS. These logs shall include: date; sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; sufficient information to unequivocally identify which QC samples (i.e., LCS, Preparation blank) correspond to each batch digested; comments describing any significant sample changes or reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; indication of pH less than or equal to 2; PE preparation information (e.g., as-received PEs to final digestate); identification of the sample preparer(s) [signature(s)]; and sufficient information to identify the concentrations and volumes of reagents added to the samples.

- 2.4.8.5 Analysis Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic form shall be maintained for all analytical sequences to enable their reconstruction in time. The analysis logs shall record at a minimum: the date and time of analysis of each analysis within the sequence; identification that includes electronic data file IDs, Lab Sample IDs or EPA Sample IDs; analyst identification; notation of QC failures and reasons; and sample dilutions.
- 2.4.8.6 Standard and Reagent Preparation Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic format shall be maintained for the preparation of all standards and reagents. Standards shall be clearly labeled as to the identity of: the analyte or analytes, the standard ID, concentration, date prepared, expiration date of the solution, special storage requirements if any, and the preparer's signature. Standards and reagents must be traceable. Dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person.
- 2.4.8.7 Performance Evaluation (PE) Sample Instructions (only required for the Level 3 deliverable). If PE or PT audit samples are provided to the Contractor and analyzed for ICP-MS as part of the SDG, the Contractor shall submit a copy of the instructions that accompanied the sample(s) in the CSF.
- 2.4.9 Mercury Sample Data Forms and Raw Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. All reporting forms shall be arranged in sequential order in increasing alphanumeric EPA Sample Number order, where applicable. The reporting forms shall be followed by the raw data, including sample, calibration, and QC data. This shall be followed by supporting documentation, including but not limited to: Digestion Logs, Standard and Reagent Preparation Logs, Analysis Logs, and Extraction Logs for TCLP/SPLP, where applicable.

 - 2.4.9.1 Inorganic Analysis Data Sheet [Form 1-IN]. Tabulated analytical results for mercury shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.
 - 2.4.9.2 Quality Control and Calibration Data

The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are not required for Level 2a deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

 - 2.4.9.2.1 Initial and Continuing Calibration Verification [Form 2-IN]. Not required for Level 2a deliverables.
 - 2.4.9.2.2 Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.
 - 2.4.9.2.3 Matrix Spike Sample Recovery [Form 5A-IN]
 - 2.4.9.2.4 Duplicates [Form 6-IN]
 - 2.4.9.2.5 Method Detection Limit [Form 9-IN]. Not required for Level 2a deliverables.

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- 2.4.9.2.6 Analysis Log [Form 12-IN]. Not required for Level 2a deliverables.
- 2.4.9.2.7 Initial Calibration [Form 15-IN]. Not required for Level 2a deliverables.
- 2.4.9.2.8 Initial Calibration Summary [Form 16-IN]. Not required for Level 2a deliverables.

2.4.9.3 Raw Data - Only required for Level 3 deliverables.

For each reported value, the Contractor shall include in the CSF all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the verification of method and instrument parameters submitted as a part of each CSF.

2.4.9.3.1 Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.

2.4.9.3.2 All raw data shall include absorbances or concentration units for mercury.

2.4.9.3.3 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.4.9.3.4 Raw data shall be labeled with EPA Sample Numbers and appropriate codes, shown in Exhibit B, Table 5 - Codes for Labeling Data, to unequivocally identify:

- Calibration standards;
- Initial and Continuing Calibration Blanks and Preparation Blanks;
- Initial and Continuing Calibration Verification standards;
- Diluted and undiluted field samples;
- Duplicates;
- Spikes (matrix); and
- Instrument used.

2.4.9.4 Digestion Logs (only required for the Level 3 deliverables). The digestion logs shall be submitted for each preparation procedure for mercury. These logs shall include: date; sample weights and volumes, with initial sample weight/volume clearly indicated; sufficient information to unequivocally identify which Calibration Standards or QC samples [e.g., Initial Calibration Verification (ICV), Preparation Blank] correspond to each batch digested; comments describing any significant sample changes or

reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; indication of pH less than or equal to 2; PE preparation information (e.g., as-received PEs to final digestate); identification of the sample preparer(s) [signature(s)]; and sufficient information to identify the concentrations and volumes of reagents added to the samples.

- 2.4.9.5 Analysis Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic form shall be maintained for all analytical sequences to enable their reconstruction in time. The analysis logs shall record at a minimum: the date and time of analysis of each analysis within the sequence; identification that includes electronic data file IDs, Lab Sample IDs or EPA Sample IDs; analyst identification; notation of QC failures and reasons; and sample dilutions.
- 2.4.9.6 Standard and Reagent Preparation Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic format shall be maintained for the preparation of all standards, reagents, and extraction fluids. Standards shall be clearly labeled as to: the identity of the analyte or analytes, the standard ID, concentration, date prepared, expiration date of the solution, special storage requirements if any, and the preparer's signature. Standards and reagents must be traceable. Dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person.
- 2.4.9.7 Extraction Logs for TCLP and SPLP (only required for Level 3 deliverables). Logbooks shall be submitted for any extraction performed by the Contractor. These shall include: the amount of aqueous and solid phases, percent solids determination, sample weight extracted, extraction fluid used, and start and end time of extraction. For TCLP, include log for determination of extraction fluid, including sample weights and the initial and final pH determination.
- 2.4.9.8 Performance Evaluation (PE) Sample Instructions (only required for the Level 3 deliverable). If PE or PT audit samples are provided to the Contractor and analyzed for mercury as part of the SDG, the Contractor shall submit a copy of the instructions that accompanied the sample(s) in the CSF.
- 2.4.10 Cyanide Sample Data Forms and Raw Data
- Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. All reporting forms shall be arranged in sequential order in increasing alphanumeric EPA Sample Number order, where applicable. The reporting forms shall be followed by the raw data, including sample, calibration, and QC data. This shall be followed by supporting documentation, including but not limited to: Distillation Logs, Standard and Reagent Preparation Logs, Analysis Logs, and Extraction Logs for SPLP, where applicable.
- 2.4.10.1 Inorganic Analysis Data Sheet [Form 1-IN]. Tabulated analytical results for cyanide shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

Exhibit B - Section 2

2.4.10.2 Quality Control and Calibration Data

The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are not required for Level 2a deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

- 2.4.10.2.1 Initial and Continuing Calibration Verification [Form 2-IN]. Not required for Level 2a deliverables.
- 2.4.10.2.2 Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.
- 2.4.10.2.3 Matrix Spike Sample Recovery [Form 5A-IN]
- 2.4.10.2.4 Post-Digestion/Distillation Spike Sample Recovery [Form 5B-IN]
- 2.4.10.2.5 Duplicates [Form 6-IN]
- 2.4.10.2.6 Method Detection Limit [Form 9-IN]. Not required for Level 2a deliverables.
- 2.4.10.2.7 Analysis Log [Form 12-IN]. Not required for Level 2a deliverables.
- 2.4.10.2.8 Initial Calibration [Form 15-IN]. Not required for Level 2a deliverables.
- 2.4.10.2.9 Initial Calibration Summary [Form 16-IN]. Not required for Level 2a deliverables.
- 2.4.10.3 Raw Data - Only required for Level 3 deliverables.

For each reported value, the Contractor shall include in the CSF all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the verification of method and instrument parameters submitted as a part of each CSF.

- 2.4.10.3.1 Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.
- 2.4.10.3.2 All raw data shall include absorbances or concentration units for cyanide.
- 2.4.10.3.3 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.
- 2.4.10.3.4 Raw data shall be labeled with EPA Sample Numbers and appropriate codes, shown in Exhibit B, Table 5 - Codes for Labeling Data, to unequivocally identify:

- Calibration standards;

- Initial and Continuing Calibration Blanks and Preparation Blanks;
- Initial and Continuing Calibration Verification standards;
- Diluted and undiluted field samples;
- Duplicates;
- Spikes (matrix and post-distillation); and
- Instrument used.

- 2.4.10.4 Distillation Logs (only required for the Level 3 deliverables). The distillation logs shall be submitted as appropriate for each preparation procedure for cyanide. These logs shall include: date; sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; sufficient information to unequivocally identify which Calibration Standards and QC samples (e.g., ICV, Preparation Blank) correspond to each batch distilled; comments describing any significant sample changes or reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; indication of pH greater than or equal to 12; PE preparation information (e.g., as-received PEs to final distillate); identification of the sample preparer(s) [signature(s)]; and sufficient information to identify the concentrations and volumes of reagents added to the samples.
- 2.4.10.5 Analysis Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic form shall be maintained for all analytical sequences to enable their reconstruction in time. The analysis logs shall record at a minimum: the date and time of analysis of each analysis within the sequence; electronic data file IDs; Lab Sample IDs or EPA Sample IDs; analyst identification; notation of QC failures and reasons; and sample dilutions.
- 2.4.10.6 Standard and Reagent Preparation Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic format shall be maintained for the preparation of all standards, reagents, and extraction fluids. Standards shall be clearly labeled as to: the identity of the analyte or analytes, the standard ID, concentration, date prepared, expiration date of the solution, special storage requirements if any, and the preparer's signature. Standards and reagents must be traceable. Dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person.
- 2.4.10.7 Extraction Logs for SPLP (only required for Level 3 deliverables). Logbooks shall be submitted for any extraction performed by the Contractor. These shall include: the amount of aqueous and solid phases, percent solids determination, sample weight extracted, extraction fluid used, and start and end time of extraction.
- 2.4.10.8 Performance Evaluation (PE) Sample Instructions (only required for the Level 3 deliverable). If PE or PT audit samples are provided to the Contractor and analyzed for cyanide as part of the SDG, the Contractor shall submit a copy of the instructions that accompanied the sample(s) in the CSF.

Exhibit B - Section 2

2.5 Copy of Complete Sample Delivery Group File

The laboratory shall provide a copy of the CSF and a PDF file to SMO, as specified in Table 1 - Deliverable Schedule, of this Exhibit.

2.6 Electronic Data Deliverables

The Contractor shall provide the required electronic data deliverable as specified in Table 1 - Deliverable Schedule of this Exhibit.

2.6.1 Electronic Data Delivery in Staged Electronic Data Deliverable

The Contractor shall provide an EDD in SEDD format for Levels 2a, 2b, and 3. The EDD shall include analytical data for all samples in the SDG, as specified in Exhibit H - Format for Electronic Data Deliverables.

2.6.2 Portable Document Format of Complete Sample Delivery Group File

The Contractor shall provide a complete copy of the CSF, and any additional or reconciled hardcopy deliverables, in a PDF file via EXES at <https://epasmoweb.fedcsc.com>, and follow the naming convention for the PDF file. The format of the PDF file should be HCD_Case Number_SDG Number_Contract Number_Submission Type.

2.6.2.1 The following identifiers are used based on submission type:

TABLE 2. PDF SUBMISSION IDENTIFIERS

Submission Type	Identifier
First Submission	FS
Replacement Submission (if a complete replacement of the first submission PDF is required)	RS
Reconciliation Submission	R# (The # character represents the number of the reconciliation. For example, the first reconciliation submission would be identified as R1.)
Additional Data Submission	A# (The # character represents the number of the additional data submissions. For example, the first additional data submission would be identified as A1.)

2.6.2.1.1 The PDF file shall be organized in accordance with the directions provided in Exhibit B, Section 2.0 of the SOW.

2.6.2.1.2 Inorganic data shall be bookmarked using a hierarchical bookmark structure (i.e., an overview or "parent" bookmark, and a subordinate or "child" bookmark nested underneath the "parent" bookmark). The required hierarchical structure is shown in Table 3 - Hierarchical Bookmark Structure.

TABLE 3. HIERARCHICAL BOOKMARK STRUCTURE

Group Bookmark	Parent Bookmark	Child Bookmark
SDG Cover Page, Sample TR/COC Records, Form DC-1, Form DC-2, and SDG Narrative		
ICP-AES Data	Sample Data	Inorganic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
	QC Summary	Initial and Continuing Calibration Verification
		Blanks
		ICP Interference Check Sample
		Matrix Spike Sample Recovery
		Post-Digestion Spike Sample Recovery
		Duplicates
		Laboratory Control Sample
		ICP-AES Serial Dilutions
		Method Detection Limits
		ICP-AES Interelement Correction Factors
		Analysis Log
	Initial Calibration	
	Initial Calibration Summary	
	Raw Data	ICP-AES Raw Data
ICP-AES Digestion Logs		
Preparation and Analysis Logbooks		
TCLP/SPLP Logbooks		
PE/PT Instruction Forms		
ICP-MS Data	Sample Data	Inorganic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
	QC Summary	Initial and Continuing Calibration Verification
		Blanks
		ICP Interference Check Sample
		Matrix Spike Sample Recovery
		Post-Digestion Spike Sample Recovery
		Duplicates
		Laboratory Control Sample
		ICP-MS Serial Dilutions
		Method Detection Limits
		ICP-MS Internal Standard Association
		Analysis Log
		ICP-MS Tune
		ICP-MS Internal Standard Relative Intensity Summary
		Initial Calibration
Initial Calibration Summary		

TABLE 3. HIERARCHICAL BOOKMARK STRUCTURE (CON'T)

Group Bookmark	Parent Bookmark	Child Bookmark
ICP-MS Data (Cont'd)	Raw Data	ICP-MS Raw Data
		ICP-MS Digestion Logs
		Preparation and Analysis Logbooks
		PE/PT Instruction Forms
Mercury Data	Sample Data	Inorganic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
	QC Summary	Initial and Continuing Calibration Verification
		Blanks
		Matrix Spike Sample Recovery
		Duplicates
		Method Detection Limits
		Analysis Log
		Initial Calibration
	Initial Calibration Summary	
	Raw Data	Mercury Raw Data
		Mercury Digestion Logs
		Preparation and Analysis Logbooks
		TCLP/SPLP Logbooks
		PE/PT Instruction Forms
Cyanide Data	Sample Data	Inorganic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
	QC Summary	Initial and Continuing Calibration Verification
		Blanks
		Matrix Spike Sample Recovery
		Post-Distillation Spike Sample Recovery
		Duplicates
		Method Detection Limits
		Analysis Log
		Initial Calibration
	Initial Calibration Summary	
	Raw Data	Cyanide Raw Data
		Cyanide Distillation Logs
		Preparation and Analysis Logbooks
		SPLP Logbooks
PE/PT Instruction Forms		
Receiving Documents, Transfer Records, and Miscellaneous	Additional Documents	Percent Solids Log
		Receiving Logbooks
		Internal Sample, Digestate, and Distillate Transfer Chain-of-Custody Records
		Communication Logs

2.7 Preliminary Results

The Form(s) 1-IN data results (including all appropriate qualifiers and flags) shall be submitted for all samples in one SDG of a Case. Sample analysis shall follow all requirements stipulated in Exhibit D. The Contractor shall clearly identify the Preliminary Results by labeling each Form(s) 1-IN as "Preliminary Results" under the form title (i.e., under Inorganic Analysis Data Sheet). The Contractor shall also include a disclaimer on all Form(s) 1-IN stating that the "Data results contained on this Form 1-IN are for screening purposes only, and may not have been validated for CLP criteria." Sample TR/COC Records and SDG Cover Page (per Exhibit B Section 2.7.1) shall be submitted with the Preliminary Results.

- 2.7.1 The Contractor shall submit the SDG Cover Page following the specifications in Exhibit B, Sections 2.4.6 and 3.4.1. The SDG Cover Page shall be clearly labeled to indicate that the data being reported are Preliminary Results. The SDG Cover Page shall contain the following statement, verbatim: "I certify that these Preliminary Results are in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed in the SDG Narrative. Release of the data contained in this hardcopy Data Package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature." This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

2.8 Method Detection Limits and Interelement Correction Factors

The Contractor shall perform and report determination of the MDLs by the method specified in Exhibit D - Analytical Methods for each instrument used under this contract. Results for the verification of method parameters for the current period shall be submitted using Form 9-IN.

The Contractor shall also perform and report ICP-AES IEC factors (including method of determination) and wavelengths used. Results for the verification of method parameters for the current period shall be submitted using Forms 10A-IN and 10B-IN.

The Contractor shall deliver all determined MDLs to SMO and QATS electronically in the format described in Appendix A - Format Characteristics for Method Detection Limit Study Data, of Exhibit H - Format for Electronic Data Deliverables, according to the delivery schedule specified in Table 1 - Deliverable Schedule, of Exhibit B - Reporting and Deliverables Requirements.

Submission of the study data for the determination of method and instrument parameters, to QATS only, shall include the data used to determine the values reported as well as, for the IECs, the standard preparation logs, sample preparation logs, and analysis logs with analytical sequences. The Contractor shall provide MDL and IEC raw data including sample, calibration, and QC data and supporting documentation, including, but not limited to: Digestion/Distillation Logs, Standard and Reagent Preparation Logs, and Analysis Logs, where applicable, to QATS only, according to the delivery schedule specified in Table 1 - Deliverable Schedule, of Exhibit B - Reporting and Deliverables Requirements.

Exhibit B - Section 3

3.0 FORM INSTRUCTIONS

3.1 Introduction

This section contains specific instructions for the completion of all required Inorganic Data Reporting Forms.

3.2 General Information

Values shall be reported on the hardcopy forms according to the respective form instructions in this section.

- 3.2.1 The data reporting forms discussed in Exhibit B, Section 3.4, and presented in Exhibit B, Section 4.0, have been designed in conjunction with the electronic data format specified in Exhibit H - Format for Electronic Data Deliverables. Information entered on these forms shall **not** exceed the size of the field given on the form, including such laboratory-generated items as "Lab Name" and "Lab Sample ID". See Table 4 - Required Forms for Reporting Level, for a listing of required forms by reporting level.

TABLE 4. REQUIRED FORMS FOR REPORTING LEVEL

Level	Required Forms
SEDD 2a	Forms 1, 3, 5, 6, 7, 8
SEDD 2b	Forms 1-16 (all Forms)
SEDD 3	Forms 1-16 (all Forms)

- 3.2.2 All characters which appear on the data reporting forms presented in Section 4.0 shall be reproduced by the Contractor when submitting data, and the format of the forms submitted shall provide exactly the same information as that shown in the contract. No information may be added, deleted, or moved from its specified position. The names of various fields and analytes (i.e., "Lab Code", "Preparation Batch") shall appear as they are listed in Exhibit B - Reporting and Deliverables Requirements, and Exhibit C - Inorganic Target Analyte List and Contract Required Quantitation Limits, of this SOW.

3.2.3 Rounding Rules

For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is greater than or equal to 5, the absolute value of the result is to be rounded up; otherwise the absolute value of the result is rounded down. For example, -0.4365 rounds to -0.44 and -2.3464 rounds to -2.3. Also see "Rounding Rules" in Exhibit G - Glossary of Terms.

- 3.2.3.1 Before evaluating a number for being in control or out of control of a certain limit (other than the CRQL), the number evaluated shall be rounded using the above rounding rules to the significance reported for that limit. For example, the control limit for an ICV is plus or minus 10% of the true value. Then a calculated percent recovery (%R) of 110.46 shall be reported on Form 2-IN as 110, which is within the control limits of 90-110. On the other hand, a calculated %R of 110.50 shall be reported on Form 2-IN as 111, which is not within the 90-110 percent control limits.

3.2.4 Significant Figures

All results shall be transcribed to Inorganic Forms 1-IN through 16-IN from the instrument raw data to two significant figures if the value is less than 10, or three significant figures if the value is greater than or equal to 10 as described in Exhibit B - Reporting and Deliverables Requirements, and Exhibit H - Format for Electronic Data Deliverables. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. The instrument raw data files contain the raw data values. The hardcopy raw data may be a rounded or truncated representation of the instrument raw data.

3.3 Header and General Form Information

Six pieces of information are common to the header section of each data reporting form. These are Lab Name, Contract, Lab Code, Case Number (Case No.), Modified Analysis Number (MA No.), and SDG Number (SDG No.). Except as noted below for MA No., this information shall be entered on every form and shall match on all forms.

- 3.3.1 "Lab Name" shall be the name chosen by the Contractor to identify the laboratory.
- 3.3.2 "Contract" is the number of the EPA contract under which the analyses were performed.
- 3.3.3 "Lab Code" is an alphanumeric abbreviation, assigned by the EPA, to identify the laboratory and aid in data processing. This Lab Code will be assigned by the EPA at the time a contract is awarded and shall not be modified by the Contractor, except at the direction of the EPA Contracting Officer (CO). If a change of name or ownership occurs at the laboratory, the Lab Code will remain the same unless and until the Contractor is directed by the EPA CO to use another EPA-assigned Lab Code.
- 3.3.4 "Case No." is the SMO-assigned Case Number associated with the sample, and reported on the TR/COC Record or sample shipping paperwork.
- 3.3.5 "MA No." is the EPA-assigned number for analyses performed for an analytical method under the Modified Analysis clause in Exhibit A - Summary of Requirements. If samples are to be analyzed under the Modified Analysis clause, the Contractor shall list the modification reference number on all forms. If the analyses have no modified requirements, leave the "MA No." field blank.
- 3.3.6 "SDG No." is the SDG Number.
- 3.3.7 "EPA SAMPLE NO." appears either in the header information of the form or as the left column of a table summarizing data from a number of samples.
 - 3.3.7.1 All samples, leachates, blanks, matrix spikes, post-digestion/distillation spikes, duplicates, and serial dilutions shall be identified with an EPA Sample Number. For samples, an EPA Sample Number is the unique identifying number given on the TR/COC Record or sample shipping records that accompanied that sample. In order to facilitate data assessment, the sample suffixes listed in Exhibit B, Table 5 - Codes for Labeling Data, must be used.

TABLE 5. CODES FOR LABELING DATA^{1,2,3,4}

Sample	Sample Number
Sample in SDG (TCLP/SPLP Leachate included)	XXXXXX
Sample or Laboratory QC Not Part of the SDG	ZZZZZ
Duplicate	XXXXXXD
Matrix Spike	XXXXXXS
Serial Dilution	XXXXXXL
Post-Digestion/Distillation Spike	XXXXXXA
Instrument Calibration Standards	S##
Initial Calibration Verification	ICV
Initial Calibration Blank	ICB
Continuing Calibration Verification	CCV###
Continuing Calibration Blank	CCB###
Interference Check Samples:	
Solution A	ICSA
Solution AB	ICSAB
Laboratory Control Sample	LCS###
Preparation Blank (Aqueous/Water)	PBW###
Preparation Blank (Soil/Sediment)	PBS###
Preparation Blank (Wipe)	PBF###
Leachate Extraction Blank	LEB###
ICP-MS Tune Check	TUNE

Footnotes:

¹ Instrument QC samples must not be reported as ZZZZZZ.

² For samples received under the CLP for inorganic analyses, the sample number will begin with an "M".

³ The suffix that follows the "S" for the standards indicates the sequence number of the standard analysis. Beginning with S01 and continuing to the last standard analyzed.

⁴ Within an analytical method, the three-character suffix (###) shall be unique for each instance of each sample type within an SDG. The Contractor may achieve this by replacing the suffix with one to three alpha-numeric characters.

3.3.7.2 These sample numbers shall be listed on the form in ascending alphanumeric order. Thus, if MA1111 is the lowest (considering both alpha and numeric characters) EPA Sample Number within the SDG, it would be entered in the first EPA Sample Number field. Samples would be listed below it, in ascending sequence - MA1111, MA1111D, MAB124, MAB125, MAC111, etc.

3.3.8 "Matrix" is the matrix of the sample. Enter "Soil" for soil/sediment samples, "Water" for aqueous/water and leachate samples, and "Wipe" for wipes, as appropriate.

- 3.3.9 "Analytical Method" is the method used to analyze the sample. Enter "ICP-AES", "ICP-MS", "CVAA", or "Spectrophotometry", as appropriate.
- 3.3.10 "Run Batch" is the unique identifier of the analytical sequence from the EDD. Report the RunBatch identifier for the Analytical Sequence reported on the form.
- 3.3.11 "Preparation Batch" is the unique identifier of the preparation batch from the EDD. Report the PreparationBatch identifier for the preparation reported on the form.
- 3.3.12 "Preparation Method" is the method used to prepare the samples for analysis. Report the preparation method reported on the form as specified below:
- 200.7: ICP-AES aqueous/water samples
 - 200.8: ICP-MS aqueous/water samples and soil/sediment samples
 - 3050B: ICP-AES soil/sediment samples and ICP-AES wipe samples
 - 7470A: Mercury aqueous/water samples
 - 7471B: Mercury soil/sediment samples
 - Midi-distillation_Aqueous: Cyanide aqueous/water samples
 - Midi-distillation_Soil: Cyanide soil/sediment samples
 - Micro-distillation_Aqueous: Cyanide aqueous/water samples
 - Micro-distillation_Soil: Cyanide soil/sediment samples
- 3.3.13 "Concentration Units" are the units in which the analytical result is reported. Enter " $\mu\text{g/L}$ ", " mg/L ", " mg/kg ", or " μg " as appropriate.
- 3.3.14 "%Solids" is the percent solids of the soil/sediment sample as determined by the procedure in Exhibit D - General Inorganic Analysis.
- 3.3.15 "Instrument ID" is the unique identifier of the instrument with which analysis is performed.
- 3.3.16 "Analyte" is identified in Exhibit C - Inorganic Target Analyte List and Contract Required Quantitation Limits, and must be reported in the order given in Exhibit C.

3.4 Reporting Forms

3.4.1 SDG Cover Page

3.4.1.1 Purpose

This form is used to list all samples analyzed within an SDG and provide certain analytical information and general comments. It is also the document that is signed by the Laboratory Manager or designee to authorize and release all data and deliverables associated with the SDG.

3.4.1.2 Instructions

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

- 3.4.1.2.1 For samples analyzed using this SOW, enter "ISM02.4" for the SOW Number.
- 3.4.1.2.2 Under column "EPA Sample No.", enter each EPA Sample Number.
- 3.4.1.2.3 Under column "Lab Sample ID", enter each Laboratory sample identifier.

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- 3.4.1.2.4 Under column "Analysis Method", enter an "X" under each Analytical Method scheduled for analysis for each EPA Sample Number.
- 3.4.1.2.5 Each SDG Cover Page shall be signed and dated, in original, by the Laboratory Manager or the Manager's designee to authorize the release and verify the contents of all data and deliverables associated with an SDG.
- 3.4.2 Inorganic Analysis Data Sheet [Form 1-IN]
- 3.4.2.1 Purpose
- This form is used to tabulate and report sample analysis results for inorganic target analytes per analytical method (see Exhibit C - Inorganic Target Analyte List and Contract Required Quantitation Limits).
- 3.4.2.2 Instructions
- Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.2.2.1 "Lab Sample ID", enter the Laboratory sample identifier.
- 3.4.2.2.2 "Date Received" is the date (formatted MM/DD/YYYY) of sample receipt at the laboratory, as recorded on the TR/COC Record (i.e., the VTSR).
- 3.4.2.2.3 Under column "CAS No.", enter the Chemical Abstracts Service (CAS) Number for each analyte as listed in Exhibit C - Inorganic Target Analyte List and Contract Required Quantitation Limits.
- 3.4.2.2.4 Under column "Concentration", enter for each analyte, the value of the result if the concentration or mass is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions. If the concentration is less than the adjusted MDL enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions.
- 3.4.2.2.5 Under column "Q", enter result qualifiers as identified below. If additional qualifiers are used, their explicit definitions shall be included in the SDG Narrative.
- 3.4.2.2.5.1 The MDL obtained for a given preparation method, analysis method, and instrument shall be used for the qualification of the results for samples associated with that preparation method, analysis method, and instrument. Serial dilution and post-digestion/distillation spike results shall be qualified using the MDL and CRQL values utilized for the corresponding field sample.
- All three values (i.e., the instrument reading, CRQL, and MDL) shall be converted to the same units prior to determining the appropriate qualifier.
- 3.4.2.2.5.2 Specified entries and their meanings are as follows:
- X: The reported value is estimated due to interferences.
- *: QC analyses are outside control limits.
- D: The reported value is from a dilution.

J: The reported value was less than the CRQL, but greater than or equal to the MDL.

U: The result was less than the MDL. For Hardness, if the results for both Ca and Mg were less than their respective MDLs.

3.4.2.2.6 Under column "Date Analyzed", for each analyte reported, enter the date of the analysis the result is being reported from as MM/DD/YYYY.

3.4.2.2.7 Under column "Time Analyzed", for each analyte reported, enter the time of the analysis the result is being reported from in military time (HHMM).

3.4.2.2.8 In the "Comments" field, note any significant changes that occur during sample preparation (e.g., emulsion formation), any sample-specific comments concerning the analyte results, and any raw instrument results that are less than the negative CRQL (-CRQL). These notes shall also be included the SDG Narrative.

3.4.3 Initial and Continuing Calibration Verification [Form 2-IN]. This form is not required for Level 2a deliverables.

3.4.3.1 Purpose

This form is used to report analyte recoveries from calibration verification solutions.

3.4.3.2 Instructions

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

3.4.3.2.1 "Initial Calibration Verification Source" and the "Continuing Calibration Verification Source" identify the manufacturer and the solution (lot) used.

Use additional Form(s) 2-IN if more calibration verification sources were used.

3.4.3.2.2 Under column "Initial Calibration Verification", enter the following:

3.4.3.2.2.1 "ID", enter the EPA Sample Number of the ICV reported on the form.

3.4.3.2.2.2 Under column "True", enter the expected concentration or true amount of each analyte in the ICV Solution.

3.4.3.2.2.3 Under column "Found", enter the concentration of each analyte measured in the ICV Solution.

3.4.3.2.2.4 Under column "%R", enter the percent Recovery (%R) (to the nearest whole number) calculated using the following equation:

EQ. 1 ICV Percent Recovery

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

WHERE,

Found(ICV) = The found concentration of the analyte in the ICV Solution

True (ICV) = The true amount of the analyte in the ICV Solution

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3.4.3.2.2.5 Under column "%RSD", enter the percent Relative Standard Deviation (%RSD) (to the nearest whole number) of the replicates for ICP-AES and ICP-MS analysis. Leave this column blank for Hg and CN analysis.

3.4.3.2.2.5.1 Calculate the %RSD from all replicate integrations using the following equation:

EQ. 2 Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{SD}{\bar{X}} \times 100$$

WHERE,

SD = Standard deviation of ICV replicates (per analyte) from EQ. 3

\bar{X} = Mean value of the ICV replicates (per analyte) from EQ. 4

3.4.3.2.2.5.2 Equation 3 is the general formula for Standard Deviation (SD) for a statistically small set of values.

EQ. 3 Standard Deviation Calculation

$$SD = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{(n-1)}}$$

WHERE,

X_i = Each individual value used to calculate the mean

\bar{X} = The mean of n values from EQ. 4

n = Total number of values

3.4.3.2.2.5.3 Equation 4 is the general formula for the mean of a set of values (\bar{X}).

EQ. 4 Mean Value Calculation

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n}$$

WHERE,

X_i = Each individual value used to calculate the mean

n = Total number of values

3.4.3.2.3 Under column "Continuing Calibration Verification", enter the following:

3.4.3.2.3.1 "ID", enter the EPA Sample Numbers of the CCVs reported on the form.

3.4.3.2.3.2 Under column "True", enter the expected concentration or true amount of each analyte in the CCV Solution.

3.4.3.2.3.3 Under column "Found", enter the concentration of each analyte measured in the CCV Solution.

- 3.4.3.2.3.4 Under column "%R", enter the percent recovery (to the nearest whole number) calculated using the following equation:

EQ. 5 CCV Percent Recovery

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

WHERE,

Found(CCV) = The found concentration of the analyte in the CCV Solution

True(CCV) = The true amount of the analyte in the CCV Solution

- 3.4.3.2.3.5 Under column "%RSD", enter the %RSD (to the nearest whole number) of the replicates for each CCV for ICP-AES and ICP-MS analysis. Calculate the value using Equation 2 with the CCV replicates. Leave these columns blank for Hg and CN analysis.

- 3.4.3.2.4 The order of reporting ICVs and CCVs for each analyte shall follow the chronological order in which the standards were analyzed. Start with the first Form 2-IN and report from the left to the right, continuing to the following Form(s) 2-IN as appropriate. For example, the first ICV shall be reported on the first Form 2-IN.

In an analytical sequence where three CCVs were analyzed, the first CCV shall be reported in the left CCV column on the first Form 2-IN and the second CCV shall be reported in the right column of the same form. The third CCV shall be reported in the left CCV column of the second Form 2-IN. On the second Form 2-IN, the ICV column and the right CCV column shall be left empty in this example. In the previous example, if a second analytical sequence for an analyte was needed, the ICV of that analytical sequence shall be reported on a third Form 2-IN and the CCVs follow in the same fashion as explained before.

NOTE: In the case where two wavelengths are used for an analyte, all ICV and CCV results of one wavelength from all analyses shall be reported before proceeding to report the results of the second wavelength used.

- 3.4.4 Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.

- 3.4.4.1 Purpose

This form is used to report analyte concentrations found in the Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank, and Leachate Extraction Blank (LEB).

- 3.4.4.2 Instructions

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

- 3.4.4.2.1 "Preparation Blank Matrix", enter appropriate matrix (water, soil, or wipe). No abbreviations or other matrix descriptors may be used.

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- 3.4.4.2.2 "Preparation Blank Concentration Units", enter appropriate concentration units ($\mu\text{g/L}$ for water, mg/L for TCLP leachates, mg/kg for soil, or μg for wipes).
- 3.4.4.2.3 Under column "Initial Calibration Blank", enter the following:
 - 3.4.4.2.3.1 "ID", enter the EPA Sample Number of the ICB reported on the form.
 - 3.4.4.2.3.2 Under "Initial Calibration Blank", enter the concentration of each analyte in the most recent ICB.
 - 3.4.4.2.3.2.1 Enter the concentration or mass (positive or negative) for each analyte, if the absolute value of the concentration or mass is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration or mass is less than the appropriate MDL.
 - 3.4.4.2.3.2.2 Under column "Q", enter "J" if the absolute value of the analyte concentration is less than the CRQL for aqueous/water but greater than or equal to the MDL (in $\mu\text{g/L}$) determined for the default aqueous/water preparation method on that particular instrument.

For prepared calibration blanks (e.g., mercury and cyanide), the CRQL for aqueous/water, and the MDL (in $\mu\text{g/L}$ or converted to $\mu\text{g/L}$) for the preparation method, analysis, and instrument shall be used.

Enter "U" if the absolute value of the analyte in the blank is less than the MDL (in $\mu\text{g/L}$ or converted to $\mu\text{g/L}$) obtained from the default aqueous/water preparation method on that instrument (unprepared blanks) or determined for the preparation method (prepared blanks).
- 3.4.4.2.4 Under column "Continuing Calibration Blank", enter the following:
 - 3.4.4.2.4.1 "ID", enter the EPA Sample Numbers of the CCBs reported on the form.
 - 3.4.4.2.4.2 Under "Continuing Calibration Blank", enter the concentration of each analyte detected in the first required CCB analyzed after the ICB.
 - 3.4.4.2.4.2.1 Enter the concentration or mass (positive or negative) for each analyte, if the absolute value of the concentration or mass is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration or mass is less than the appropriate MDL.
 - 3.4.4.2.4.2.2 Under column "Q", enter any appropriate qualifier, as explained in Section 3.4.4.2.3.2.2.
- 3.4.4.2.5 Under column "Preparation Blank/Leachate Extraction Blank", enter the following:
 - 3.4.4.2.5.1 "ID", enter the EPA Sample Number of the Preparation Blank or LEB reported on the form.
 - 3.4.4.2.5.2 Under "Preparation Blank/Leachate Extraction Blank", enter the concentration of each analyte in the Preparation Blank or LEB.

- 3.4.4.2.5.2.1 Enter the concentration or mass (positive or negative) for each analyte, if the absolute value of the concentration or mass is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration or mass is less than the appropriate MDL.
- 3.4.4.2.5.2.2 Under the column "Q", enter the appropriate qualifier, as explained in Section 3.4.4.2.3.2.2.
- 3.4.4.2.6 The order of reporting ICBs and CCBs for each analyte shall follow the chronological order in which the blanks were analyzed, starting with the first Form 3-IN and reporting from left to right and continuing to additional Form(s) 3-IN. If LEBs are analyzed, they shall be reported on a separate Form 3-IN from any Preparation Blanks.
- NOTE: In the case where two wavelengths are used for an analyte, all ICB, CCB, and Preparation Blank and LEB results of one wavelength from all analyses shall be reported before proceeding to report the results of the second wavelength used.
- 3.4.5 ICP Interference Check Sample [Form 4-IN]. This form is not required for Level 2a deliverables.
- 3.4.5.1 Purpose
This form is used to report ICS results for each ICP-AES or ICP-MS instrument used in SDG analyses.
- 3.4.5.2 Instructions
Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.5.2.1 "ICSA Source" and "ICSB Source", identify the manufacturer and the solution (lot) used. For EPA solutions, include the source name and number (e.g., ICSA-1206) as provided in the accompanying solution instructions.
- 3.4.5.2.2 Under column "True ICSA", enter the expected concentration or true amount of each analyte present in ICSA. Enter "0" for each analyte with no specified true value in ICSA.
- 3.4.5.2.3 Under column "True ICSAB", enter the expected concentration or true amount of each analyte present in ICSAB. Enter "0" for each analyte with no specified true value in ICSAB.
- 3.4.5.2.4 Under column "Found ICSA", enter the measured concentration (positive, negative, or zero) for each analyte and interferent. Enter the concentration of each analyte and interferent for ICP-AES, and of each analyte and interferent for ICP-MS in the initial analysis of ICSA as required in Exhibit D. For ICP-MS, do not enter the interferent elements carbon, chloride, molybdenum, phosphorus, sulfur, and titanium. Report as provided in the instructions accompanying the material.
- 3.4.5.2.5 Under column "Found ICSA %R", enter the value of the percent recovery (to the nearest whole number) calculated for True ICSA greater than zero using the following equation. If "True ICSA" equals zero, leave field blank.

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EQ. 6 ICSA Percent Recovery

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

WHERE,

Found(ICSA) = The found concentration of the analyte in the ICSA Solution

True(ICSA) = The true amount of the analyte in the ICSA Solution

3.4.5.2.6 Under column "Found ICSAB", enter the measured concentration (positive, negative, or zero) for each analyte and interferent. Enter the concentration of each analyte and interferent for ICP-AES, and of each analyte and interferent for ICP-MS in the initial analysis of ICSB as required in Exhibit D. For ICP-MS, do not enter the interferent elements carbon, chloride, molybdenum, phosphorus, sulfur, and titanium. Report as provided in the instructions accompanying the material.

3.4.5.2.7 Under column "Found ICSAB %R", enter the percent recovery (to the nearest whole number) calculated for True ICSAB greater than zero using the following equation. If "True ICSAB" equals zero, leave field blank.

EQ. 7 ICSAB Percent Recovery

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

WHERE,

Found (ICSAB) = The found concentration of the analyte in the ICSAB Solution

True (ICSAB) = The true amount of the analyte in the ICSAB Solution

3.4.5.2.8 If more ICS analyses were required, submit additional Form(s) 4-IN as appropriate.

3.4.5.2.8.1 The order of reporting ICSs for each analyte shall follow the chronological order in which the standards were analyzed, starting with the first Form 4-IN and continuing to the following Form(s) 4-IN as appropriate.

NOTE: In the case where two wavelengths are used for an analyte, all ICSA and ICSAB results of one wavelength from all analyses shall be reported before proceeding to report the results of the second wavelength used.

3.4.6 Matrix Spike Sample Recovery [Form 5A-IN]

3.4.6.1 Purpose

This form is used to report results for the pre-digestion/distillation spike.

3.4.6.2 Instructions

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

- 3.4.6.2.1 Under column "Control Limit %R", enter "75-125" if the sample result is less than or equal to four times the Spike Added (SA) value. If the sample result is greater than four times the SA value, leave this field empty.
- 3.4.6.2.2 Under column "Spiked Sample Result (SSR)", enter the measured value, in appropriate units, for each relevant analyte in the matrix spike sample. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the adjusted MDL.
- 3.4.6.2.3 Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.
- 3.4.6.2.4 Under column "Sample Result (SR)", enter the measured value for each required analyte in the sample (reported in "EPA SAMPLE NO." box) on which the matrix spike was performed. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the adjusted MDL.
- 3.4.6.2.5 Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.
- 3.4.6.2.6 Under column "Spike Added (SA)", enter the expected concentration or true amount of each analyte added to the sample. The same concentration units shall be used for "SSR", "SR", and "SA". If the "SA" concentration is specified in the contract, then the value added and reported shall be the specific concentration, corrected for spiked sample weight and percent solids or spiked sample volume.
- 3.4.6.2.7 Under column "%R", enter the percent recovery (to the nearest whole number) for all spiked analytes calculated using the following equation. The percent recovery shall be reported, whether it is negative, positive, or zero.

EQ. 8 Spike Percent Recovery

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

WHERE,

- SSR = Spiked Sample Result (µg/L or mg/kg)
- SR = Sample Result (original) (µg/L or mg/kg). When the sample concentration is less than the MDL, use SR=0.
- SA = Spike Added Theoretical Result (µg/L or mg/kg)

- 3.4.6.2.8 Under column "Q", enter "*" if the Spike Percent Recovery (%R) is out of the control limits (75-125%) and the Sample Result (SR) is less than or equal to four times the SA.
- 3.4.6.2.9 If different samples were used for spike sample analysis of different analytes, additional Form(s) 5A-IN shall be submitted for each sample as appropriate.
- 3.4.6.2.9.1 In the instance where there is more than one spike sample per matrix, per SDG, if one spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG.

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3.4.7 Post-Digestion/Distillation Spike Sample Recovery [Form 5B-IN]

3.4.7.1 Purpose

This form is used to report results for the post-digestion/distillation spike recovery which is based upon the addition of a known quantity of analyte to an aliquot of the digested or distilled sample.

3.4.7.2 Instructions

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

3.4.7.2.1 Under column "Control Limit %R", enter "75-125" if a Post-Digestion Spike was required for the analyte.

3.4.7.2.2 Under column "Spiked Sample Result (SSR)", enter the measured value for each analyte in the post-digestion/distillation spike sample. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the adjusted MDL.

3.4.7.2.3 Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.7.2.4 Under column "Sample Result (SR)", enter the measured value for the concentration of each analyte in the sample (reported in "EPA SAMPLE NO." box) on which the spike was performed. Enter the value if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the adjusted MDL.

3.4.7.2.5 Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.7.2.6 Under column "Spike Added (SA)", enter the expected concentration or true amount of each analyte added to the sample. If the "SA" concentration is specified in the contract, the value added and reported shall be that specific concentration.

3.4.7.2.7 Under column "%R", enter the percent recovery (to the nearest whole number) for all spiked analytes using the following equation. Percent recovery shall be reported, whether it is negative, positive, or zero.

EQ. 9 Post-Digestion/Distillation Spike Percent Recovery

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

WHERE,

SSR = Spiked Sample Result ($\mu\text{g/L}$ or mg/kg)

SR = Sample Result (original) ($\mu\text{g/L}$ or mg/kg). When the sample concentration is less than the MDL, use $SR=0$.

SA = Spike Added Theoretical Result ($\mu\text{g/L}$ or mg/kg)

3.4.7.2.8 If different samples were used for spike sample analysis of different analytes, additional Form(s) 5B-IN shall be submitted for each sample as appropriate.

- 3.4.7.2.9 Under column "Q", enter "*" if the Spike %R is out of the control limits (75-125%).
- 3.4.8 Duplicates [Form 6-IN]
- 3.4.8.1 Purpose
- The duplicates form is used to report results of duplicate analyses. Duplicate analyses are required for all analyte results.
- 3.4.8.2 Instructions
- Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.8.2.1 Under column "Control Limit", enter the CRQL (corrected for the original sample weight and percent solids, if necessary) for the analyte if either the sample or duplicate value was less than 5 times the CRQL. If the sample and duplicate values were greater than or equal to 5 times the CRQL, or if the sample and duplicate values were less than the CRQL, leave the field empty.
- 3.4.8.2.2 Under column "Sample (S)", enter the measured value for the concentration of each analyte in the sample (reported in "EPA SAMPLE NO." box) on which a duplicate analysis was performed. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the adjusted MDL.
- 3.4.8.2.3 Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.
- 3.4.8.2.4 Under column "Duplicate (D)", enter the measured value for each analyte in the duplicate sample. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the adjusted MDL.
- 3.4.8.2.5 Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.
- 3.4.8.2.6 For soil/sediment samples, the concentration of the original sample shall be computed using the weight and percent solids of the original sample. The concentration of the duplicate sample shall be computed using the weight of the duplicate sample, but the percent solids of the original sample.
- 3.4.8.2.7 Under column "RPD", enter the absolute value (to the nearest whole number) of the Relative Percent Difference (RPD) for all analytes detected above the CRQL in either the sample or the duplicate, calculated using the following equation:

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EQ. 10 Duplicate Sample Relative Percent Difference

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

WHERE,

S = Sample result

D = Duplicate result

If the analyte concentration is less than the MDL in either "S" or "D", a value of zero shall be substituted for "S" or "D". If the analyte concentration is less than the CRQL in both "S" and "D", leave the "RPD" field empty.

3.4.8.2.8 Under column "Q", enter "*" if the duplicate analysis for the analyte is outside control limits. If both sample and duplicate values are greater than or equal to 5 times the CRQL, then the RPD must be less than or equal to 20% to be in control. If either the sample or duplicate value is less than 5 times the CRQL, then the absolute difference between the sample and duplicate values shall be less than the CRQL to be in control. If both values are below the CRQL, then no control limit is applicable.

3.4.9 Laboratory Control Sample [Form 7-IN]

3.4.9.1 Purpose

This form is used to report results for the aqueous/water, soil/sediment, and wipe LCSs.

3.4.9.2 Instructions

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

3.4.9.2.1 Under column "True", enter the value of the expected concentration or true amount of each analyte in the LCS.

3.4.9.2.2 Under column "Found", enter the concentration of each analyte found in the LCS.

3.4.9.2.3 Under column "%R", enter the percent recovery (to the nearest whole number) calculated using the following equation:

EQ. 11 LCS Percent Recovery

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

WHERE,

Found (LCS) = The found concentration at each analyte in the LCS. If the analyte concentration is less than the MDL, a value of zero shall be substituted for the Found LCS.

True (LCS) = The true amount of each analyte in the LCS

3.4.9.2.4 Submit additional Form(s) 7-IN as appropriate if more than one LCS was required.

3.4.10 ICP-AES and ICP-MS Serial Dilutions [Form 8-IN]

3.4.10.1 Purpose

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

3.4.10.2 Instructions

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

3.4.10.2.1 Under column "Initial Sample Result (I)", enter the measured value, corrected for any dilutions, for each analyte. Enter the value if the concentration is greater than or equal to the adjusted MDL; or enter the adjusted CRQL if the concentration is less than the adjusted MDL.

3.4.10.2.2 Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.10.2.3 Under column "Serial Dilution Result (S)", enter the measured value for each analyte in the diluted sample. Enter the value if the concentration is greater than or equal to the adjusted MDL; or enter the adjusted CRQL if the concentration is less than the adjusted MDL.

3.4.10.2.4 Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.10.2.5 Under column "% Difference", enter the absolute value (to the nearest whole number) of the percent difference, between the original sample and the diluted sample (adjusted for dilution) using the following equation:

EQ. 12 Serial Dilution Percent Difference

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

WHERE,

I = Initial sample result. If the analyte concentration is less than the MDL concentration, leave the "% Difference" field empty.

S = Serial dilution result. If the analyte concentration is less than the MDL, a value of zero shall be substituted for "S".

3.4.10.2.6 Under column "Q", enter "*" if the percent difference is greater than 10% and the original sample concentration (reported on Form 1-IN) is greater than 50 times the adjusted MDL.

3.4.11 Method Detection Limit [Form 9-IN]

3.4.11.1 Purpose

This form documents the MDL for each preparation method and instrument that the Contractor used to obtain data for the SDG. Only the methods, instruments, and wavelengths used to generate data for the SDG shall be included. A copy of the MDLs reported on Form(s) 9-IN shall be included with each CSF.

3.4.11.2 Instructions

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

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- 3.4.11.2.1 Under column "Wavelength/Mass", enter the wavelength in nanometers (nm) or the mass in atomic mass units (u) for each analyte for which an MDL has been established. If more than one wavelength or mass is used for an analyte, use additional Form(s) 9-IN as appropriate to report the MDLs.
- 3.4.11.2.2 Under column "MDL", enter the MDL as determined by the Contractor for each analyte analyzed by the instrument for which the ID is listed on this form.
- When calculating MDL values, always round up to the appropriate significant figure (e.g., 14.81 rounds to 14.9 and 146.6 rounds to 147). This deviation from the rounding rule is necessary to prevent the reporting of detected values for results that fall in the noise region of the calibration curve.
- NOTE: Zeroes used to set the decimal point in a number less than one are not significant, but all trailing zeroes are significant.
- For example, a calculated MDL value of 0.074 µg/L will be reported as 0.074 and a calculated MDL value of 0.1 or 0.08 will be reported as 0.10 and 0.080, respectively.
- 3.4.11.2.3 The MDLs for Hardness, TCLP, or SPLP are not required to be reported.
- 3.4.11.2.4 Under column "Date Analyzed", enter the date analyzed (formatted as MM/DD/YYYY) for the analyte. Note that the date shall not exceed the analysis dates in the CSF or precede them by more than one year.
- 3.4.12 ICP-AES Interelement Correction Factors [Form 10A-IN]. This form is not required for Level 2a deliverables.
- 3.4.12.1 Purpose
- This form documents for each ICP-AES instrument the IEC factors applied by the Contractor to obtain data for the SDG. A copy of the results of the IEC factors shall be included with each CSF on Form 10A-IN and Form 10B-IN as appropriate.
- 3.4.12.2 Instructions
- Complete the header information according to instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.12.2.1 "Date", enter the date (formatted as MM/DD/YYYY) on which these correction factors were determined for use.
- 3.4.12.2.2 Under column "Wavelength", enter the wavelength in nm used for each ICP-AES analyte. If more than one wavelength is used, submit additional Form(s) 10A-IN or Form(s) 10B-IN as appropriate.
- 3.4.12.2.3 Under columns "Al", "Ca", "Fe", and "Mg", enter the correction factor (negative, positive, or zero) for each ICP-AES analyte to the number of decimal places stored by the instrument and used for correcting the analyte results. Correction factors for one additional analyte shall be reported using the empty column and list the analyte's chemical symbol in the blank two-space header field provided for that column.

3.4.12.2.4 If corrections are not applied for an analyte, a zero [0] shall be entered for that analyte to indicate that the corrections were determined to be zero. Correction factors for more than one additional analyte shall be reported using Form 10B-IN.

NOTE: Correction factors for Al, Ca, Fe, and Mg are all required and are to be listed first (as they appear on Form 10A-IN).

3.4.13 ICP-AES Interelement Correction Factors [Form 10B-IN]. This form is not required for Level 2a deliverables.

3.4.13.1 Purpose

This form is used if correction factors for analytes other than Al, Ca, Fe, Mg, and one more analyte of the Contractor's choice were applied to the analytes analyzed by ICP-AES.

3.4.13.2 Instructions

Complete this form following the instructions for Form 10A-IN (see Exhibit B, Section 3.4.12) by listing the chemical symbol for additional analytes in the heading of the empty columns in the two-space fields provided.

3.4.13.2.1 Columns of correction factors for additional analytes shall be entered left to right starting on Form 10A-IN and proceeding to Form 10B-IN, according to the alphabetical order of their chemical symbols.

3.4.14 ICP-MS Internal Standard Association [Form 11-IN]. This form is not required for Level 2a deliverables.

3.4.14.1 Purpose

This form is used to report the associated internal standards for each target analyte for each ICP-MS instrument used in analysis.

3.4.14.2 Instructions

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

3.4.14.2.1 "Date", enter the date (formatted as MM/DD/YYYY) on which the ICP-MS tune was performed. This date shall not exceed the dates of analysis by ICP-MS in the CSF.

3.4.14.2.2 Under column "Assoc. Internal Standard 1", enter the chemical symbol of the internal standard associated with each target analyte in the analytical sequence.

3.4.14.2.3 Under column "Assoc. Internal Standard 2", if a second internal standard is used for the analyte, then enter the chemical symbol of the second internal standard associated with each target analyte in the analytical sequence. Otherwise leave blank.

3.4.15 Analysis Log [Form 12-IN]. This form is not required for Level 2a deliverables.

3.4.15.1 Purpose

This form is used to report the analytical sequence.

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- 3.4.15.1.1 An analytical sequence is defined as the totality of analyses performed by an instrument throughout the sequence initiated by, and including, the first SOW-required calibration standard or tune standard, and terminated by, and including, the CCV and CCB following the last SOW-required analytical sample.
- 3.4.15.1.2 All field samples and all QC analyses (including tunes, calibration standards, ICVs, CCVs, ICBs, CCBs, ICSSs, LCSSs, Preparation Blanks, LEBs, PE samples, duplicates, serial dilutions, matrix spikes, and post-digestion/distillation spikes) associated with the SDG shall be reported on Form 12-IN. The analytical sequence shall be continuous and inclusive of all analyses performed on the particular instrument during the analytical sequence.
- 3.4.15.1.3 Submit one Form 12-IN per analytical sequence. If more analyses were performed in the analytical sequence than will fit on one form, submit additional Form(s) 12-IN as appropriate.
- 3.4.15.1.4 The Analysis Logs shall be ordered chronologically. Each analytical sequence shall start on a separate Form 12-IN. Therefore, an instrument calibration or tune shall be the first entry on the form for each new analytical sequence. In addition, the analytical sequence is considered to have ended if it is interrupted for any reason, including termination for failing QC parameters.
- 3.4.15.2 Instructions
- Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.15.2.1 "Start Date:", enter the date (formatted as MM/DD/YYYY) on which the analytical sequence was started.
- 3.4.15.2.2 "End Date:", enter the date (formatted as MM/DD/YYYY) on which the analytical sequence was ended.
- 3.4.15.2.3 Under column "EPA Sample No.", enter the EPA Sample Number of each analysis, including all QC operations applicable to the SDG (formatted according to Exhibit B, Table 5 - Codes for Labeling Data). All EPA Sample Numbers shall be listed in increasing chronological (date and time) order of analysis, continuing to the next Form 12-IN for the analytical sequence, if applicable. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical sequence, shall be reported. Those analyses shall be identified with the EPA Sample Number of "ZZZZZZ".
- 3.4.15.2.4 Under column "D/F", enter the dilution factor by which the final digestate or distillate needed to be diluted for each analysis to be performed. The dilution factor does not include the dilution inherent in the preparation as specified by the preparation procedures in Exhibit D. The dilution factor is required for all entries on Form 12-IN.
- NOTE: For a particular sample, a dilution factor of "1.0" shall be entered if the digestate or distillate was analyzed without adding any further volume of dilutant.

3.4.15.2.5 For EPA-supplied solutions such as ICVs and ICSSs, a dilution factor shall be entered if the supplied solution had to be diluted to a dilution different from that specified by the instructions provided with the solution. The dilution factor reported in such a case shall be that which would make the reported true values on the appropriate form for the solution equal to those that were supplied with the solution by the EPA. For instance, ICV-2(0887) has a true value of 104.0 µg/L at a 20-fold dilution. If the solution is prepared at a 40-fold dilution, a dilution factor of "2.0" shall be entered on Form 13-IN and the uncorrected instrument reading is compared to a true value of 52 µg/L. In this example, Form 2-IN will have a true value of 104.0 regardless of the dilution used. The found value for the ICV shall be corrected for the dilution listed on Form 12-IN using the following equation:

EQ. 13 ICV/CCV Correction for Dilution

$$\text{Found value on Form 2-IN} = \text{Instrument readout } (\mu\text{g/L}) \times \text{D/F}$$

WHERE,

D/F = Dilution Factor

3.4.15.2.6 Under column "Time", enter the time (in military format - HHMM) that each analysis was performed.

3.4.15.2.7 Under column "Analytes", enter the chemical symbol for each analyte reported (target and non-target) from the analytical sequence in alphabetical order by name. The Contractor is not required to report analytes (target or interferent) that are not analyzed in that analytical sequence. Enter "X" in the column of the designated analyte to indicate that the analyte value was used from the reported analysis to report data in the SDG. Leave the box empty if that analysis was not used to report the particular analyte.

3.4.15.2.7.1 Entering "X" appropriately is very important. The "X" is used to link the samples with their related QC. It also links the dilution factor with the appropriate result reported on Forms 1-IN - 8-IN. For each analyte result reported on any of the Forms 1-IN - 8-IN, there shall be one, and only one, properly identified entry on Form 12-IN for which an "X" is entered in the column for that analyte.

3.4.15.2.7.2 If, on Form 12-IN, an "X" is entered in the column for an analyte for a field sample associated with a dilution factor greater than 1.0, flag the data for that analyte with a "D" on the appropriate Form 1-IN.

3.4.16 ICP-MS Tune [Form 13-IN]. This form is not required for Level 2a deliverables.

3.4.16.1 Purpose

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

3.4.16.2 Instructions

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

Exhibit B - Section 3

- 3.4.16.2.1 "Date:", enter the date (formatted as MM/DD/YYYY) the ICP-MS tune was performed. This date shall not exceed the dates of analysis by ICP-MS in the CSF.
- 3.4.16.2.2 Under column "Avg. Measured Mass (u)", enter the average mass calculated from the five or more tune integrations (in atomic mass units) measured for each isotope to one decimal point.
- 3.4.16.2.3 Under column "Avg. Peak Width (u)", enter the average peak width calculated from the analysis (in atomic mass units) at the percent of peak height recommended by the instrument manufacturer for each isotope to one decimal point.
- 3.4.16.2.4 Under column "%Height", enter the percent of peak height at which the Average Peak Width was measured to the nearest whole number.
- 3.4.16.2.5 Under column "%RSD", enter the percent Relative Standard Deviation of the absolute signals (intensities) for each isotope calculated from the five or more tune integrations using the following equation:

EQ. 14 Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{SD}{\bar{X}} \times 100$$

WHERE,

- SD = Standard deviation of Tune replicates (per isotope) from EQ. 3
- \bar{X} = Mean value of the Tune replicates (per isotope) from EQ. 4

- 3.4.17 ICP-MS Internal Standards Relative Intensity Summary [Form 14-IN]. This form is not required for Level 2a deliverables.

3.4.17.1 Purpose

This form is used to report the relative internal standard intensity levels during an ICP-MS analytical sequence. The relative intensity of each of the internal standards in all analyses performed by ICP-MS must be reported on the form.

3.4.17.2 Instructions

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

- 3.4.17.2.1 "Start Date:", enter the date (formatted as MM/DD/YYYY) on which the analytical sequence was started.
- 3.4.17.2.2 For "End Date:", enter the date (formatted as MM/DD/YYYY) on which the analytical sequence was ended.
- 3.4.17.2.3 Under column "Time", enter the time (in military format - HHMM) that each analysis was performed.
- 3.4.17.2.4 Under column "Internal Standards %RI For:", enter the chemical symbol and elemental expression number of the internal standard in the "Element" header field provided to indicate the internal standard and elemental expression for which the Relative Intensity (RI) of the internal standards will be calculated in that column.

3.4.17.2.4.1 In the "Element" column, enter the internal standard relative intensity (to the nearest whole number) of the internal standard for each sample analysis listed on the form (excluding samples identified as "ZZZZZZ"). The internal standard percent relative intensity (%RI) is calculated using the following equation:

EQ. 15 Internal Standard Percent Relative Intensity

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

WHERE,

I_o = The intensity of the internal standard in the blank calibration standard

I_n = The intensity of the internal standard in the EPA Sample Number

3.4.17.2.5 Under "Q" column to the right of each "Element" column, enter an "*" if the %RI for a field sample, PE, duplicate, or spike is less than 60 or greater than 125; otherwise leave the field empty.

3.4.17.2.6 Columns of internal standard RI must be entered left to right, starting with the internal standards of the lower mass on the first Form 14-IN and proceeding to the following Form 14-IN as appropriate. All Forms 14-IN for the lowest numeric instrument must be reported in ascending order by the Start Date before proceeding to the next Form 14-IN.

3.4.17.3 All field samples and all QC samples (including calibration standards, ICVs, CCVs, ICBs, CCBs, ICSs, LCS, Preparation Blanks, LEBs, serial dilutions, duplicates, PE samples, and spikes) associated with the SDG must be reported on Form 14-IN. The analytical sequence must be continuous and inclusive of all analyses performed on the particular instrument during the analytical sequence.

3.4.17.4 Submit one Form 14-IN per analytical sequence. If more analyses were performed in the analytical sequence, than will fit on one form, submit additional Form(s) 14-IN as appropriate. Each new analytical sequence must be started on the first line of Form 14-IN.

3.4.17.5 If more than one ICP-MS instrument or analytical sequence is used, submit additional Form(s) 14-IN as appropriate. All analytical sequences for the lowest alphanumeric instrument must be reported in ascending order before proceeding to the analytical sequences for the next highest instrument.

3.4.18 Initial Calibration [Form 15-IN]. This form is not required for Level 2a deliverables.

3.4.18.1 Purpose

This form is used to report instrument response and concentration data for each standard in the initial calibration of an instrument.

3.4.18.2 Instructions

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

Exhibit B - Section 3

- 3.4.18.2.1 "Start Date:", enter the date (formatted as MM/DD/YYYY) that the calibration began.
- 3.4.18.2.2 Under column "True", enter the expected concentration or true amount of each analyte in the calibration standard or level. It is not required to enter a value for each analyte in every standard, so long as a value is entered for each concentration used to calibrate the instrument for the analyte.
- 3.4.18.2.3 Under column "Found", enter the measured concentration or amount of each analyte in the calibration standard obtained when the calibration standard was refitted to the calibration equation.
- 3.4.18.2.4 Under column "%D", enter the percent difference for each analyte at each concentration or amount used to calibrate the instrument other than the blank standard. Calculate the percent difference (reported to the nearest whole number) according to the following equation:

EQ. 16 Percent Difference

$$\%D = \frac{\text{Found} - \text{True}}{\text{True}} \times 100$$

WHERE,

True = The expected concentration in the calibration standard

Found = The measured concentration in the calibration standard when the response is refitted to the calibration equation

- 3.4.18.2.5 Since a minimum of six levels of calibration are required (a blank plus five standards), submit a minimum of two Forms 15-IN for each calibration performed. Submit a set of Forms 15-IN for each calibration performed for each instrument used to analyze samples.
- 3.4.19 Initial Calibration Summary [Form 16-IN]. This form is not required for Level 2a deliverables.
- 3.4.19.1 Purpose
- This form is used to report instrument response and concentration data for each standard in the initial calibration of an instrument.
- 3.4.19.2 Instructions
- Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.19.2.1 For "Start Date:", enter the date (formatted as MM/DD/YYYY) that the calibration began.
- 3.4.19.2.2 Under column "Corr. Coeff", enter the correlation coefficient calculated for the calibration curve for each analyte calibrated in that analytical sequence to at least four significant figures.
- 3.4.19.2.3 Under column "Slope", enter the calculated slope of the calibration curve for each analyte calibrated in that analytical sequence to at least three significant figures.

- 3.4.19.2.4 Under column "Intercept", enter the calculated intercept of the calibration curve for each analyte calibrated in that analytical sequence to at least three significant figures.
- 3.4.19.2.5 Under column "Calib. Type", enter the calibration type for each analyte calibrated. Report "Lin. Reg" for linear regression; "LR Blank" for linear regression with blank force; "WLR" for weighted linear regression; or "WLR Blank" for weighted linear regression with blank force, as appropriate.
- 3.4.19.2.6 Under column "Weighting", enter the weighting factor for the calibration curve for each analyte calibrated in that analytical sequence. Report "Inverse Conc" for the inverse of the concentration; "Inverse Square Conc" for the inverse square of concentration; "Variance" for variance; "Inverse Variance" for the inverse of the variance; "Standard Deviation" for standard deviation; "Inverse Stand Dev" for the inverse of the standard deviation; "Inverse Square Stand Dev" for the inverse square of the standard deviation; or "None" if no weighting factor was applied.
- 3.4.19.2.7 Submit one set of Forms 16-IN for each calibration performed for each instrument used to analyze samples in the reported SDG.

3.5 Sample Log-In Sheet [Form DC-1]

3.5.1 Purpose

This form is used to document the receipt and inspection of samples and containers. At least one original Form DC-1 is required for each sample shipping container (e.g., cooler). If the samples in a single sample shipping container must be assigned to more than one SDG, the original Form DC-1 shall be placed with the deliverables for the SDG that has the lowest alpha-numeric number and a copy of Form DC-1 shall be placed with the deliverables for the other SDG(s). The copies should be identified as "copy(ies)", and the location of the original should be noted on the copies.

3.5.2 Instructions

- 3.5.2.1 Sign and date the airbill. (If an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information).
- 3.5.2.2 Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in Item 1.
- 3.5.2.3 Record the custody seal numbers in Item 2.
- 3.5.2.4 Open the container, remove the enclosed sample documentation, and record the presence/absence of EPA forms (i.e., TR/COC Records, packing lists) and airbills or airbill stickers in Items 3 and 4. Specify if there is an airbill present or an airbill sticker in Item 4. Record the airbill or sticker number in Item 5.
- 3.5.2.5 Remove the samples from the shipping container(s), examine the samples and the Sample Tags (if present), and record the condition of the sample bottles (i.e., intact, broken, leaking) and presence or absence of Sample Tags in Items 6 and 7.

Exhibit B - Section 3

- 3.5.2.6 Record the presence or absence of a shipping container temperature indicator bottle in Item 8.
- 3.5.2.7 Record the shipping container temperature in Item 9. If ice is present, that shall be noted in the "Remarks" column.
- 3.5.2.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and mark the appropriate answer in Item 10.
- 3.5.2.9 The log-in date should be recorded at the top of Form DC-1; record the date and time of shipping container receipt at the laboratory in Items 11 and 12.
- 3.5.2.10 If there are no problems observed during receipt, sign and date (include the time) Form DC-1 and the TR/COC Record, and write the sample numbers in the "EPA Sample #" column.
- 3.5.2.11 Record the pH for all aqueous/water samples received.
- 3.5.2.12 Record the appropriate Sample Tags and assigned laboratory numbers, if applicable.
- 3.5.2.13 Any comments should be made in the "Remarks" column.
- 3.5.2.14 For Items 1, 3, 4, 6, 7, 8, and 10, circle the appropriate response. Responses can be underlined if this form is completed by automated equipment. Unused columns and spaces shall be crossed out, initialed, and dated.
- 3.5.2.15 If there are problems observed during receipt (including samples that have not been preserved to the proper pH) or an answer marked with an asterisk (e.g., "absent*") was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log and in the SDG Narrative. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

3.6 Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

3.6.1 Purpose

The CSF Inventory Sheet is used to record both the inventory of CSF documents and the number of documents in the original Sample Data Package which is sent to the EPA Region.

3.6.2 Instructions

- 3.6.2.1 Organize all EPA-CSF documents as described in Exhibit B, Sections 2.0 and 3.0. Assemble the documents in Exhibit B, Section 2.0 in the order specified on Form DC-2, and stamp each page with the consecutive number. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record in the "Comments" section on Form DC-2 all intentional gaps in the page numbering sequence (for example, "page numbers not used, XXXX-XXXX, XXXX-XXXX"). If there are no documents for a specific document type, enter an "NA" in the empty space.
- 3.6.2.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review Form DC-2 to determine if it is most appropriate to place them under Categories 66 through 68. Category 68 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

3.6.2.3 If it is necessary to insert new or inadvertently omitted documents, the Contractor shall follow these steps:

- Number all documents to be inserted with the next sequential numbers and file the inserts in their logical positions within the CSF (e.g., document to be inserted between pages 6 and 7 shall be numbered as 6a, 6b, 6c, etc.). Identify where the inserts are filed in the CSF by recording the document numbers and their locations under the "Other Records" section of Form DC-2 (e.g., documents to be inserted between pages 6 and 7 shall be numbered as 6a, 6b, 6c, etc.).

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

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EXHIBIT B
INORGANIC FORMS

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EPA SAMPLE NO.

FORM 13-IN
ICP-MS TUNE

--

Lab Name: _____ Contract: _____

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

Instrument ID: _____ Date: _____

Run Batch: _____

Element - Mass	Avg. Measured Mass (u)	Average Peak Width (u)	%Height	%RSD
Be - 9				
Mg - 24				
Mg - 25				
Mg - 26				
Co - 59				
In - 113				
In - 115				
Pb - 206				
Pb - 207				
Pb - 208				

FORM DC-1
SAMPLE LOG-IN SHEET

Lab Name		Page of
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	SDG No.	MA No.

Remarks:	
1. Custody Seal(s)	Present/Absent* Intact/Broken
2. Custody Seal Nos.	_____
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*
4. Airbill	Airbill/Sticker Present/Absent*
5. Airbill No.	_____
6. Sample Tags	Present/Absent*
Sample Tag Numbers	Listed/Not Listed on Traffic Report/Chain of Custody Record
7. Sample Condition	Intact/Broken*/Leaking
8. Shipping Container Temperature Indicator Bottle	Present/Absent*
9. Shipping Container Temperature	_____
10. Does information on Traffic Reports/Chain of Custody Records and Sample Tags agree?	Yes/No*
11. Date Received at Lab	_____
12. Time Received	_____

	EPA Sample #	Aqueous/ Water Sample pH	Corresponding		Remarks: Condition of Sample Shipment, etc.
			Sample Tag #	Assigned Lab #	
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

FORM DC-2
FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LAB NAME	_____
LAB CODE	_____
CONTRACT NO.	_____
CASE NO.	_____ SDG NO. _____
MA NO.	_____
SOW NO.	_____

All documents delivered in the Complete SDG File must be original documents where possible. (Reference - Exhibit B Section 2.4)

	<u>PAGE NOS.</u>		<u>CHECK</u>	
	<u>FROM</u>	<u>TO</u>	<u>LAB</u>	<u>REGION</u>
1. SDG Cover Page	_____	_____	_____	_____
2. Traffic Report/Chain of Custody Record(s)	_____	_____	_____	_____
3. Sample Log-In Sheet (DC-1)	_____	_____	_____	_____
4. CSF Inventory Sheet (DC-2)	_____	_____	_____	_____
5. SDG Narrative	_____	_____	_____	_____
Inorganic Analysis				
ICP-AES				
6. Inorganic Analysis Data Sheet (Form 1-IN)	_____	_____	_____	_____
7. Initial and Continuing Calibration Verification (Form 2-IN)	_____	_____	_____	_____
8. Blanks (Form 3-IN)	_____	_____	_____	_____
9. ICP Interference Check Sample (Form 4-IN)	_____	_____	_____	_____
10. Matrix Spike Sample Recovery (Form 5A-IN)	_____	_____	_____	_____
11. Post-Digestion/Distillation Spike Sample Recovery (Form 5B-IN)	_____	_____	_____	_____
12. Duplicates (Form 6-IN)	_____	_____	_____	_____
13. Laboratory Control Sample (Form 7-IN)	_____	_____	_____	_____
14. ICP-AES and ICP-MS Serial Dilutions (Form 8-IN)	_____	_____	_____	_____
15. Method Detection Limit (Form 9-IN)	_____	_____	_____	_____
16. ICP-AES Interelement Correction Factors (Form 10A-IN)	_____	_____	_____	_____
17. ICP-AES Interelement Correction Factors (Form 10B-IN)	_____	_____	_____	_____
18. Analysis Log (Form 12-IN)	_____	_____	_____	_____
19. Initial Calibration (Form 15-IN)	_____	_____	_____	_____

FORM DC-2
FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

	<u>PAGE NOS.</u>		<u>CHECK</u>	
	<u>FROM</u>	<u>TO</u>	<u>LAB</u>	<u>REGION</u>
20. Initial Calibration Summary (Form 16-IN)	_____	_____	_____	_____
21. ICP-AES Raw Data	_____	_____	_____	_____
22. ICP-AES Preparation Log Books, Preparation records, Analysis records, and PE Instructions	_____	_____	_____	_____
ICP MS				
23. Inorganic Analysis Data Sheet (Form 1-IN)	_____	_____	_____	_____
24. Initial and Continuing Calibration Verification (Form 2-IN)	_____	_____	_____	_____
25. Blanks (Form 3-IN)	_____	_____	_____	_____
26. ICP Interference Check Sample (Form 4-IN)	_____	_____	_____	_____
27. Matrix Spike Sample Recovery (Form 5A-IN)	_____	_____	_____	_____
28. Post-Digestion/Distillation Spike Sample Recovery (Form 5B-IN)	_____	_____	_____	_____
29. Duplicates (Form 6-IN)	_____	_____	_____	_____
30. Laboratory Control Sample (Form 7-IN)	_____	_____	_____	_____
31. ICP-AES and ICP-MS Serial Dilutions (Form 8-IN)	_____	_____	_____	_____
32. Method Detection Limit (Form 9-IN)	_____	_____	_____	_____
33. ICP-MS Internal Standard Association (Form 11-IN)	_____	_____	_____	_____
34. Analysis Log (Form 12-IN)	_____	_____	_____	_____
35. ICP-MS Tune (Form 13-IN)	_____	_____	_____	_____
36. ICP-MS Internal Standards Relative Intensity Summary (Form 14-IN)	_____	_____	_____	_____
37. Initial Calibration (Form 15-IN)	_____	_____	_____	_____
38. Initial Calibration Summary (Form 16-IN)	_____	_____	_____	_____
39. ICP-MS Raw Data	_____	_____	_____	_____
40. ICP-MS Preparation Log Books, Preparation records, Analysis records, and PE Instructions	_____	_____	_____	_____
Mercury				
41. Inorganic Analysis Data Sheet (Form 1-IN)	_____	_____	_____	_____
42. Initial and Continuing Calibration Verification (Form 2-IN)	_____	_____	_____	_____
43. Blanks (Form 3-IN)	_____	_____	_____	_____
44. Matrix Spike Sample Recovery (Form 5A-IN)	_____	_____	_____	_____
45. Duplicates (Form 6-IN)	_____	_____	_____	_____
46. Method Detection Limit (Form 9-IN)	_____	_____	_____	_____
47. Analysis Log (Form 12-IN)	_____	_____	_____	_____
48. Initial Calibration (Form 15-IN)	_____	_____	_____	_____

FORM DC-2
FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

- | | | | | |
|---|--|--|--|--|
| 49. Initial Calibration Summary (Form 16-IN) | | | | |
| 50. Mercury Raw Data | | | | |
| 51. Mercury Preparation Log Books, Preparation records, Analysis records, and PE Instructions | | | | |

Cyanide

- | | | | | |
|---|--|--|--|--|
| 52. Inorganic Analysis Data Sheet (Form 1-IN) | | | | |
| 53. Initial and Continuing Calibration Verification (Form 2-IN) | | | | |
| 54. Blanks (Form 3-IN) | | | | |
| 55. Matrix Spike Sample Recovery (Form 5A-IN) | | | | |
| 56. Post-Digestion/Distillation Spike Sample Recovery (Form 5B-IN) | | | | |
| 57. Duplicates (Form 6-IN) | | | | |
| 58. Method Detection Limit (Form 9-IN) | | | | |
| 59. Analysis Log (Form 12-IN) | | | | |
| 60. Initial Calibration (Form 15-IN) | | | | |
| 61. Initial Calibration Summary (Form 16-IN) | | | | |
| 62. Cyanide Raw Data | | | | |
| 63. Cyanide Preparation Log Books, Preparation records, Analysis records, and PE Instructions | | | | |

Additional

- | | | | | |
|--|--|--|--|--|
| 64. Percent Solids Determination Log | | | | |
| 65. EPA Shipping/Receiving Documents
Airbill (No. of Shipments _____)
Sample Tags
Sample Log-In Sheet (Lab) | | | | |
| 66. Misc. Shipping/Receiving Records
(list all individual records)
Communication Logs

_____ | | | | |
| 67. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)

_____ | | | | |
| 68. Other Records (describe or list)
Communication Logs

_____ | | | | |

FORM DC-2
FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

69. Comments:

Completed by:
(CLP Lab)

(Signature)

(Print Name & Title)

(Date)

Audited by:
(EPA)

(Signature)

(Print Name & Title)

(Date)

EXHIBIT C

INORGANIC TARGET ANALYTE LIST AND
CONTRACT REQUIRED QUANTITATION LIMITS

NOTE: The Contract Required Quantitation Limit (CRQL) values listed on the following pages are based on the analysis of samples according to the specifications given in Exhibit D.

Changes to the CRQL may be requested under the Modified Analysis (MA) clause in the contract.

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Exhibit C - Inorganic Target Analyte List and Contract
Required Quantitation Limits

Table of Contents

<u>Section</u>	<u>Page</u>
1.0 ICP-AES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS	5
2.0 ICP-MS TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS	6
3.0 MERCURY BY COLD VAPOR ATOMIC ABSORPTION TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS	6
4.0 CYANIDE BY SPECTROPHOTOMETRY TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS	6

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1.0 ICP-AES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 1. ICP-AES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS^A

Analyte Name	CAS Number	CRQL			
		Water ^D (µg/L)	Soil ^B (mg/kg)	Wipe (µg)	TCLP (mg/L)
Aluminum	7429-90-5	200	20	20	--
Antimony	7440-36-0	60	6	6	--
Arsenic	7440-38-2	10	1	1	5
Barium	7440-39-3	200	20	20	100
Beryllium	7440-41-7	5	0.5	0.5	--
Cadmium	7440-43-9	5	0.5	0.5	1
Calcium	7440-70-2	5000	500	500	--
Chromium	7440-47-3	10	1	1	5
Cobalt	7440-48-4	50	5	5	--
Copper	7440-50-8	25	2.5	2.5	--
Iron	7439-89-6	100	10	10	--
Lead	7439-92-1	10	1	1	5
Magnesium	7439-95-4	5000	500	500	--
Manganese	7439-96-5	15	1.5	1.5	--
Nickel	7440-02-0	40	4	4	--
Potassium	7440-09-7	5000	500	500	--
Selenium	7782-49-2	35	3.5	3.5	1
Silver	7440-22-4	10	1	1	5
Sodium	7440-23-5	5000	500	500	--
Thallium	7440-28-0	25	2.5	2.5	--
Vanadium	7440-62-2	50	5	5	--
Zinc	7440-66-6	60	6	6	--
Hardness (total)	Hardness	33 ^C	--	--	--

2.0 ICP-MS TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 2. ICP-MS TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS^A

Analyte Name	CAS Number	CRQL	
		Water (µg/L)	Soil ^B (mg/kg)
Aluminum	7429-90-5	20	--
Antimony	7440-36-0	2	1
Arsenic	7440-38-2	1	0.5
Barium	7440-39-3	10	5
Beryllium	7440-41-7	1	0.5
Cadmium	7440-43-9	1	0.5
Calcium	7440-70-2	500	--
Chromium	7440-47-3	2	1
Cobalt	7440-48-4	1	0.5
Copper	7440-50-8	2	1
Iron	7439-89-6	200	--
Lead	7439-92-1	1	0.5
Magnesium	7439-95-4	500	--
Manganese	7439-96-5	1	0.5
Nickel	7440-02-0	1	0.5
Potassium	7440-09-7	500	--
Selenium	7782-49-2	5	2.5
Silver	7440-22-4	1	0.5
Sodium	7440-23-5	500	--
Thallium	7440-28-0	1	0.5
Vanadium	7440-62-2	5	2.5
Zinc	7440-66-6	2	1

3.0 MERCURY BY COLD VAPOR ATOMIC ABSORPTION TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 3. MERCURY BY COLD VAPOR ATOMIC ABSORPTION TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

Analyte Name	CAS Number	Water ^D (µg/L)	CRQL	
			Soil ^B (mg/kg)	TCLP (mg/L)
Mercury	7439-97-6	0.2	0.1	0.2

4.0 CYANIDE BY SPECTROPHOTOMETRY TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 4. CYANIDE BY SPECTROPHOTOMETRY TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

Analyte Name	CAS Number	CRQL	
		Water ^D (µg/L)	Soil ^B (mg/kg)
Cyanide	57-12-5	10	0.5

Endnotes:

- A. Changes to the Inorganic Target Analyte List (TAL) (e.g., adding an additional analyte) may be requested under the Modified Analysis clause in the contract.
- B. The CRQLs for soil/sediment are based on 100% solids and on the minimum weights and volumes specified in Exhibit D. The moisture content of the samples must be used to adjust the CRQL values appropriately.
- C. Hardness (total) is reported as a calculation in mg/L.
- D. Use the water CRQLs for Synthetic Precipitation Leaching Procedure (SPLP).

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