

Validation of Metals for the Contract Laboratory Program (CLP) based on
SOW ILMO5.3 (SOP Revision 13)



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Standard Operating Procedure
 USEPA Region 2
 Evaluation of Metals Data for the Contract Laboratory Program
 Data Assessment and Contract Compliance Review

SOP: HW-2 Revision 13

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1.0 **Scope**

- 1.1 This Standard Operating Procedure (SOP) applies to the evaluation of Routine Analytical Services (RAS) inorganic data generated in accordance with the EPA Contract Laboratory Program (CLP) protocols.
- 1.2 This Region 2 inorganic data validation SOP is used to determine the usability of analytical data generated from water and soil/sediment samples collected from Superfund sites in EPA Region 2.
- 1.3 Data should be generated and validated in accordance with the site specific Project Quality Objectives (PQOs) developed prior to the sample collection event. This SOP can be customized to validate the data according to the site specific PQOs. If the site specific DQOs are not available, this SOP must be used in its entirety.
- 1.4 This SOP is based, for the most part, upon analytical and quality assurance requirements specified in the Statement of Work SOW-ILM05.3, as well as in the final (October 2004) of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. The SOP Checklist, Appendix A.1, provides guidance in conducting the data validation. The result of the use of this SOP is a **Total Review** of the data: **Technical plus Contract - Compliance Review**.

2.0 **Contract Compliance Review**

This type of review is the first step in data validation which is carried out to ensure that the CLP laboratory has analyzed the environmental samples in accordance with the Statement of Work (SOW), and provided a data package which is both complete and compliant. This means that laboratory's procedures were performed exactly as specified in the CLP Statement of Works (SOW) and the data package contains all the deliverables including the information required under the contract.

2.1 **Completeness**

The data validator must check the entire data package to ensure that all deliverables required under the CLP contract are present and legible. In addition, copies of the Contract Compliance Screening (CCS) report, re-submittal from the laboratory, and Regional documentation should also be present in the data package. In Region 2, the data package completeness check is currently performed by the Regional Sample Control Coordinator (RSCC) for each Sample Delivery Group (SDG). The data package is not released to the data validator until all the required deliverables are received from the laboratory.

2.2 **Compliance**

The data validator must check to ensure that all steps from sample receipt through sample preparation, analysis, data calculation and reporting are documented, and the information/data required under the contract is present in the appropriate reporting Forms and laboratory logs.

2.3 **Contract Compliance Screening (CCS)**

This screening step essentially checks the data package for the Completeness and Compliance requirements, and is performed by the Sample Management Office (SMO) currently operated by Computer Sciences Corporation (CSC), an EPA contractor. The CCS Report outlines the incomplete and non-compliant items as "Defects" in the data package, and is sent to the laboratory which is required to

provide additional or missing information/data required under the contract. The CCS Report for each SDG is transmitted electronically by the SMO to the Regional office. The CCS Report is intended to aid the data validator in locating any problems, both corrected and uncorrected. The incorrect original deliverable(s) of the data package must be replaced by the re-submittal(s) received from the laboratory in response to the CCS Report. The data validation should, however, be carried out even if the CCS Report is not available.

Web-based CCS is available for CLP laboratories to check their data prior to its delivery to EPA.

3.0 Technical Review

Technical review of the RAS data is carried out on the complete and compliant data to ensure its **validity** (i.e., data is of known quality and scientifically valid) and **usability** (i.e., data set is sufficiently complete and of sufficient quality to support a decision or an action described in the specific objectives of a data collection activity). The technical review process provides information on analytical limitations of data, if any, based on specific Quality Assurance/Quality Control (QA/QC) criteria. This is accomplished by performing an in-depth review of both the field deliverables which document the field sampling activities, and the laboratory analytical data deliverables which document the laboratory activities carried out to generate the reported data. Essentially, the validator shall first ensure that the data package is complete and compliant. The validator shall then evaluate data/information on all these deliverables (Final data sheets, Forms for QC analyses Chain-of-Custody/Traffic Report Forms, raw data, etc.) against the QA/QC acceptance criteria specified in the SOP "Checklist" (Appendix A.1). The validator must answer each question in the "Checklist" and take an appropriate action as required under "Action" to qualify the data. As a result of the technical review, the data validator may qualify some of the data as **rejected** or as **estimated**. The data validator shall write a **Data Review Narrative** documenting the qualified data and the reason(s) for the qualification.

- 3.1 If the **raw data** necessary to support the reported results are not provided, the data validation must not be performed. The laboratory must be contacted to obtain missing raw data.
- 3.2 If batch quality control analyses are performed on samples other than **site specific samples**, data must not be validated or at best be considered as estimated. The data user must be notified of this action.
- 3.3 QA/QC Acceptance Criteria
In order that reviews be consistent among reviewers, QA/QC protocol (stated in Appendix A.1) should be strictly adhered to. If a lab provides more than one set of QC analyses or more than one particular QC analysis for an SDG, the validator shall use the worst QC analysis to evaluate the SDG data. Professional judgement should only be used in the rare instances not addressed in the "Checklist".

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3.4 Data Validation Flags

Three types of data validation flags (J, R & U) are used in Region 2 to qualify the data.

3.4.1 Flag "R" indicates Rejected Data

Sample results determined to be unacceptable must preferably be lined over and flagged "R" with a red pencil only on the Inorganic Analysis Data Sheets (CLP Form I's). Data rejected on the basis of an unacceptable QC analysis should be excluded from further review or consideration. Data are rejected when associated QC analysis results exceed the expanded control limits of the QC criteria. The rejected data are known to contain significant errors based on documented information. The data user **must not** use the rejected data to make environmental decisions.

3.4.2 Flag "J" indicates Estimated Data

Sample results determined to be estimated must be flagged "J" with a red pencil only on the CLP Form I's. Data are flagged (J) when a QC analysis falls outside the primary acceptance limits. The qualified "J" data are not excluded from further review or consideration. However, only one flag (J) is applied to a sample result even though several associated QC analyses may fail. The "J" data may be biased high or low.

3.4.3 Flag "U" indicates Non-Detects

Sample results \geq MDL associated with a contaminated blank are flagged "U" with a red pencil only on Form I's.

4.0 Contractual Qualifiers

The CLP laboratory applies contractual qualifiers on all Form I's and the QC Forms when QC analyses are outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets with the exception of U and J. The contractual qualifiers and their meanings are as follows:

N : This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.

E : This qualifier indicates the presence of interference, and is applied when the ICP serial dilution analysis is outside the control limits.

* : This qualifier indicates the lack of precision, and is applied to sample results on Form I's and Form VI when the Lab Duplicate analysis is outside the control limits.

U : This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analysis is indicated by the Contract Required Quantitation Limit (CRQL) of that analyze suffixed with "U".

J : This is a concentration qualifier that the laboratory applies to a positive result below the CRQL (i.e., \geq MDL but $<$ CRQL).

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

4.0 **Rounding Rule**

The data reviewer must follow the standard practice to round off percent recoveries on the QC reporting forms.

5.0 **Data Review Narrative (Appendix A.2)**

The data review narrative should be written using the format of Appendix A.2. The narrative should indicate the QC analyses outside the acceptance limits and the actions taken to qualify the associated data. The narrative should be prepared on a Personal Computer or a typewriter. If hand-written, under no circumstances should a pencil be used to write the narrative. The Data Review Narrative should be written in four (4) Sections: (i) Data Case Description, (ii) Complete SDG File (CSF) Audit Section, (iii) Technical Review Section, and (iv) Contract-Problems/Non-Compliance Section.

5.1 **Data Case Description Section**

The data validator must briefly describe the data case in this Section, outlining important information such as the number of samples, their matrix, sampling date(s), analysis (TAL metals, mercury or cyanide), samples used for QC analyses, Field Blank(s), Field Duplicates, etc.

5.2 **Complete SDG File (CSF) Audit Section**

The data validator must perform an audit on each SDG in the data package to ensure that all SDG-specific documents (sampling, samples shipping and receiving, telephone contact logs, etc.) are present in the data case. The audit shall also discover any discrepancy in the deliverables. In Region 2, this audit is currently performed by the ESAT data validator and its findings reported under "Comments" on a CSF inventory checklist. The validator informs the CLP Project Officer (PO) of the missing or additional information/deliverable required for data validation. The PO then contacts the lab for the desired deliverable/information. The findings of the CSF audit are reported in the CSF Section of the Data Review Narrative (Appendix A.2).

5.3 **Technical Review Section**

The data validator shall report in this Section only the rejected (R) and estimated data (J) and the data rendered non-detects (U) as a result of technical review. It is imperative that the data reviewer highlights (i) QC analysis criteria applied to reject (R) or flag (J, U) the data, (ii) Samples rejected (R) or flagged (J, U), and (iii) the QC analysis out of control limits. The rest of the data that are not qualified (rejected or estimated) are not reported in this Section, and should be considered **fully useable**.

5.4 **Contract-Problems/Non-Compliance Section**

All the CLP non-compliant items detected during data review must be reported in this Section.

6.0 **Computer-Aided Data Review and Evaluation (CADRE)**

CADRE is a computer program that performs semi-automated Quality Assurance (QA) and Quality Control (QC) checks of results from the chemical analysis of soil and water samples according to the CLP protocols. After the CADRE data qualification is complete, a Lotus 1,2,3 spreadsheet or an XLS spreadsheet with data validation qualifiers (R,J,U) is generated for each SDG. Currently, Sample Management Office (SMO) performs this task using Data Assessment Tool (DAT), a software-driven process, and forwards to the Regions the customized electronic spreadsheets (Lotus 1,2,3 or XLS spreadsheet) and QC reports via the DART (Data Assessment Rapid Transmittal) system. Manual data validation is performed in conjunction with electronic data validation which can only be done by a trained and experienced data validator. The manual data review complements CADRE's findings to complete an assessment of data quality in a shorter time than by a solely manual process. The data validator must review the XLS or Lotus 1,2,3 spreadsheet against Form I's to ensure that the same results on Form I's and the Spreadsheet are qualified with the same data validation qualifiers. The spreadsheet for each SDG is provided with the Data Review Narrative.

7.0 **Performance Evaluation Sample(PES)Based Data Validation Strategy**

7.1 **Scope and Summary**

This strategy offers the use of Performance Evaluation Samples (PES) in the data validation process as a means of ensuring the quality of the CLP data while significantly reducing the validation time. The single blind PES provided by EPA (or any other reputable firm) is analyzed with samples of each matrix in a Sample Delivery Group (SDG). A software program (e.g., PEAC TOOLS, SPS Web or equivalent) is used to determine whether or not the PES results fall within the previously statistically determined acceptance limits ("Action Low" and "Action High") for the Contaminants of Concern (COC). The PES results falling within the Action Limits are considered as acceptable results and may be designated as "Passed" analytes, and results of the analytes falling outside the Action Limits are considered as unacceptable and may be designated as "Failed" analytes. In either case ("Passed" Analytes or "Failed" analytes), the associated data is validated according to the Region 2 data validation SOP HW-2 in conjunction with the latest version of the WinCadre QC reports. The following strategy (procedure) is used:

7.2 **"Passed" COC**

If the COC in an SDG are within statistically generated Action Limits, the data validation is conducted according to QC analyses indicated by check marks (√) in the "Review COC For" column of the Table I. The SDG samples are validated using the Region 2 data validation SOP in

conjunction with the latest version of the WinCADRE QC reports. The validation flags (J, R, U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS spreadsheet. Corrections, if needed, are then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.3 "Failed" COC

If the COC in an SDG are not within the statistically generated Action Limits, the data validation is conducted according to the data validation SOP QC Criteria indicated by check marks (√) in the "Review COC For" column of Table II. The SDG samples are validated using the Region 2 data validation SOP in conjunction with the latest version of the WinCADRE QC reports. The data validation flags (J,R,U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS Spreadsheet. Corrections, if needed, are then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.4 COC "Not Evaluated"

Acceptance limits for the analytes not present/spiked in the PE sample are not provided on the PES Scoring Evaluation Report. Such analytes will be marked as "Not Evaluated" in the PES Evaluation Column. These analytes will be validated much the same way as the "Failed Analytes".

The failed analytes and the analytes not present/spiked in the PE sample require data validation according to the QC criteria specified in Table II, and are identified by the TOPO in the TDF for the Case/SDG.

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Table 1

Passed PES - All Contaminants of Concern are within the limits
 (Action Low \leq PES Result \leq Action High)

QC Criteria	Review COC for
Holding Time & Preservation	√
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	√
Blanks-Initial & Continuing	
Preparation Blank	
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	
Post Digestion Spike	
Laboratory Duplicate	
Field Duplicates Comparison	√
Lab Control Sample	
ICP Serial Dilution	
Field Blank Contamination	√
Percent Solids	√
Transcription/Computation Check	
Raw Data	
Total vs. Dissolved Concentrations Comparison	√

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied. The Contract
- Compliance can be checked after the PES validation strategy is applied.

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Table II

Failed PES - Contaminants of Concern are not within the limits
 (PES Result \leq Action Low, PES Result \geq Action High **OR** The Limits Not Established)

QC Criteria	Review COC for
Holding Time & Preservation	√
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	√
Blanks-Initial & Continuing	
Preparation Blank	√
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	√
Post Digestion Spike	
Laboratory Duplicate	√
Field Duplicates Comparison	√
Lab Control Sample	√
ICP Serial Dilution	√
Field Blank Contamination	√
Percent Solids	√
Transcription/Computation Check	√
Raw Data	
Total vs. Dissolved Concentrations Comparison	√

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied.
- The Contract Compliance can be checked after the PES validation strategy is applied.

8.0 Sampling Trip Report

The sampler prepares a Sampling Trip Report for each sampling event and sends it to the RSCC. The report provides details of all activities performed for each sampling event on the Superfund site. It also lists the field QC samples such as Field Duplicates, Field/Rinse Blanks, sampling time and date for each sample, and samples associated with each field/rinse blank. The validator must use this information to evaluate the Field Duplicate pairs as well as the samples associated with contaminated Field/Rinse Blanks.

9.0 Telephone Record Log (Appendix A.3)

A Telephone Record Log (Appendix A.3) must be written by the data validator when a deliverable is missing or a clarification is needed about a lab procedure. The data validator should outline a basic profile of the Case on the Telephone Record Log Form, clearly indicating the reason(s) for inquiry and forward this Form to CLP PO/TOPO who will contact the lab to receive the missing document or information. The original Telephone Record Log is kept in the data package and a copy attached to the Data Review Narrative.

10.0 Request for Re-Analysis (Appendix A.6)

Data validator must note all items of contract non-compliance in the Data Review Narrative. If holding times and sample storage times have not been exceeded, the Project Officer (PO) may request re-analysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record" form (Appendix A.4).

11.0 CLP Data Assessment Summary Form (Appendix A.7)

Fill in the total number of analytes performed by different methods and the number of analytes rejected (R) or flagged (J) as estimated due to corresponding quality control criteria. Place an "X" in boxes wherever analyses were not performed, or criteria do not apply.

12.0 Data Review Log:

It is recommended that the data validator maintain a log of the reviews completed to document:

- a. Case number
- b. SDG # (s)
- c. number of samples
- d. matrix of samples
- e. contract laboratory
- f. site name
- g. start-date of the data case review
- h. completion-date of the data case review
- i. actual hours spent
- j. reviewer's signature

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13.0 Record of Communication -

This is a Regional document prepared and provided by the RSCC for each data package. The ROC indicates the Case #, site name, samples and sample matrix and the laboratory name. The presence of a ROC in a data package is an indication that the package has been reviewed by the RSCC for completeness and is ready for data validation.

14.0 Forwarded Paperwork

Upon completion of review, the following are to be forwarded to EPA for final review:

- a. Data package
- b. Completed data assessment checklist (Appendix A.1,original)
- c. Original and a copy of completed data review narrative Appendix A.2)
- d. CLASS Contract Compliance Screening (CCS) report
- e. Telephone Record Log (Appendix A.3)
- f. Field Duplicates Form (Appendix A.4)
- g. Total/Dissolved Concentrations Form
(Appendix A.5)
- h. CLP Re-analysis Request/Approval Record Form (Appendix A.6)
- i. Data Assessment Summary Form (Appendix A.7)
- j. CADRE Spreadsheet on a computer diskette.

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ACRONYMS

AA	Atomic Absorption
AOC	Analytical Operations/Data Quality Center
CADRE	Computer-Aided Data Review and Evaluation
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CO	Contracting Officer
COC	Contaminants of Concern
CRI	CRQL Check Standard
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
CVAA	Cold Vapor AA
DART	Data Assessment Rapid Transmittal
DAT	Data Assessment Tool
DF	Dilution Factor
DQO	Data Quality Objective
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Sample
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LRS	Linear Range Sample
MDL	Method Detection Limit
NIST	National Institute of Standards and Technology
OERR	Office of Emergency and Remedial Response
OSWER	Office of Solid Waste and Emergency Response
PB	Preparation Blank
PE	Performance Evaluation
%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%RSD	Percent Relative Standard Deviation
%S	Percent Solids
PO	Project Officer
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center

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SDG Sample Delivery Group
 SMO Sample Management Office
 SOP Standard Operating Procedure
 SOW Statement of Work
 TAL Target Analyze List
 TR/COG Traffic Report/Chain of Custody Documentation

Inorganic Target Analyze List And Contract Required Quantitation Limits (CRQLs)

Analyze	CAS Number	ICP-AES CRQL	ICP-AES CRQL	ICP-MS CRQL
		Water Ug/L	Soil mg/kg	Water Ug/L
Aluminum	7429-90-5	200	20	---
Antimony	7440-36-0	60	6	2
Arsenic	7440-38-2	10	1	1
Barium	7440-39-3	200	20	10
Beryllium	7440-41-7	5	0.5	1
Cadmium	7440-43-9	5	0.5	1
Calcium	7440-70-2	5000	500	-----
Chromium	7440-47-3	10	1	2
Cobalt	7440-48-4	50	5	1
Copper	7440-50-8	25	2.5	2
Iron	7439-89-6	100	10	----
Lead	7439-92-1	10	1	1
Magnesium	7439-95-4	5000	500	-----
Manganese	7439-96-5	15	1.5	1
Mercury	7439-97-6	0.2	0.1	---
Nickel	7440-02-0	40	4	1
Potassium	7440-09-7	5000	500	-----
Selenium	7782-49-2	35	3.5	5
Silver	7440-22-4	10	1	1
Sodium	7440-23-5	5000	500	-----
Thallium	7440-28-0	25	2.5	1
Vanadium	7440-62-2	50	5	1
Zinc	7440-66-6	60	6	2
Cyanide	57-12-5	10	2.5	----

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Site:

Case #:

SDG #:

Samples: Soil Water

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Appendix A.1

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YES NO N/A

A.1.1 **Contract Compliance Screening Report**

Present?

 ___ ___

ACTION: If no, contact RSCC/PO.

A.1.2 **Record of Communication (from RSCC)**

Present?

 ___ ___

ACTION: If no, request from the RSCC.

A.1.3 **Sampling Trip Report**

Present and complete?

 ___ ___

ACTION: If no, contact RSCC/PO.

A.1.4 **Chain of Custody/Sample Traffic Report**

Present?

 ___ ___

Legible?

 ___ ___

Signature of sample custodian
present?

 ___ ___

ACTION: If no, contact RSCC/WAM/PO.

A.1.5 **Cover Page**

Present?

 ___ ___

Is the Cover Page properly filled in
and the verbatim signed by the lab
manager or the manager's designee?

 ___ ___

Do the sample identification numbers
on the Cover Page agree with sample
Identification numbers on:

(a) Traffic Report Sheet?

 ___ ___

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	<u>YES</u>	<u>NO</u>	<u>N/A</u>
(b) Form I's?	[]	__	__
Is the number of samples on the Cover Page the same as the number of samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data Case?	[]	__	__

ACTION:

If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

A.1.6 SDG Narrative, DC-1 & DC-2 Form

Is the SDG Narrative present?	[]	__	__
Is Sample Log-In Sheet(Form DC-1) present and complete?	[]	__	__
Is Complete SDG Inventory Sheet(Form DC-2) present and complete?	[]	__	__

ACTION:

If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.

A.1.7 Form I to XV

A.1.7.1 Are all the Form I through Form XV labeled with:

Laboratory Name?	[]	__	__
Laboratory Code?	[]	__	__
RAS/Non-RAS Case No.?	[]	__	__
SDG No.?	[]	__	__

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	<u>YES</u>	<u>NO</u>	<u>N/A</u>
Contract No.?	<input type="checkbox"/>	_	_

ACTION:

If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

A.1.7.2 After comparing values on Forms I-IX against the raw data, do any computation/transcription errors exceed 10% of the reported values on the Forms for:

- | | | | |
|---------------------------------------|---|--------------------------|---|
| (a) all analytes analyzed by ICP-AES? | _ | <input type="checkbox"/> | _ |
| (b) all analytes analyzed by ICP-MS? | _ | <input type="checkbox"/> | _ |
| (c) Mercury? | _ | <input type="checkbox"/> | _ |
| (d) Cyanide? | _ | <input type="checkbox"/> | _ |

ACTION:

If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

A.1.8 Raw Data

Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

A.1.8.1 Digestion/Distillation Log

Digestion Log for ICP-AES (Form XII) present?	<input type="checkbox"/>	_	_
--	--------------------------	---	---

Digestion Log for ICP-MS (Form XII) present?	<input type="checkbox"/>	_	_
---	--------------------------	---	---

Digestion Log for mercury (Form XII) present?	<input type="checkbox"/>	_	_
--	--------------------------	---	---

Distillation Log for cyanide (Form XII) present?	<input type="checkbox"/>	_	_
---	--------------------------	---	---

Are pH values for metals and

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YES NO N/A

cyanide reported for each aqueous sample?

 _ _

Are percent solids calculations present for soils/sediments?

 _ _

Are preparation dates present on the sample preparation logs/bench sheets?

 _ _

NOTE:

Digestion/Distillation log must include weights, volumes, and dilutions used to obtain the reported results.

A.1.8.2 Is the analytical instrument real-time printouts present for:

ICP-AES?

 _ _

ICP-MS?

 _ _

Mercury?

 _ _

Cyanide?

 _ _

Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:

Legible?

 _ _

Properly labeled?

 _ _

Are all field samples, QC samples and field QC samples present on:

Digestion/Distillation log?

 _ _

Instrument Printouts?

 _ _

ACTION:

If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.

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YES NO N/A

present and complete?

[] — —

ACTION:

If no, prepare Telephone Record Log and contact CLP PO/TOPO for submittal from the laboratory.

A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

Is the calculation error less than 10% of the correct result? [] — —

Are results on Form I's reported in correct units (ug/L for aqueous and MG/KG for soils)? [] — —

Are results on Form I'S reported by correct significant figures? [] — —

Are soil sample results on Form I's corrected for percent solids? [] — —

Are all "less than MDL" values reported by the CRQLs and coded with "U"? [] — —

Are values less than the CRQLs but greater than or equal to the MDLs flagged with "J"? [] — —

Are appropriate contractual quality control and Method qualifiers used? [] — —

ACTION:

If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.

A.1.10.3 Do EPA sample identification numbers and the corresponding laboratory sample identification numbers match on the Cover Page, Form I's and in the raw data?

[] — —

Was a brief physical description

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of the samples before and after digestion given on the Form I's?	[___]	___	___
Was any sample result outside the mercury/cyanide calibration range or the ICP-AES/ICP-MS linear range diluted and noted on the Form I?	[___]	___	___

ACTION:

If no for any of the above, note under the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.11 Initial Calibration

A.1.11.1	Is a record of at least 2 point (A blank and a standard)calibration present for ICP-AES analysis?	[___]	___	___
	Is a record of at least 2 point (a blank and a standard)calibration present for ICP-MS analysis?	[___]	___	___
	Is a record of at least 5 point calibration (a blank & 4 standards)present for Hg analysis?	[___]	___	___
	Is a record of at least 4 point calibration (a blank & 4 standards)present for cyanide?	[___]	___	___

ACTION:

If incomplete or no initial calibration was performed, reject (R) and red-line the associated data (detects & non-detects).

	Is one initial calibration standard at the CRQL level for cyanide and mercury?	[___]	___	___
--	--	-------	-----	-----

ACTION:

If no, write in the Contract Problem/ Non-Compliance Section of the Data Review Narrative .

A.1.11.2	Is the curve correlation coefficient ≥ 0.995 for:			
----------	--	--	--	--

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	<u>YES</u>	<u>NO</u>	<u>N/A</u>
Mercury Analysis?	[]	___	___
Cyanide Analysis?	[]	___	___
ICP-AES (more than 2 point Calib.)?	[]	___	___
ICP-MS (more than 2 point calib.)?	[]	___	___

ACTION:

If no, qualify the associated sample results \geq MDL as estimated "J" and non-detects as "UJ".

NOTE:

The correlation coefficient shall be calculated by the data validator using standard concentrations and the corresponding instrument response (e.g. absorbance, peak area, peak height, etc.).

A.1.12 Initial and Continuing Calibration Verification- Form IIA

A.1.12.1 Present and complete for every metal and cyanide?	[]	___	___
Present and complete for ICP-AES and ICP-MS when both these methods were used for the same analyte?	[]	___	___

ACTION:

If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO for re-submittal from the laboratory.

A.1.12.2 Was a Continuing Calibration Verification performed every 10 samples or every 2 hours whichever is more frequent?	[]	___	___
--	-----	-----	-----

ACTION:

If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.12.3 Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples?	[]	___	___
--	-----	-----	-----

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YES NO N/A

ACTION:

If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative and qualify results \geq MDL as estimated (J).

A.1.12.2 Circle on each Form IIA all percent recoveries that are outside the contract windows.

Are ICV/CCVs within control limits for:

Metals - 90-110%R?	[___]	___	___
Hg - 80-120%R?	[___]	___	___
Cyanide - 85-115%R?	[___]	___	___

ACTION:

If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows:

Qualify as estimated (J) all detects and non-detects, if the ICV/CCV %R is between 75-89%(65-79% for Hg; 70-84% for CN). Qualify only positive results(\geq MDL) as "J" if the ICV/CCV %R is between 111-125%(121-135% for Hg;116-130% for CN). Reject (R) and red-line only detects if the recovery is greater than 125% (135% for Hg; 130% for CN). Reject (R) and red-line all associated results (hits and non-detects)if the recovery is less than 75%(65% for Hg;70% for CN).

NOTE:

For ICV that does not fall within the acceptance limits, qualify all samples reported from the analytical run.

A.1.12.3 Was the distilled ICV or mid-range standard for cyanide within acceptance limits (85-115%)?

[___] ___ ___

ACTION:

If no, Qualify all cyanide results \geq MDL as "J".

A.1.13 CRQL Standard Analysis - Form IIB

A.1.13.1 For each ICP-AES run, was a CRI

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(CRQL or MDL when MDL > CRQL)
 standard analyzed?

(Note: CRI is not required for Al, Ba, Ca, Fe, Mg, Na and K.)

YES	NO	N/A
[]	—	—

For each ICP-MS run, was a CRI (CRQL or MDL when MDL > CRQL) standard analyzed for each mass/isotope used for the analysis?

[]	—	—	—
-----	---	---	---

For each mercury run, was a CRQL standard analyzed?

[]	—	—
-----	---	---

For each cyanide run, was a CRQL standard analyzed?

[]	—	—
-----	---	---

ACTION:

If no for any of the above, write this deficiency in the Contract Problems/ Non-Compliance Section of the Data Review Narrative, inform CLP PO and flag results in the affected ranges (detects <2xCRQL) as J and non-detects UJ.

The affected ranges are:

ICP-AES Analysis - *True Value ± CRQL

ICP-MS Analysis - *True Value ± CRQL

Mercury Analysis - *True Value ± CRQL

Cyanide Analysis - *True Value ± CRQL

* True value of the CRQL Standard

A.1.13.2 Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis?

[]	—	—
-----	---	---

ACTION:

If no, write in the Contract Problem/ Non-Compliance Section of the "Data Review Narrative".

A.1.13.3 Circle on each Form IIB all percent recoveries that are outside the acceptance windows.

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Is the CRQL standard within control limits for:			
Metals(ICP-AES/ICP-MS)- 70 - 130%?	[___]	___	___
Mercury- 70 - 130%?	[___]	___	___
Cyanide - 70 - 130%?	[___]	___	___

ACTION:

If no, flag detects <2xCRQL as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-69%. Flag(J) only detects <2xCRQL if the recovery is between 131% and ≤180%. If the recovery is less than 150%, reject(R) and red-line non-detects and detects < 2xCRQL, and flag (J) detects between 2xCRQL and ICV/CCV. Reject and red-line only detects <2xCRQL and flag (J) detects ≥ 2xCRQL but < ICV/CCV if the recovery is > 180%.

NOTE:

1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard
2. Flag (J) or reject (R) only the final sample results on Form I's when Sample raw data are within the affected ranges and the CRQL standard is outside the acceptance windows.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

A.1.14 Initial and Continuing Calibration Blanks - Form III

A.1.14.1	Present and complete for all the instruments used for the metals and cyanide analyses?	[___]	___	___
	Was an initial Calibration Blank analyzed after ICV?	[___]	___	___
	Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?	[___]	___	___
	Were the ICB & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by			

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YES NO N/A

but \leq CRQL to CRQL with a "U".

A.1.15.2.2 When the MDL \leq CRQL, is any Preparation Blank value greater than its CRQL?

___ [___] ___

If yes, is the Prep. Blank value greater than the value of the associated Field Blank collected and analyzed with the SDG samples?

___ [___] ___

If yes, is the lowest concentration of that analyte in the associated samples less than 10 times the Preparation Blank value?

___ [___] ___

ACTION:

If yes, reject (R) and red-line all associated sample results greater than the CRQL but less than the Prep. Blank value. Flag as "J" detects $>$ Prep. Blank value but $<10 \times$ Prep. Blank. If the sample result \geq MDL but \leq CRQL, replace it with CRQL-U.

If the Prep. Blank value is less than the same analyte value in the Field Blank, do not qualify the sample results due to the Prep. Blank criteria.

NOTE:

Convert soil sample result to mg/Kg on wet weight basis to compare with the soil Prep. Blank result on Form III.

A.1.15.2.3 Is the Prep. Blank concentration below the negative CRQL?

___ [___] ___

ACTION:

If yes, flag (J) all associated sample results less than $10 \times$ CRQL. Qualify non-detects as estimated (UJ).

A.1.15.2.4 When the MDL is greater than the CRQL, is the preparation blank concentration on Form III greater than two times the MDL?

___ [___] ___

ACTION:

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YES NO N/A

If yes, reject (R) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.

A.1.16 ICP-AES/ICP-MS Interference Check Sample (ICS) - Form IV

NOTE: Not required for CN, Hg, Al, Ca, Fe and Mg.

A.1.16.1 Present and complete? [___] ___ ___

Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples? [___] ___ ___

Was ICS analyzed at the beginning of the ICP-MS analytical run? [___] ___ ___

ACTION:

If no, flag as estimated (J) all sample results.

A.1.16.2 ICP-AES Method

A.1.16.2.1 ICSA Solution:

For ICP-AES, are the ICSA "Found" analyte values within the control limits \pm of CRQL of the true/established mean value? [___] ___ ___

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV? [___] ___ ___

ACTION:

If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated only sample results \geq MDL

for which the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag non-detects as "UJ" and detects as "J".

A.1.16.2.3 **ICSAB Solution**

For ICP-AES, are all analyte results in ICSAB within the control limits of 80-120 of the true/established mean value?

[__] __ __

If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSAB Solution on Form IV?

[__] __ __

ACTION:

If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated those associated sample results \geq MDL for which the ICSAB analyte recovery is greater than 120% but \leq 150%. If the ICSAB recovery falls within 50-79%, qualify sample results \geq MDL as "J" and non-detects as "UJ". Reject (R) and red-line all sample results (detects & non-detects) for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only positive results.

A.1.16.3 **ICP-MS Method**

A.1.16.3.1 **ICSA Solution:**

For ICP-MS, are the ICSA "Found" analyte values within the control limits of \pm CRQL of the true/established mean value?

[__] __ __

ACTION:

If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated only sample results \geq MDL if the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag the associated sample detects as "J" and non-detects as "UJ".

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YES NO N/A

A.1.16.3.3 ICSAB Solution

For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater?

[] — —

ACTION:

If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated those associated sample results \geq MDL for which the ICSAB analyte recovery is greater than 120% but \leq 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results \geq MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only detects (\geq MDL).

A.1.17 **Spiked Sample Recovery: Pre-Digestion/Pre-Distillation)-Form V A**
 Note: Not required for Ca, Mg, K, and Na (both matrices); Al and Fe (soil only)

A.1.17.1 Was Matrix Spike analysis performed:

For each matrix type?

[] — —

For each SDG?

[] — —

On one of the SDG samples?

[] — —

For each concentration range (i.e., low, med., high)?

[] — —

For each analytical Method (ICP-AES, ICP-MS, Hg, CN) used?

[] — —

Was a spiked sample prepared and analyzed with the SDG samples?

[] — —

ACTION:

If no for any of the above, flag as estimated (J) all the positive data for which a spiked sample was not analyzed.

NOTE:

If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.

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A.1.17.2 Was a field blank or PE sample used
 for the spiked sample analysis?

___ [___] ___

ACTION:

If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.

A.1.17.3 Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.

Are all recoveries within the control limits when sample concentrations are less than or equal to four times the spike concentrations?

[___] ___ ___

NOTE:

Disregard the out of control spike recoveries for analytes whose concentrations are greater than or equal to four times the spike added.

Are results outside the control limits (75-125%) flagged with Lab Qualifier "N" on Form I's and Form VA?

[___] ___ ___

ACTION:

If no for any of the above, write in the Contract - Problems/Non-Compliance Section of the Data Review Narrative.

A.1.17.4 Aqueous

Are any spike recoveries:

(a) less than 30%?

___ [___] ___

(b) between 30-74%?

___ [___] ___

(c) between 126-150%?

___ [___] ___

(d) greater than 150%?

___ [___] ___

ACTION:

If the matrix spike recovery is less than 30%, reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data \geq MDL as "J" and non-detects

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YES NO N/A

as "UJ". If between 126-150%, flag (J)
 all data \geq MDL as "J". If greater than 150%,
 reject (R) and red-line all associated data \geq MDL.

(NOTE: Replace "N" with "J", "R" as appropriate.)

A.1.17.5 Soil/Sediment

Are any spike recoveries:

- | | | | |
|------------------------|-----|-------|-----|
| (a) less than 10%? | ___ | [___] | ___ |
| (b) between 10-74%? | ___ | [___] | ___ |
| (c) between 126-200%? | ___ | [___] | ___ |
| (d) greater than 200%? | ___ | [___] | ___ |

ACTION:

If yes for any of the above, proceed
 as follows:

If the matrix spike recovery is less
 than 10%, reject (R) and red-line all
 associated data (detects & non-detects);
 if between 10-74%, qualify all associated
 data \geq MDL as "J" and non-detects as "UJ";
 if between 126-200%, flag (J) all associated
 data \geq MDL as "J" If greater than 200%, reject
 (R) and red-line all associated data \geq MDL.
 (NOTE: Replace "N" with "J" or "R" as appropriate.)

A.1.18 Lab Duplicates) - Form VI

A.1.18.1 Was the lab duplicate analysis performed:

- | | | | |
|--|-------|-----|-----|
| For each SDG? | [___] | ___ | ___ |
| On one of the SDG samples? | [___] | ___ | ___ |
| For each matrix type? | [___] | ___ | ___ |
| For each concentration range
(low or med.)? | [___] | ___ | ___ |
| For each analytical Method
(ICP-AES/ICP-MS, Hg, CN) Used? | [___] | ___ | ___ |
| Was a lab duplicate prepared and
analyzed with the SDG samples? | [___] | ___ | ___ |

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YES NO N/A

ACTION:

If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.

NOTE:

If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

A.1.18.2 Was a Field Blank or PE sample used for the Lab Duplicate analysis?

ACTION:

If yes, flag as estimated (J) all SDG sample results (hits & non-detects) for which Field Blank or PE sample was used for duplicate analysis.

A.1.18.3 Circle on each Form VI all values that are:

RPD > 20%, or

Absolute Difference > CRQL

Are all values within control limits (RPD \leq 20% or absolute difference \leq \pm CRQL)?

If no, are all results outside the control limits flagged with an "*" (Lab Qualifier) on Form VI and on all Form I's?

ACTION:

If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.

NOTE:

The laboratory is not required to report on Form VI the RPD when both values are non-detects.

A.1.18.4 **Aqueous**

A.1.18.4.1 When sample and duplicate values are both \geq 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

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	<u>YES</u>	<u>NO</u>	<u>N/A</u>
is any RPD > 20% but < 100%?	___	[___]	___
is any RPD ≥ 100%?	___	[___]	___

ACTION:

If the RPD is > 20% but < 100%, flag (J) as estimated the associated sample data ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line the associated sample data ≥ CRQL.

(NOTE: Replace "*" with "J" or "R" as appropriate.)

A.1.18.4.2 When the sample and/or duplicate value < 5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate values:

> ± CRQL?	___	[___]	___
> ± 2xCRQL?	___	[___]	___

ACTION:

If the absolute difference is > CRQL, flag as estimated all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 2xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but < 5xCRQL.

NOTE:

1. Replace "*" with "J", "UJ" or "R" as appropriate.)
2. If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.18.5 **Soil/Sediment**

A.1.18.5.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%?	___	[___]	___
is any RPD ≥ 120%?	___	[___]	___

ACTION:

If the RPD is ≥ 35% and < 120%, flag (J) as estimated the associated sample

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QC criteria stated in Sections A.1.19.2 and A.1.19.3.

NOTE:

1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this the criteria to qualify the results.

A.1.19.2 Circle all values on the Form (Appendix A.4) for Field Duplicates that have:

RPD \geq 20% or

Difference $> \pm$ CRQL

When sample and duplicate values are both $\geq 5 \times$ CRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD \geq 20%?

is any RPD \geq 100%?

ACTION:

If the RPD is >20% but < 100%, flag (J) only the associated sample and its Field Duplicate results \geq CRQL. If the RPD is \geq 100%, reject (R) and red-line only the associated sample and its Field Duplicate result \geq CRQL.

A.1.19.3 When the sample and/or duplicate value(s) $< 5 \times$ CRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:

$> \pm$ CRQL?

$> \pm 2 \times$ CRQL?

ACTION:

If the absolute difference is $> CRQL$, flag detects $\geq MDL$ but $< 5 \times CRQL$ as "J" and non-detects as "UJ". If the difference is $> 2 \times CRQL$, reject (R) and red-line non-detects

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YES NO N/A

and results \geq MDL but $< 5 \times \text{CRQL}$ of the sample and its Field Duplicate.

Soil/Sediment Field Duplicates

A.1.19.4 Was a soil field duplicate pair collected and analyzed?
 (Check Sampling Trip Report) [] — —

ACTION:

If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I's. Calculate and report RPD when sample and its duplicate values are both greater than $5 \times \text{CRQL}$. Calculate and report the absolute difference when at least one value (sample or duplicate) is $< 5 \times \text{CRQL}$. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

NOTE:

1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when $\text{MDL} > \text{CRQL}$.
4. If one value is $> \text{CRQL}$ and the other value is non-detect, calculate the absolute difference between the value $> \text{CRQL}$ and the MDL, and apply the criteria to qualify the results.

A.1.19.5 Circle on each Appendix A.4 all values that have:

RPD $\geq 35\%$, or Difference $> \pm 2 \times \text{CRQL}$
 When sample and duplicate values are both $\geq 5 \times \text{CRQL}$ (substitute MDL for CRQL when $\text{MDL} > \text{CRQL}$),

is any RPD $\geq 35\%$ but $< 120\%$? — [] —

is any RPD $\geq 120\%$? — [] —

ACTION:

If the RPD is $\geq 35\%$ but $< 120\%$,

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YES NO N/A

flag only the associated sample and its Field Duplicate results \geq CRQL as "J". If the RPD is \geq 120%, reject (R) and red-line only the sample and its Field Duplicate results \geq CRQL.

A.1.19.6 When the sample and/or duplicate value(s) $< 5 \times$ CRQL (substitute MDL for CRQL when MDL $>$ CRQL), is the absolute difference between sample and Field Duplicate:

> $\pm 2 \times$ CRQL?	___	[___]	___
> $\pm 4 \times$ CRQL?	___	[___]	___

ACTION:

If the absolute difference is $> 2 \times$ CRQL, flag Sample and its Field Duplicate results \geq MDL but $< 5 \times$ CRQL as "J" and non-detects as "UJ". If the difference is $> 4 \times$ CRQL, reject (R) and red-line non-detects and detects \geq MDL but $< 5 \times$ CRQL of the sample and its Field Duplicate.

A.1.20 **Laboratory Control Sample (LCS) - Form VII**

A.1.20.1 Was one LCS prepared and analyzed for:

Each SDG?	[___]	___	___
Each matrix type?	[___]	___	___
Each batch samples digested/distilled?	[___]	___	___
For each Method (ICP-AES, ICP-MS, Hg, CN) used?	[___]	___	___
Was an LCS prepared and analyzed with the samples?	[___]	___	___

ACTION:

If no for any of the above, prepare Telephone Record Log and contact CLP PO or TOPO for submittal of the LCS results. Flag (J) as estimated all the data for which an LCS was not analyzed.

NOTE:

If only one LCS was analyzed for

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YES NO N/A

more than 20 samples, then the first 20 samples analyzed are not flagged(J), but all additional samples must be qualified (J).

A.1.20.2 Aqueous LCS

Circle on each Form VII the LCS percent recoveries outside control limits 80-120%.

NOTE: 1. Use digested ICV as LCS for aqueous mercury
 2. Use distilled ICV as LCS for aqueous cyanide

Is any LCS recovery:

Less than 50%?	___	[]	___
Between 50% and 79%?	___	[]	___
Between 121% and 150%?	___	[]	___
Greater than 150%?	___	[]	___

ACTION:

If the LCS recovery is less than 50%, reject (R) and red-line all associated sample data (detects & non-detects); for a recovery between 50-79%, flag detects as "J" all non-detects as "UJ". if the LCS recovery is between 121-150%, flag only detects as "J". if the recovery is greater than 150%, reject (R) and red-line all detects.

A.1.20.3 Solid LCS

If an analyte's MDL is equal to or greater than the true value of LCS, disregard the "Action" below for that analyte even though the LCS is out of control limits.

Is the LCS "Found" value greater than the Upper Control Limit reported on Form VII?

___ [] ___

ACTION:

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If yes, flag (J) all the associated detects \geq MDL as estimated (J).

YES NO N/A

Is the LCS "Found" value lower than the Lower Control Limit reported on Form VII?

___ [___] ___

ACTION:

If yes, flag detects as "J" and non-detects as "UJ".

A.1.21 **ICP-AES/ICP-MS Serial Dilution - Form VIII**

NOTE: Serial dilution analysis is required only when the initial concentration is equal to or greater than 50 x MDL.

A.1.21.1 Was a Serial Dilution analysis performed:

For each SDG?

[___] ___ ___

On one of the SDG samples?

[___] ___ ___

For each matrix type?

[___] ___ ___

For each concentration range (low or med.)?

[___] ___ ___

Was a Serial Dilution sample analyzed with the SDG samples?

[___] ___ ___

ACTION:

If no for any of the above, flag as estimated (J) detects \geq MDL of all the SDG samples for which the ICP Serial Dilution Analysis was not performed.

A.1.21.2 Was a Field Blank or PE sample used for the Serial Dilution Analysis?

___ [___] ___

ACTION:

If yes, flag as estimated (J) detects \geq MDL of all the SDG samples

A.1.21.3 Circle on Form VIII the Percent Differences (%D) between sample results and its dilution results that are outside the control limits $\pm 10\%$

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when initial concentrations \geq 50 x MDLs.

Are results outside the control limits flagged with an "E" (Lab Qualifier) on Form VIII and all Form I's?

YES NO N/A

[] — —

ACTION:

If no, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.21.4 Are any %D values:

> 10%?

— [] —

\geq 100%?

— [] —

ACTION:

If the Percent Difference (%D) is greater than 10%, flag (J) as estimated all associated samples whose raw data \geq MDL; if the %D is \geq 100%, reject (R) and red-line all associated samples with raw data \geq MDL.

(NOTE: Replace "E" with "J" or "R" as appropriate.)

A.1.22 Total/Dissolved or Inorganic/Total Analytes

A.1.22.1 Were any analyses performed for dissolved as well as total analytes on the same sample(s)?
 Were any analyses performed for inorganic as well as total analytes on the same sample(s)?

— [] —

— [] —

ACTION:

If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic) and total analyte concentrations. Compute each difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled:

- (1) The dissolved (or inorganic) concentration is greater than total concentration, and
- (2) greater than or equal to 5xMDL.

A.1.22.2 Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20%?

— [] —

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YES NO N/A

A.1.22.3 Is any dissolved(or inorganic) concentration greater than its total concentration by more than 50%? []

ACTION:

If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.

A.1.23 **Field Blank - Form I**
NOTE: Designate "Field Blank" as such on Form I

A.1.23.1 Was a Field/Rinsate Bank collected and analyzed with the SDG samples? []

If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL(or 2xMDL when MDL>CRQL)? []

If yes, circle the Field Blank value on Form I that is greater than the CRQL, (or 2 x MDL when MDL > CRQL).

Is any Field Blank value greater than CRQL also greater than the Preparation Blank value? []

If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria? []

ACTION:

If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results) greater than the CRQL but less than the Field Blank value. Reject on Form I's the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value. If the sample result \geq MDL but \leq CRQL, replace it with CRQL-U.

If the Field Blank value is less than the

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YES NO N/A

Prep.Blank value, do not qualify the sample results due to the Field Blank criteria.

NOTE:

1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.

A.1.24 Verification of Instrumental Parameters - Form IX, XA, XB, XI

A.1.24.1 Is verification report present for:

Method Detection Limits (Form IX-Annually)?	[___]	___	___
ICP-AES Interelement Correction Factors (Form XA & XB -Quarterly)?	[___]	___	___
ICP-AES & ICP-MS Linear Ranges (Form XI-Quarterly)?	[___]	___	___

ACTION:

If no, contact CLP PO/TOPO for submittal from the laboratory.

A.1.24.2 Method Detection Limits - Form IX

A.1.24.2.1 Are MDLs present on Form IX for:

All the analytes?	[___]	___	___
All the instruments used?	[___]	___	___
Digested and undigested samples and Calib.Blanks?	[___]	___	___
ICP-AES and ICP-MS when both instruments are used for the same analyte?	[___]	___	___

ACTION:

If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/ Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than ½ CRQL.

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		<u>YES</u>	<u>NO</u>	<u>N/A</u>
A.1.24.2.2	Is MDL greater than the CRQL for any analyte?	___	[___]	___
	If yes, is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?	[___]	___	___
	<u>ACTION:</u> If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.			
A.1.24.3	<u>Linear Ranges - Form XI</u>			
A.1.24.3.1	Was any sample result higher than the high linear range for ICP-AES or ICP-MS?	___	[___]	___
	Was any sample result higher than the highest calibration standard for mercury or cyanide?	___	[___]	___
	If yes for any of the above, was the sample diluted to obtain the result reported on Form I?	[___]	___	___
	<u>ACTION:</u> If no, flag (J) as estimated the affected detects (\geq MDL) reported on Form I.			
A.1.25	<u>ICP-MS Tune Analysis - Form XIV</u>			
A.1.25.1	Was the ICP-MS instrument tuned prior to calibration?	[___]	___	___
	<u>ACTION:</u> If no, reject (R) and red-line all sample data for which tuning was not performed.			
A.1.25.2	Was the tuning solution analyzed or scanned at least five times consecutively?	[___]	___	___
	Were all the required isotopes spanning the analytical range present in the tuning solution?	[___]	___	___
	Was the mass resolution within			

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YES NO N/A

0.1 amu for each isotope in the tuning solution?

[] — —

Was %RSD less than 5% for each isotope of each analyte in the tuning solution?

[] — —

ACTION:

If no for any of the above, qualify all results \geq MDL associated with that Tune as estimated "J", and all non-detects associated with that Tune as "UJ".

A.1.26 ICP-MS Internal Standards - Form XV

A.1.26.1 Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)?

[] — —

Were all the target analyte masses bracketed by the masses of the five internal standards?

[] — —

ACTION:

If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.

A.1.26.2 Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank?

[] — —

If no, was the original sample diluted two fold, Internal Standard added and the sample re-analyzed?

[] — —

Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)?

[] — —

ACTION:

If no for any of the above, flag detects as "J" and non-detects "UJ" of all the analytes with atomic masses between the atomic mass of the internal standard lighter

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than the affected internal standard, and the atomic mass of the internal standard heavier than the affected internal standard.

A.1.27 Percent Solids of Sediments

A.1.27.1 Are percent solids in sediment(s):

< 50%? _____ [] _____

ACTION:

If yes, qualify as estimated (J) all detects and non-detects of a sample that has percent solids less than 50% (i.e., moisture content greater than 50%).

NOTE:

Flag(J) only the sample results that were not previously flagged due to other QC criteria.

Inorganic Data Review Narrative

Case# _____ Site: _____ Matrix: Soil _____
SDG# _____ Lab: _____ Water _____
Sampling Team: _____ Reviewer: _____ Other _____

A.2.1 Data Validation Flags:

The following flags may have been applied in red by the data validator and must be considered by the data user.

- J - This flag indicates the result qualified as estimated
- R and Red-Line - A red-line drawn through a sample result indicates unusable value. The red-lined data are known to contain significant errors based on documented information and must not be used by the data user.
- U - This data validation qualifier is applied to sample results \geq MDL when associated blank is contaminated
- Fully Usable Data - The results that do not carry "J" or "red-line" are fully usable.

A.2.2 Laboratory Qualifiers:

The CLP laboratory applies a contractual qualifier on all

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Form I'S and the QC Form when a QC analysis is outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets. These qualifiers and their meanings are as follows:

N: This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.

E: This qualifier indicates the the presence of interference, and is applied when the ICP serial dilution is outside the control limits.

*: This qualifier indicate the lack of precision , and is pplied on Fom I'S and Form VI when the Lab Duplicate analysis is outside the control limits.

U: This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analyte is indicated by the Contract Required Quantitation Limit (CRQL) of that analyte suffixed with "U".

J: This is also a concentration qualifier that laboratory applies to a positive result below the CRQL.

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

A.2.3.1 Data Case Description:

A.2.3.2 CSF Audit:

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A.2.3.3 Technical Review:

A.2.3.4 Contract-Problem/Non-Compliance:

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HWSS Reviewer: _____ Date: _____
Signature

Contractor Reviewer: _____ Date: _____
Signature

Verified by: _____ Date: _____
Signature

Contract Laboratory Program
REGION II/LABORATORY COMMUNICATION SYSTEM

Telephone Record Log

CASE #
SDG #

Date of Call: _____

ESAT Reviewer/Date: _____

Type of Analysis: Inorganic

Laboratory Name: _____

Lab Contact: _____

Call Initiated By: Laboratory X Region II

Inquiry made in reference to data for the following sample number(s):

Summary of Questions/Issues Discussed:

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YES NO N/A

Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Total/Dissolved Concentrations

Lab Code Case No. SDG No. Sample Matrix: Water

Concentration: ug/L

ANALYTE	TOTAL	C	DISSOLVED	C	DIFFERENCE	Q	M
ALUMINUM							
ARSENIC							
BARIUM							
BERYLLIUM							
CADMIUM							
CALCIUM							
CHROMIUM							
COBALT							
COPPER							
IRON							
LEAD							
MAGNESIUM							
MAGNESE							
MERCURY							

NICKEL						
POTASSIUM						
SELENIUM						
SILVER						
SODIUM						
THALLIUM						
VANADIUM						
ZINC						
CYANIDE						

**CONTRACT LABORATORY PROGRAM
CLP RAS RE-ANALYSIS REQUEST/APPROVAL RECORD**

SECTION A (TO BE COMPLETED BY REGIONAL SENDING OFFICIAL)

Initiated By:

Name, Affiliation, Phone Number

Case Number: _____

- OLM
- OLC
- ILM

Details of Re-Analysis Request:

● Laboratory Name /Contract Number: _____

● Affected Sample Number(s) and Fraction(s): _____

● Reason for Re-Analysis: _____

● Contract Statement of Work Citation*: _____

● Comments: _____

● * PROVIDE SOW CITATION THAT SUPPORTS THIS REQUEST

RE-ANALYSIS Billable () Not Billable ()

● Approved By: _____
Authorized Regional Sending CLP PO Signature

Date: _____

SECTION B (TO BE COMPLETED BY SMO)

Name of SMO Contact _____

Date: _____

Date of Laboratory Notification (Verbal): _____

Re-analysis Start Date: _____

Data Due Date: _____

Return completed form to:
Sample Management Office (SMO)

Distribution: (1) CLP PO Copy (2) Regional Sending Official Copy (3) SMO File Copy (4) Laboratory Copy
Final 9/3/99

CLP DATA ASSESSMENT SUMMARY FORM (INORGANICS)

Type of Review: _____ Date: _____ Case# _____ SDG# _____

Site: _____ Lab Name: _____

Reviewer's Initials: _____ Number of Samples: _____

Analytes Rejected (R) Due to Exceeding Review Criteria

	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup. Lab.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	Tuning ICP-MS	Total Analytes	Rejection %
ICP-AES														
ICP-MS														
Mercury														
Cyanide														
Total														

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YES NO N/A