NATIONAL FUNCTIONAL GUIDELINES
for Inorganic Superfund Data Review

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This document can be obtained from the EPA's Contract Laboratory Program (CLP) website at:
http://www.epa.gov/superfund/programs/clp/guidance.htm

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<u>**ACRONYMS AND ABBREVIATIONS**</u>

I. Terminology

The following acronyms and abbreviations may be found throughout this document. For further definition, see Appendix A: Glossary at the end of the document.

CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
COR	Contracting Officer Representative
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
DF	Dilution Factor
DQO	Data Quality Objective
EDM	EXES Data Manager
EPA	United States Environmental Protection Agency
EXES	Electronic Data Exchange and Evaluation System
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
LEB	Leachate Extraction Blank
MDL	Method Detection Limit
NFG	National Functional Guidelines
%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%RSD	Percent Relative Standard Deviation
%Solids	Percent Solids
PE	Performance Evaluation
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference

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
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TR/COC	Traffic Report/Chain of Custody
TSS	Total Suspended Solids

II. Target Analyte List

The EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Inorganic Superfund Methods (Multi-Media, Multi-Concentration) ISM02.2 applies CLP analytical methods for the isolation, detection, and quantitation of the following target analytes and parameter:

Al	Aluminum
Sb	Antimony
As	Arsenic
Ba	Barium
Be	Beryllium
Cd	Cadmium
Ca	Calcium
Cr	Chromium
Co	Cobalt
Cu	Copper
CN	Cyanide
Fe	Iron
Pb	Lead
Mg	Magnesium
Mn	Manganese
Hg	Mercury
Ni	Nickel
K	Potassium
Se	Selenium
Ag	Silver
Na	Sodium
Tl	Thallium
V	Vanadium
Zn	Zinc
Hardness	

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INTRODUCTION

I. Purpose of Document

This document contains guidance to aid the data reviewer in determining the usability of analytical data generated using the United States Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) Statement of Work (SOW) for Inorganic Superfund Methods (Multi-Media, Multi-Concentration) ISM02.2. The SOW includes metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES), metals by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), mercury, and cyanide analytical methods.

The guidelines presented in this document are designed to assist the reviewer in evaluating (a) whether the analytical data meet the technical and Quality Control (QC) criteria specified in the SOW, and (b) the usability and extent of bias of any data that do not meet these criteria. This document contains definitive guidance in areas, such as blanks, calibration verification standards, Interference Check Samples (ICSs), QC audit samples, and instrument performance checks (e.g., tuning), in which performance is fully under a laboratory's control. General guidance is provided to aid the reviewer in making subjective judgments regarding the use of data that are affected by site conditions (e.g., sample matrix effects) and do not meet SOW-specified requirements.

II. Limitations of Use

This guidance is specific to the review of analytical data generated using CLP SOW ISM02.2. It applies to the current version of the SOW, as well as future versions that contain editorial changes. To use this document effectively, the reviewer should have an understanding of the analytical methods and a general overview of the Sample Delivery Group (SDG) or Case at hand. This guidance is not appropriate for use in conducting contract compliance reviews and should be used with caution in reviewing data generated using methods other than CLP SOW ISM02.2, although the general types of QC checks, the evaluation procedures, and the decisions made after consideration of the evaluation criteria may be applicable to data from any similar method.

While this document is a valuable aid in the data review process, other sources of guidance and information, along with professional judgment, are useful in determining the ultimate usability of the data. This is particularly critical in those cases where data do not meet SOW-specified technical and QC criteria. To make the appropriate judgments, the reviewer needs to gain a complete understanding of the intended use of the data and is strongly encouraged to establish a dialogue with the data user prior to and following data review, to discuss usability issues and resolve questions regarding the review.

III. Document Organization

Following this introduction, the document is presented in two major parts: Part A – General Data Review, which applies to all methods; and Part B – Method-Specific Data Review. In Part B, each method is addressed individually in a stand-alone format. A complete list of acronyms used in this document appears preceding this introduction, and a Glossary is appended as Appendix A.

IV. For Additional Information

For additional information regarding the CLP and the services it provides, refer to the CLP website at http://www.epa.gov/superfund/programs/clp/index.htm

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PART A: GENERAL DATA REVIEW

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I. Preliminary Review

A preliminary review should be performed on the data, prior to embarking on the method-specific review (see Part B). During this process, the reviewer should compile the necessary data package elements to ensure that all of the information needed to determine data usability is available. The preliminary review also allows the reviewer to obtain an overview of the Case or Sample Delivery Group (SDG) under review.

The initial review should include, but is not limited to, verification of the exact number of samples, their assigned number and matrices, and the Contractor laboratory name. It should take into consideration all the documentation specific to the sample data package, which may include Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) Record, SDG Narrative, and other applicable documents.

The reviewer should be aware that minor modifications to the Statement of Work (SOW) that have been made through a Modified Analysis request, to meet site-specific requirements, could affect certain validation criteria such as Contract Required Quantitation Limits (CRQLs) and Target Analyte Lists (TALs). Therefore, these modifications should be applied during the method-specific review (Part B) process.

The Cases or SDGs routinely have unique field quality control (QC) samples that may affect the outcome of the review. These include field blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The reviewer should verify that the following items are identified in the sampling records (e.g., TR/COC Records, field logs, and/or contractor tables):

1. The United States Environmental Protection Agency (EPA) Region where the samples were collected; and

2. The complete list of samples with information on:

    a. Sample matrix

    b. Field blanks (if applicable)

    c. Field duplicates (if applicable)

    d. Field spikes (if applicable)

    e. PE samples (if applicable)

    f. Sampling dates

    g. Sampling times

    h. Shipping dates

    i. Preservatives

    j. Types of analysis

    k. Contractor laboratory

The laboratory's SDG Narrative is another source of general information which includes notable problems with matrices; insufficient sample volume for analysis or reanalysis; samples received in broken containers; preservation information; and unusual events. The reviewer should also inspect any email or telephone/communication logs in the data package detailing any discussion of sample and/or analysis issues between the laboratory, the Contract Laboratory Program (CLP) Sample Management Office (SMO), and the EPA Region.

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP), or similar document, for the project for which the samples were analyzed, to assist in the determination of final usability of the analytical data. The reviewer should contact the appropriate Regional Laboratory

Contracting Officer Representative (COR) to obtain copies of the QAPP and relevant site information.

For data obtained through the CLP, the Staged Electronic Data Deliverable (SEDD) generated by the CLP laboratories is subjected to the following reviews via the Electronic Data Exchange and Evaluation System (EXES): 1) automated data assessment for Contract Compliance Screening (CCS) based on the technical and QC criteria in CLP SOW ISM02.2, and 2) automated data validation based on the criteria in the *EPA CLP National Functional Guidelines (NFG) for Inorganic Superfund Data Review*. In addition, completeness checks are manually performed on the hardcopy data. The automated CCS results and hardcopy data issues are subsequently included in a CCS defect report that is provided to the laboratory. The laboratory may then submit a reconciliation package for any missing items or to correct non-compliant data identified in the report. The automated data validation results are summarized in criteria-based NFG reports that are provided to the EPA Regions. The data reviewer can access the CCS and NFG reports through the EXES Data Manager (EDM) via the SMO Portal and may use them in determining data usability.

For more information about EXES and EDM, refer to the following CLP website:

http://www.epa.gov/superfund/programs/clp/data_assessment.htm

For access to the SMO Portal, refer to the following CLP website to contact the Regional Laboratory COR from the Region where the data review is being performed and to obtain the necessary username and password information:

http://www.epa.gov/superfund/programs/clp/contacts.htm

For concerns or questions regarding the data package, contact the Regional Laboratory COR from the Region where the samples were collected.

II. Data Qualifier Definitions

The following definitions provide brief explanations of the national qualifiers assigned to results during the data review process. The reviewer should use these qualifiers as applicable. If the reviewer chooses to use additional qualifiers, a complete explanation of those qualifiers shall accompany the data review.

Table 1. Data Qualifiers and Definitions

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

III. Data Review Narrative

The reviewer should complete a Data Review Narrative that includes comments that address the problems identified during the review process and state the limitations of the data associated with a Case or SDG. The CLP Sample Numbers, analytical methods, extent of the problem(s), and assigned qualifiers should also be listed in the document.

The Data Review Narrative, including the Inorganic Data Review Summary form (see Appendix B), must be provided together with the laboratory data to the appropriate recipient(s). A copy of the Data Review Narrative should also be submitted to the Regional Laboratory COR assigned oversight responsibility for the Contractor laboratory.

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PART B: METHOD-SPECIFIC DATA REVIEW

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ICP-AES DATA REVIEW

The inorganic data requirements for Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) to be reviewed during validation are listed below:

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Example Analytical Sequence

This is an example of an analytical sequence:

S##
S##
S##
S##
S##
S##
ICV
ICB
ICSA
ICSAB
CCV###
CCB###
samples
CCV###
CCB###
samples
CCV###
CCB###, etc.

* Suffix ## and ### are as specified in Exhibit B of the Statement of Work (SOW).

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I. <u>**Preservation and Holding Times**</u>

A. Review Items

Form 1-IN, Form 12-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; shipping container temperature; holding time; and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on the sample conditions and the holding time of the sample.

C. Criteria

1. The technical holding time is determined from the date of collection, or the date Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction is complete, to the date of analysis.

2. The technical holding time criteria for aqueous/water samples and leachate samples from TCLP or SPLP is 180 days, preserved (with nitric acid) to pH ≤ 2.

3. The technical holding time criteria for soil/sediment samples is 180 days, based on the technical holding time criteria for aqueous/water samples.

4. The technical holding time criteria for wipe samples is 180 days, based on the technical holding time criteria for aqueous/water samples.

5. Soil/sediment samples shall be maintained at $\leq 6°C$ (but not frozen) from the time of collection until receipt at the laboratory. All aqueous/water and soil/sediment samples must be stored at $\leq 6°C$ (but not frozen) from the time of sample receipt until digestion. The TCLP and SPLP leachates must be stored at $\leq 6°C$ (but not frozen) from the time of the leaching procedure completion until digestion. Wipe samples should be maintained at room temperature until preparation.

D. Evaluation

Establish technical holding times by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form 12-IN and the raw data; also consider using information in the Complete SDG File (CSF), as it may be helpful in the assessment. Verify that the analysis dates on the Form 12-IN and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication of problems with the samples, the sample integrity may be compromised. Use professional judgment to evaluate the effect of the problem on the sample results.

E. Action

NOTE: Apply the action to each field sample for which the preservation or holding time criteria was not met.

1. If the pH of aqueous/water samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH of the sample to ≤ 2 at the time of sample receipt. Also determine if the laboratory adjusted the pH to ≤ 2 for the TCLP and SPLP leachates after completion of the leaching procedure. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

2. If soil/sediment samples are not maintained at $\leq 6°C$ (but not frozen) from the time of collection until receipt at the laboratory, detects should be qualified as estimated low (J-) and non-detects as unusable (R).

3. If technical holding times are exceeded, use professional judgment to determine the reliability of the data, based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

4. Due to limited information concerning holding times for soil/sediment and wipe samples, use discretion when deciding whether to apply the aqueous/water holding time criteria to soil/sediment and wipe samples. If they are applied, document this action in the Data Review Narrative.

5. If aqueous/water and soil/sediment samples are received with shipping container temperatures > 10°C, use professional judgment to determine the reliability of the data, or qualify detects as estimated (J) and non-detects as estimated (UJ).

6. When the holding times are exceeded, annotate any possible consequences for the analytical results in the Data Review Narrative, and note it for Regional Laboratory Contracting Officer Representative (COR) action.

Table 2. Preservation and Holding Time Actions for ICP-AES Analysis

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with pH > 2 and pH not adjusted	Use professional judgment J-	Use professional judgment R
TCLP/SPLP leachate samples with pH >2 and pH not adjusted	Use professional judgment J-	Use professional judgment R
Soil/sediment samples not maintained at ≤ 6°C (but not frozen) from time of collection until receipt at the laboratory	Use professional judgment J-	Use professional judgment R
Technical Holding Time: Aqueous/water and TCLP/SPLP leachate samples > 180 days	J-	R
Technical Holding Time: Soil/sediment and wipe samples > 180 days	J-	R
Samples received > 10ºC*	Use professional judgment J	Use professional judgment UJ

*   For samples received with shipping container temperatures > 10ºC, Regional policy or project Data Quality Objectives (DQO) may allow the use of higher temperature criteria before assessing any actions for the affected samples.

II.   Calibration

A. Review Items

Form 2-IN, Form 12-IN, Form 15-IN, Form 16-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on initial calibration and calibration verification.

C. Criteria

1. Initial Calibration

    The instruments shall be successfully calibrated each time the instrument is set up and after Continuing Calibration Verification (CCV) failure. The calibration date and time shall be included in the raw data.

    a. A blank and at least five calibration standards shall be used to establish each calibration curve. At least one of these standards shall be at or below the Contract Required Quantitation Limit (CRQL) but above the Method Detection Limit (MDL). All measurements shall be within the instrument working range where the interelement correction factors are valid. A minimum of three replicate exposures are required for standardization, for all Quality Control (QC), and sample analyses. The average result of all the multiple exposures for the standardization, QC, and sample analyses shall be used. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient ≥ 0.995. The calculated percent differences (%Ds) for all of the non-zero standards must be within $\pm30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

2. Initial and Continuing Calibration Verification

    The acceptance criteria for the Initial Calibration Verification (ICV) and CCV standards are presented in Table 3:

Table 3. Acceptance Criteria for ICV and CCV Standards for ICP-AES Analysis

Analytical Method	Inorganic Analytes	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
ICP-AES	Metals	90	110

    a. Initial Calibration Verification

        1) Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). If the ICV Percent Recovery (%R) falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

        2) Only if the ICV is not available from the United States Environmental Protection Agency (EPA), analyses shall be conducted using a certified solution of the analytes from an independent commercial standard source, at a concentration level other than that used for instrument calibration, but within the calibrated range.

        3) The ICV solution shall be analyzed at each analytical wavelength used for analysis.

    b. Continuing Calibration Verification

        1) To ensure accuracy during the course of each analytical sequence, the CCV shall be analyzed and reported for each wavelength used for the analysis of each analyte.

2) The CCV standard shall be analyzed at a frequency of every two hours during an analytical sequence. The CCV standard shall also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

3) The analyte concentration(s) in the CCV standard(s) shall be different than the concentration used for the ICV, and at a concentration equivalent to the mid-level of their respective calibration curves.

4) The same CCV standard solution shall be used throughout the analysis for an SDG.

5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation

1. Verify that the instrument was calibrated each time the instrument was set up, utilizing a blank and at least five calibration standards, one of which was at or below the CRQL but above the MDL.

2. Confirm that the measurements were within the working calibration range, and were the average result of at least three replicate exposures.

3. Verify that the ICV and CCV standards were analyzed for each analyte at the specified frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.

4. Recalculate one or more of the ICV and CCV %R(s) using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form 2-IN.

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

Where,

Found (value) = Concentration (in µg/L) of each analyte measured in the analysis of the ICV or CCV solution

True (value) = Concentration (in µg/L) of each analyte in the ICV or CCV source

E. Action

NOTES: For initial calibrations or ICV standards that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical sequence.

1. If the instrument was not calibrated each time the instrument was set up, qualify detects and non-detects as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and at least one at or below CRQL but above the MDL), use professional judgment to qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

2. If the correlation coefficient is < 0.995, the %Ds are outside the ±30% limit, or the y-intercept ≥ CRQL, qualify detects as estimated (J) and non-detects as estimated (UJ).

3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:

   a. If the ICV or CCV %R is < 75%, use professional judgment to qualify detects as estimated low (J-) or unusable (R), and non-detects as unusable (R).

    b.   If the ICV or CCV %R falls within the range of 75-89%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).

    c.   If the ICV or CCV %R falls within the range of 90-110%, detects and non-detects should not be qualified.

    d.   If the ICV or CCV %R falls within the range of 111-125%, qualify detects as estimated high (J+). Non-detects should not be qualified.

    e.   If the ICV or CCV %R is > 125%, use professional judgment to qualify detects as estimated high (J+) or unusable (R). Non-detects should not be qualified.

4.   If the laboratory failed to provide adequate calibration information, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory to request the necessary information. If the information is unavailable, use professional judgment to assess the data.

5.   Annotate the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.

6.   If calibration criteria are grossly exceeded, note this for Regional Laboratory COR action.

NOTE: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 4. Calibration Actions for ICP-AES Analysis

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed	R	R
Calibration incomplete	Use professional judgment J or R	Use professional judgment UJ or R
Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL	J	UJ
ICV/CCV %R < 75%	Use professional judgment J- or R	Use professional judgment R
ICV/CCV %R 75-89%	J-	UJ
ICV/CCV %R 90-110%	No qualification	No qualification
ICV/CCV %R 111-125%	J+	No qualification
ICV/CCV %R > 125%	Use professional judgment J+ or R	No qualification

III.   Blanks

A. Review Items

Form 1-IN, Form 3-IN, Form 12-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the blank responses by determining the existence and magnitude of contamination resulting from laboratory (or field) activities or baseline drift during analysis.

C. Criteria

1. No contaminants should be found in the blank(s).

2. The Initial Calibration Blank (ICB) shall be analyzed at each mass used for analysis after the analytical standards, but not before analysis of the ICV during the initial calibration of the instrument (see Section II.C.1).

3. A Continuing Calibration Blank (CCB) shall be analyzed at each wavelength used for the analysis, immediately after every CCV. The CCB shall be analyzed at a frequency of every two hours during the analytical sequence. The CCB shall be analyzed at the beginning of the analytical sequence, and again after the last CCV that was analyzed after the last analytical sample of the analytical sequence. The CCB result (absolute value) shall not exceed the CRQL of each analyte for which analysis is performed.

4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every SDG, or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water or a clean wipe processed through the appropriate sample preparation and analysis procedure.

5. If the concentration of any analyte in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with the analyte's concentration < 10x the Preparation Blank concentration and > CRQL, should be redigested and reanalyzed for that analyte. The laboratory is not to correct the sample concentration for the blank value.

6. If the concentration of any analyte in the Preparation Blank is < (-CRQL), all associated samples with the analyte's concentration < 10x the CRQL should be redigested and reanalyzed.

7. At least one Leachate Extraction Blank (LEB) shall be prepared and analyzed for each batch of samples extracted by TCLP or SPLP. The LEB consists of reagent water processed through the extraction procedure. Post-extraction, the LEB shall be processed through the appropriate sample preparation and analysis procedure.

D. Evaluation

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the specified frequency and sequence during the analysis, and Preparation Blanks were prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).

2. Review the results reported on Form 3-IN, as well as the raw data for all blanks, and verify that the results were accurately reported.

3. Evaluate all of the associated blanks for the presence of target analytes. Verify that if the concentration of any target analyte was > CRQL in a Preparation Blank, all associated samples with the analyte's concentration > CRQL but < 10x the Preparation Blank concentration were redigested and reanalyzed for that analyte. Verify that if a concentration was < (-CRQL) in a Preparation Blank, all associated samples with the analyte's concentration < 10x CRQL were

redigested and reanalyzed. Verify that if the absolute value of any target analytes was > CRQL in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

E. Action

NOTES: For ICBs that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCBs that do not meet the technical criteria, apply the action to all associated samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical sequence.

For Preparation Blanks that do not meet the technical criteria, apply the action to all associated samples prepared in the same preparation batch. For LEBs that do not meet the technical criteria, apply the action to all associated samples extracted in the same extraction batch.

1. If the appropriate blanks were not analyzed with the specified frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.

3. Some general "technical" review actions include:

a. For any blank (including Preparation Blanks and LEBs) reported with detects ≤ CRQLs, report detects ≤ CRQLs at the CRQLs and qualify as non-detect (U). For any blank (including Preparation Blanks and LEBs) reported with detects ≤ CRQLs, use professional judgment to qualify the sample results > CRQLs. Non-detects should not be qualified.

b. For any blank (including Preparation Blanks and LEBs) reported with a negative result, ≤ (-MDL) but ≥ (-CRQL), carefully evaluate and determine its effect on the sample data. Use professional judgment to assess the data.

c. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form 1-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form 3-IN. It may be easier to work with the raw data and/or convert the ICB or CCB results to the same units as the soil/sediment samples for comparison purposes.

4. Specific "method" actions include:

a. If an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, non-detects should not be qualified. Report detects ≤ CRQLs at the CRQLs and qualify as non-detect (U). Report sample results that are > CRQLs but < ICB/CCB Results at ICB/CCB Results and use professional judgment to qualify as non-detect (U) or unusable (R). Use professional judgment to qualify sample results ≥ ICB/CCB Results. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

b. If an ICB or a CCB result is < (-CRQL), the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, use professional judgment to qualify non-detects as estimated (UJ) or unusable (R). Use professional judgment to qualify detects ≤ CRQL, or qualify as estimated low (J-). Use professional judgment to qualify sample results that are > CRQLs as estimated low (J-).

c. If the concentration of any analyte in the Preparation Blank/LEB is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10x the Preparation Blank/LEB concentration. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and > CRQL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, report the sample results at Preparation Blank Results; use professional judgment to qualify the results as estimated high (J+) or unusable (R). Report results < 10x the LEB concentration and > CRQL in the samples associated with the LEB at LEB Results; use professional judgment to qualify the results as estimated high (J+) or unusable (R). Report detects ≤ CRQLs in the samples associated with the Preparation Blank/LEB at CRQLs and qualify as non-detect (U). Non-detects and sample results that are ≥ 10x the Preparation Blank/LEB Results should not be qualified. If the laboratory failed to redigest and reanalyze the samples associated with the Preparation Blank, record it in the Data Review Narrative, and note it for Regional Laboratory COR action.

d. For any Preparation Blank or LEB reported with a negative result, < (-CRQL), use professional judgment to qualify detects ≤ CRQL, or qualify as estimated low (J-). Qualify sample results that are ≥ CRQLs as estimated low (J-), and qualify non-detects as estimated (UJ). Sample results that are ≥ 10x CRQLs should not be qualified.

Table 5. Blank Actions for ICP-AES Analysis

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Detect ≤ CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	Detect or non-detect	Use professional judgment
ICB/CCB	> CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL but < ICB/CCB Result	Report at ICB/CCB Result and qualify as non-detect (U) or unusable (R)
		≥ ICB/CCB Result	Use professional judgment
ICB/CCB	< (-CRQL)	Non-detect	Use professional judgment to qualify as estimated (UJ) or unusable (R)
		Detect ≤ CRQL	Use professional judgment or (J-)
		> CRQL	Use professional judgment to qualify as estimated low (J-)

Blank Type	Blank Result	Sample Result	Action
Preparation Blank/LEB	Detect ≤ CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
Preparation Blank/LEB	≤ (-MDL) but ≥ (-CRQL)	Detect or non-detect	Use professional judgment
Preparation Blank/LEB	> CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as a non-detect (U)
		> CRQL but < 10x the Preparation Blank/LEB Result	Report at Preparation Blank/LEB Result and use professional judgment to qualify results as estimated high (J+) or unusable (R)
		≥ 10x the Preparation Blank/LEB Result	No qualification
Preparation Blank/LEB	< (-CRQL)	Non-detect	Qualify as estimated (UJ)
		Detect ≤ CRQL	Use professional judgment or (J-)
		< 10x CRQL	Qualify results that are ≥ CRQL as estimated low (J-)
		≥ 10x CRQL	No qualification

IV.   Interference Check Sample

A. Review Items

Form 4-IN, Form 12-IN, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the instrument's ability to overcome interferences typical of those found in samples.

C. Criteria

1. The Interference Check Sample (ICS) consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A, for all wavelengths used for each analyte reported by ICP-AES.

2. An ICS must be analyzed undiluted at the beginning of each sample analysis sequence. The ICS is not to be analyzed prior to the ICV, and shall be immediately followed by a CCV, followed by a CCB.

3. Results for the analysis of ICS Solution A must fall within the control limits of ± CRQL or ± 20% of the true value (whichever is greater) for the analytes and interferents included in the solution.

4. Results for the analysis of ICS Solution AB must fall within the control limits of ± CRQL or ± 20% of the true value (whichever is greater) for the analytes and interferents included in the solution.

5. If the value of an ICS result exceeds ± 2x the CRQL, or ± 20% of true value (whichever is greater) criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the new calibration then reverified, and all analytical samples since the last compliant ICS reanalyzed.

6. The ICS should be obtained from the EPA, if available, and analyzed according to the instructions supplied with the solutions. If the ICS is not available from the EPA, an independent ICS solution shall be prepared using certified standards with the interferent and analyte concentrations at the levels specified in the method.

D. Evaluation

1. Verify, using Form 12-IN and the raw data [Inductively Coupled Plasma (ICP) instrument printout], that the ICS was analyzed at the specified frequency and sequence during the analytical sequence.

2. Evaluate the ICS raw data for results with an absolute value that is ≥ MDL for those analytes that are not present in the ICS solution.

3. Recalculate, using the raw data and the following equation, one or more of the analyte %Rs, and verify that the recalculated value agrees with the laboratory-reported values on Form 4-IN.

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

Where,

Found (value)	=	Concentration (in µg/L) of each analyte interferent measured in the analysis of ICS Solution A or ICS Solution AB
True (value)	=	Concentration (in µg/L) of each analyte or interferent in ICS Solution A or ICS Solution AB

4. If the value of an ICS result exceeds the ± CRQL or ± 20% of true value (whichever is greater) criteria, and the laboratory failed to terminate the analysis and take the appropriate corrective action, note this for Regional Laboratory COR action and record the situation in the Data Review Narrative. Use professional judgment to assess the data.

E. **Action**

NOTE: For an ICS that does not meet the technical criteria, apply the action to all samples reported from the analytical sequence.

1. If the ICS was not analyzed at the specified frequency, qualify detects and non-detects as unusable (R). If the ICS was analyzed, but not in the proper sequence, use professional judgment to qualify detects and non-detects.

2. The raw data may not contain results for interferents. In this case, use professional judgment to qualify the data. If the data contains results for interferents, apply the following actions to samples with concentrations of interferents that are within 10% of the levels of the interferents in the ICS:

   a. If the ICS Solution AB %R for an analyte or interferent is < 50%, qualify detects as estimated low (J-) and non-detects as unusable (R).

   b. If the ICS %R for an analyte or interferent falls within the range of 50-79% [or the ICS found value is < (true value - CRQL), whichever is lower], qualify detects as estimated low (J-), and non-detects as estimated (UJ).

   c. If the ICS %R for an analyte or interferent falls within the range of 80-120%, detects and non-detects should not be qualified.

   d. If the ICS %R for an analyte or interferent is > 120% [or the ICS found value is > (true value + CRQL), whichever is greater], qualify detects as estimated high (J+). Non-detects should not be qualified.

   e. If the ICS %R for an analyte or interferent is above 150%, use professional judgment to determine the qualifications of the associated sample data.

3. If sample results that are ≥ MDLs are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify detects as estimated high (J+). Non-detects should not be qualified.

4. If negative sample results are observed for analytes that are not present in the ICS solution, and their absolute values are ≥ MDLs, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with levels of interferents that are comparable to or higher than the levels found in the ICS, qualify detects < 10x the absolute value of the negative result as estimated low (J-), and qualify non-detects as estimated (UJ).

NOTE: The same result units should be used when comparing analyte results in samples to those in the ICS. Unit conversion may be necessary when soil/sediment or wipe samples are evaluated.

5. In general, ICP-AES sample data can be accepted if the concentrations of Aluminum (Al), Calcium (Ca), Iron (Fe), and Magnesium (Mg) in the sample are found to be less than or equal to their respective concentrations in the ICS. If these elements are present at concentrations greater than the level in the ICS, or other elements are present in the sample at > 10 mg/L, investigate the possibility of other interference effects as given in the ICP-AES method or as indicated by the laboratory's interelement correction factors reported on Forms 10A-IN and 10B-IN for that particular instrument. The analyte concentration equivalents presented in the method should be considered only as estimated values since the exact value of any analytical system is instrument-

specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is > 2x the CRQL, and also > 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).

6. If the raw data does not contain results for the interferents, annotate this in the Data Review Narrative.

7. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be extremely complex. Use professional judgment to determine the need for the associated sample data to be qualified; obtain additional information from the laboratory, if necessary. Record all interpretive situations in the Data Review Narrative.

8. If the ICS acceptance criteria are grossly exceeded, note the specifics for Regional Laboratory COR action.

Table 6. Interference Check Actions for ICP-AES Analysis

Criteria	Action	
	Detect	**Non-detect**
ICS not analyzed	R	R
ICS not analyzed in the proper sequence	Use professional judgment	Use professional judgment
ICSAB %R < 50%	J-	R
ICS %R 50-79% [or ICS found value is < (true value – CRQL) whichever is lower]	J-	UJ
ICS %R 80-120%	No qualification	No qualification
ICS %R > 120% (or ICS found value is > (true value + CRQL) whichever is greater]	J+	No qualification
ICS %R > 150%	Use professional judgment	Use professional judgment
Sample results ≥ MDLs, but not present in ICS	J+	No qualification
Negative sample results, but not present in ICS	J- for results < 10x (|negative sample result|)	UJ

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V.   Laboratory Control Sample

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A. Review Items

Form 7-IN, preparation logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the recovery of the digested Laboratory Control Sample (LCS).

C. Criteria

1. Aqueous/water, soil/sediment and wipe LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples.

    a. One LCS shall be prepared and analyzed for every group of aqueous/water or soil/sediment samples in an SDG, or with each batch of samples digested, whichever is more frequent. The LCS shall be spiked such that the final digestate contains each analyte at 2x the CRQL for the associated matrix.

    b. One LCS shall be prepared and analyzed for each group of wipe samples in an SDG, or with each batch of wipe samples digested, whichever is more frequent. The wipe LCS shall be spiked such that the final digestate contains each analyte at 2x the CRQL for the associated matrix.

    c. All LCS %Rs must fall within the control limits of 70-130%, except for Antimony (Sb) and Silver (Ag) which must fall within the control limits of 50-150%. If the %R for the aqueous/water and soil/sediment LCS falls outside of the control limits, the analysis should be terminated, the problem corrected, and the samples prepared with that LCS redigested and reanalyzed. No corrective actions are required for wipe LCSs when the %R is outside the control limits.

D. Evaluation

1. Verify, using Form 7-IN, preparation logs, and raw data, that the appropriate number of required LCSs were prepared and analyzed for the SDG.

2. Evaluate Form 7-IN and verify that all results for each analyte fall within the established control limits.

    a. Check the raw data to verify that the %Rs on Form 7-IN were accurately transcribed. Recalculate one or more of the reported %Rs using the following equation:

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

    Where,

Found (value)	=	Concentration of each analyte (in µg/L, mg/kg, or µg) measured in the analysis of the LCS
True (value)	=	Concentration of each analyte (in µg/L, mg/kg, or µg) in the LCS

3. Verify that the LCS was prepared at the same time as the associated samples using the same procedures.

E. Action

NOTE: If the LCS criteria are not met, the laboratory performance and method accuracy are in question. Use professional judgment to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data associated with an LCS that does not meet the required criteria.

For an LCS that does not meet the technical criteria, apply the action to all samples in the same preparation batch.

1. If the required LCS was not analyzed at the specified frequency, use professional judgment to determine if the associated sample results should be qualified; obtain additional information from the laboratory, if necessary. If a laboratory fails to analyze a LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries, record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ).

2. Aqueous/Water and Soil/Sediment LCS:

    a. If LCS %R is < 40% (< 20% for Ag and Sb), qualify detects as estimated low (J-) and non-detects as unusable (R).

    b. If the LCS %R falls within the range of 40-69% (20-49% for Ag and Sb), qualify detects as estimated low (J-) and non-detects as estimated (UJ).

    c. If the LCS %R falls within the range of 70-130%, detects and non-detects should not be qualified.

    d. If the LCS %R is > 130% (150% for Ag and Sb), qualify detects as estimated high (J+). Non-detects should not be qualified.

    e. If the LCS %R is > 150% (170% for Ag and Sb), qualify detects as unusable (R). Non-detects should not be qualified.

3. Wipe LCS:

    a. If the LCS %R is < 40%, qualify detects as estimated low (J-) and non-detects as unusable (R).

    b. If the LCS %R is in the range of 40-69%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).

    c. If the LCS %R is within 70-130%, detects and non-detects should not be qualified.

    d. If the LCS %R is > 130%, qualify detects as estimated high (J+). Non-detects should not be qualified.

4. Annotate the potential effects on the data due to out-of-control LCS results in the Data Review Narrative.

Table 7. LCS Actions for ICP-AES Analysis

Criteria	Action	
	Detect	**Non-detect**
Aqueous/Water and Soil/Sediment %R < 40% (< 20% Ag, Sb)	J-	R
Aqueous/Water and Soil/Sediment %R 40-69% (20-69% Ag, Sb)	J-	UJ
Aqueous/Water and Soil/Sediment %R 70-130%	No qualification	No qualification
Aqueous/Water and Soil/Sediment %R > 130% (150% Ag, Sb)	J+	No qualification
Aqueous/Water and Soil/Sediment %R > 150% (170% Ag, Sb)	R	No qualification
Wipe %R < 40%	J-	R
Wipe %R 40-69%	J-	UJ
Wipe %R 70-130%	No qualification	No qualification
Wipe %R > 130%	J+	No qualification

VI.   Duplicate Sample Analysis

A. Review Items

Cover Page, Form 6-IN, instrument printouts, and raw data.

B. Objective

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis.

C. Criteria

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.

2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each SDG. Duplicates are not required for wipe samples. Duplicates cannot be averaged for reporting on Form 1-IN. Additional duplicate sample analyses may be required by EPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.

3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ 5x the CRQL.

4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form 6-IN. If both samples are non-detects, the RPD is not calculated for Form 6-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

D. Evaluation

1. Verify, using the Cover Page and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.

2. Verify, using Form 6-IN and the raw data, that all duplicate results for each analyte fall within the established control limits.

3. Verify that a field blank or PE sample was not used for duplicate analysis.

4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form 6-IN:

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

Where,

RPD   = Relative Percent Difference
S   = Sample Result (original)
D   = Duplicate Result

E. Action

> **NOTE**:   For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. Two determinations are: 1) only some samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified; or 2) no samples are sufficiently similar to the sample used for the duplicate, and thus only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.

2. If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is > 20%, qualify detects as estimated (J) and non-detects as estimated (UJ).

3. If RPD > 100%, use professional judgment to determine if the associated sample data should be qualified.

4. If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is ≤ 20%, detects and non-detects should not be qualified.

5. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate > CRQL, qualify detects as estimated (J) and non-detects as estimated (UJ).

6. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate ≤ CRQL, detects and non-detects should not be qualified.

7. If a field blank or PE sample was used for the duplicate sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Use professional judgment when evaluating the data.

8. Annotate the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 8. Duplicate Sample Actions for ICP-AES Analysis

Criteria	Action	
	Detect	**Non-detect**
Both original sample and duplicate sample results are ≥ 5x the CRQL and RPD > 20%*	J	UJ
RPD > 100%	Use professional judgment	Use professional judgment
Both original sample and duplicate sample results are ≥ 5x the CRQL and RPD is ≤ 20%	No qualification	No qualification
Original sample or duplicate sample result < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	J	UJ
Original sample or duplicate sample result < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate ≤ CRQL	No qualification	No qualification

*   The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

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VII.   Spike Sample Analysis

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A. Review Items

Cover Page, Form 5A-IN, Form 5B-IN, instrument printouts, and raw data.

B. Objective

The objective of the spiked sample analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Criteria

1. Samples identified as field blanks or PE samples cannot be used for spiked sample analysis.

2. At least one spiked sample (pre-digestion) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each SDG. Matrix Spikes are not required for wipe samples.

3. When the Matrix Spike recovery falls outside of the control limits and the sample result is < 4x the spike added, a post-digestion spike shall be performed for those analytes that do not meet the specified criteria. An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the CRQL, whichever is greater.

    NOTE: Post-digestion spikes are not required for Ag.

4. The spike %R shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is $\geq$ 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.

5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample." The average of the duplicate results cannot be used for the purpose of determining %R.

    NOTE: The final spike concentrations required for the various target analytes are presented in the methods described in the SOW.

D. Evaluation

1. Verify, using the Cover Page, Form 5A-IN, and raw data, that the appropriate number of required spiked samples was prepared and analyzed for the SDG.

2. Verify that a field blank or PE sample was not used for the spiked sample analysis.

3. Verify, using Form 5A-IN, and the raw data, that all pre-digestion spiked sample results for each required analyte fall within the established control limits. If not, verify that a post-digestion spike was prepared and analyzed.

4. Recalculate, using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms 5A-IN & 5B-IN:

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

Where,

SSR   =   Spiked Sample Result
SR   =   Sample Result
SA   =   Spike Added

NOTE: When the sample result is < MDL or reported as a non-detect, use SR = 0 only for the purpose of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms 5A-IN & 5B-IN.

E.   Action

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions). Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. Two determinations are: 1) only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified; or 2) no samples are sufficiently similar to the sample used for the Matrix Spike, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.

2. If a field blank or PE sample was used for the spiked sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Use professional judgment when evaluating the data. Detects should be qualified as estimated (J) and non-detects qualified as estimated (UJ).

3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-digestion spike was not performed, note this for Regional Laboratory COR action.

4. If the Matrix Spike %R is < 30%, verify that a post-digestion spike was analyzed. If the post-digestion spike %R is < 75% or the analysis was not performed, qualify detects as estimated low (J-), and qualify non-detects as unusable (R). If the post-digestion spike %R is ≥ 75%, qualify detects as estimated (J) and qualify non-detects as estimated (UJ).

5. If the Matrix Spike %R falls within the range of 30-74%, verify that a post-digestion spike was analyzed (if required when sample concentration is < 4x spike added). If the post-digestion spike %R < 75% or the analysis was not performed, qualify detects as estimated low (J-) and non-detects as estimated (UJ). If the post-digestion spike %R ≥ 75%, qualify detects as estimated (J) and non-detects as estimated (UJ).

6. If the Matrix Spike %R falls within the range of 75-125%, no post-digestion spike is required. Detects and non-detects should not be qualified.

7. If the Matrix Spike %R is > 125%, verify that a post-digestion spike was analyzed (if required when sample concentration is < 4x spike added). If the post-digestion spike %R > 125% or the analysis was not performed, qualify detects as estimated high (J+); non-detects should not be qualified. If the post-digestion spike %R ≤ 125%, qualify detects as estimated (J); non-detects should not be qualified.

8. Annotate the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 9. Spike Sample Actions for ICP-AES Analysis

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike %R < 30% Post-digestion spike %R < 75%	J-	R
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R 30-74% Post-digestion Spike %R < 75%	J-	UJ
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R > 125% Post-digestion spike %R > 125%	J+	No qualification
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	J	No qualification
Matrix Spike %R < 30% No post-digestion spike performed (e.g., not required for Ag)	J-	R
Matrix Spike %R 30-74% No post-digestion spike performed (e.g., not required for Ag)	J-	UJ
Matrix Spike %R 75-125% Post-digestion spike not required	No qualification	No qualification
Matrix Spike %R > 125% No post-digestion spike performed (e.g., not required for Ag)	J+	No qualification

NOTE: The above control limits are **method requirements** for spike samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 10 %R and 150 %R for the lower and upper limits) to be assessed against spike and post-digestion spike soil samples.

VIII.   Serial Dilution

A. Review Items

Form 1-IN, Form 8-IN, instrument printouts, and raw data.

B. Objective

The objective of the serial dilution analysis is to determine whether or not significant physical or chemical interferences exist due to sample matrix.

C. Criteria

1. An ICP Serial Dilution analysis shall be performed on a sample from each group of samples with a similar matrix type (e.g., water, soil or wipe) or for each SDG, whichever is more frequent.

2. Samples identified as field blanks or PE samples cannot be used for the ICP Serial Dilution analysis.

3. If the analyte concentration is sufficiently high (concentration in the original sample is > 50x the MDL), the %D between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution (concentration in the serial dilution sample is ≥ CRQL) shall be ≤ 10%.

NOTE: The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., %D > 15%) to be assessed against serial dilution soil samples.

D. Evaluation

1. Verify that a field blank or PE sample was not used for the serial dilution analysis.

2. Check the raw data and recalculate the %D using the following equation. Verify that the serial dilution analysis results and the calculated %D results agree with the values reported by the laboratory on Form 8-IN:

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

   Where,

   I   =   Initial Sample Result
   S   =   Serial Dilution Result

3. Check the raw data for any evidence of positive or negative interference (results from the diluted sample which are significantly different than the original sample), possibly due to high levels of dissolved solids in the sample, ionization effects, etc.

E. Action

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions). Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. Two determinations are: 1) only some of the samples in the SDG are similar to the serial dilution sample, and that only these samples should be qualified; or, 2) no samples are sufficiently similar to the sample used for serial dilution, and thus only the field sample used to prepare the serial dilution sample should be qualified.

1. If the appropriate number of serial dilution samples was not analyzed for each matrix, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. If a field blank or PE sample was used for the serial dilution analysis, record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ).

2. If the analyte concentration in the original sample is > 50x the MDL, its concentration in the serial dilution sample is ≥ CRQL, and the %D > 10%, qualify detects as estimated (J) and non-detects as estimated (UJ).

3. If the analyte concentration in the original sample is > 50x the MDL, its concentration in the serial dilution sample is ≥ CRQL, and the %D is ≤ 10%, detects and non-detects should not be qualified.

4. If the analyte concentration in the original sample is > 50x the MDL, its concentration in the serial dilution sample is ≥ CRQL, and %D is ≥ 100%, use professional judgment to determine if the associated sample data should be qualified.

5. If the analyte concentration in the original sample is > 5x the CRQL and its concentration in the serial dilution sample is < CRQL, detects and non-detects should not be qualified.

6. If evidence of positive or negative interference is found, use professional judgment to qualify the associated sample data. Annotate the potential effects on the reported data in the Data Review Narrative.

Table 10. Serial Dilution Actions for ICP-AES Analysis

Criteria	Action	
	Detect	**Non-detect**
Sample concentration > 50x MDL, serial dilution sample concentration ≥ CRQL, and %D > 10%*	J	UJ
Sample concentration > 50x MDL, serial dilution sample concentration ≥ CRQL, and %D ≤ 10%	No qualification	No qualification
Sample concentration > 50x MDL, serial dilution sample concentration ≥ CRQL, and %D is ≥ 100%	Use professional judgment	Use professional judgment
Sample concentration > 5x CRQL and serial dilution sample concentration < CRQL	No qualification	No qualification
Interferences present	Use professional judgment	Use professional judgment

*   The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., %D > 15%) to be assessed against serial dilution soil samples.

IX.   Regional Quality Assurance and Quality Control

A. Review Items

Form 1-IN, instrument printouts, and raw data.

B. Objective

The objective is to use results from the analysis of Regional QA/QC samples such as field blanks, PE samples, blind spikes, and blind blanks to determine the validity of the analytical results.

C. Criteria

Criteria are determined by each Region.

D. Evaluation

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples to the acceptance criteria for the specific PE samples if possible.

Calculate the RPD between field duplicates and provide this information in the Data Review Narrative.

E. Action

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Regional Laboratory COR action.

X.   Overall Assessment of Data

A. Review Items

Entire sample data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

B. Objective

The objective is to provide the overall assessment on data quality and usability.

C. Criteria

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method. All sample results must be within the linear calibration ranges per methods. Percent Solids (%Solids) must be properly used for all applicable matrix result calculations.

D. Evaluation

Examine the raw data to verify that correct calculations of the sample results were reported by the laboratory. Digestion logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form 1-IN through Form 16-IN).

1. Evaluate any technical problems not previously addressed.

2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).

3. Verify that appropriate methods and amounts were used in preparing the samples for analysis. If reduced volumes were used, verify that the laboratory had received Regional Laboratory COR approval for the use of the reduced volume.

4. Verify that there are no transcription or reduction errors (e.g., dilutions, %Solids, sample weights, etc.) on one or more samples. Recalculate %Solids for at least 10% of the samples and verify that the calculated %Solids agree with that reported by the laboratory.

5. Verify that MDLs are properly reported and that they are not greater than the respective CRQLs.

6. Verify that results fall within the calibrated range(s) of the ICP instrument(s) (Form 15-IN).

7. If appropriate information is available, assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the SOPs, and communication with the user concerning the intended use and desired quality of these data.

E. Action

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

2. Use professional judgment to qualify detects and non-detects if the MDL exceeds CRQL.

3. If a sample is not diluted properly when sample results exceed the upper limit of the calibration range, qualify detects as estimated (J).

4. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Annotate any discrepancies between the data and the SDG Narrative for Regional Laboratory COR action. If sufficient information on the intended use and required quality of the data is available, include an assessment of the data usability within the given context.

5. If any discrepancies are found, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory to obtain additional information for resolution. If a discrepancy remains unresolved, use professional judgment to determine if qualification of the data is warranted.

XI.   Calculations

Aqueous/Water Samples:

The concentrations determined in the digestate are to be reported in units of µg/L:

$$\text{Concentration (µg/L)} = C \times \frac{V_f}{V} \times DF$$

Where,

C   =   Instrument value in µg/L (the average of all replicate exposures)
V_f =   Final digestion volume (mL)
V   =   Initial aliquot amount (mL)
DF   =   Dilution Factor

Soil/Sediment Samples:

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of mg/kg:

$$\text{Concentration (mg/kg dry weight)} = C \times \frac{V_f}{W \times S} \times DF/1000$$

Where,

C   =   Instrument value in µg/L (the average of all replicate exposures)
V_f =   Final digestion volume (mL)
W   =   Initial aliquot amount (g)
S   =   %Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)
DF   =   Dilution Factor

Wipe Mass:

$$\text{Mass (µg)} = C \times V_f \times DF/1000$$

Where,

C   =   Instrument value in µg/L (The average of all replicate exposures)
V_f =   Final digestion volume (mL)
DF   =   Dilution Factor

Adjusted MDL/Adjusted CRQL Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, substitute the value of the MDL (µg/L) or CRQL (µg/L) into the "C" term in the equation above.

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

$$\text{Adjusted MDL or CRQL (mg/kg)} = C \times \frac{W_M}{W \times S} \times \frac{V_f}{V_M} \times DF$$

Where,

C   =   MDL or CRQL (mg/kg)
W_M =   Minimum method required aliquot amount (g) (1.00 g or 0.50 g)
W   =   Initial aliquot amount (g)
V_M =   Method required final sample digestion volume (mL) (100 mL or 50 mL)
V_f =   Final digestion volume (mL)
S   =   % Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)
DF   =   Dilution Factor

Hardness (Total) in Aqueous/Water Samples:

Total Hardness is defined as the sum of calcium and magnesium concentration, expressed as calcium carbonate in mg/L.

Calculate Total Hardness for Aqueous/Water samples as follows:

$$\text{Hardness (mg/L)} = [\text{Conc. Ca (mg/L)} \times 2.497] + [\text{Conc. Mg (mg/L)} \times 4.118]$$

Where,

Conc. Ca (mg/L)   = Calcium concentration (μg/L) / 1000
Conc. Mg (mg/L)   = Magnesium concentration (μg/L) / 1000

ICP-MS DATA REVIEW

The inorganic data requirements for Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) to be reviewed during validation are listed below:

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Example Analytical Sequence

The following is an example of an analytical sequence:

Tune
S##
S##
S##
S##
S##
S##
ICV
ICB
ICSA
ICSAB
CCV###
CCB ###
samples
CCV###
CCB###
samples
CCV###
CCB###, etc.

*Suffix ## and ### are as specified in Exhibit B of the Statement of Work (SOW).

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I.   **Preservation and Holding Times**

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A. Review Items

Form 1-IN, Form 12-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; shipping container temperature; holding time; and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on sample conditions and the technical holding time of the sample.

C. Criteria

1. The technical holding time is determined from the date of collection to the date of analysis.

2. The technical holding time criteria for aqueous/water samples is 180 days, preserved (with nitric acid) to pH $\leq$ 2. The addition of nitric acid to adjust the pH is only required for aqueous/water samples.

3. The technical holding time criteria for soil/sediment samples is 180 days, based on the technical holding time criteria for aqueous/water samples.

4. Soil/sediment samples shall be maintained at $\leq$ 6ºC (but not frozen) from the time of collection until receipt at the laboratory. All aqueous/water and soil/sediment samples must be stored at $\leq$ 6ºC (but not frozen) from the time of sample receipt until digestion.

D. Evaluation

Establish technical holding times by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form 12-IN and the raw data; also consider using information in the Complete SDG File (CSF), as it may be helpful in the assessment. Verify that the analysis dates on the Form 12-IN and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication of problems with the samples, the sample integrity may be compromised. Use professional judgment to evaluate the effect of the problem on the sample results.

E. Action

NOTE: Apply the action to each field sample for which the preservation or holding time criteria was not met.

1. If the pH of aqueous/water metals samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH to $\leq$ 2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

2. If soil/sediment samples are not maintained at $\leq$ 6ºC (but not frozen) from the time of collection until receipt at the laboratory, detects should be qualified as estimated low (J-) and non-detects as unusable (R).

3. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

4. Due to limited information concerning holding times for soil/sediment samples, use discretion when deciding whether to apply the aqueous/water holding time criteria to soil/sediment samples. If they are applied, annotate this in the Data Review Narrative.

5. If samples are received with shipping container temperatures > 10°C, use professional judgment to determine the reliability of the data, or qualify detects as estimated (J) and non-detects as estimated (UJ).

6. When the holding times are exceeded, annotate any possible consequences for the analytical results in the Data Review Narrative, and note it for Regional Laboratory Contracting Officer Representative (COR) action.

Table 11. Preservation and Holding Time Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with pH > 2 and pH not adjusted	Use professional judgment J-	Use professional judgment R
Soil/sediment samples not maintained at ≤ 6°C (but not frozen) from time of collection until receipt at laboratory	Use professional judgment J-	Use professional judgment R
Technical Holding Time: Aqueous/water samples > 180 days	J-	R
Technical Holding Time: Soil/sediment samples > 180 days	J-	R
Samples received > 10ºC*	Use professional judgment J	Use professional judgment UJ

*   For samples received with shipping container temperatures > 10ºC, Regional policy or project Data Quality Objectives (DQO) may allow the use of higher temperature criteria before assessing any actions for the affected samples.

II.   Tune Analysis

A. Review Items

Form 13-IN, instrument printouts, and raw data.

B. Objective

The ICP-MS tune serves as an initial demonstration of instrument stability and precision.

C. Criteria

1. Prior to calibration, the laboratory shall analyze or scan the ICP-MS tuning solution, containing 100 µg/L of Beryllium (Be), Magnesium (Mg), Cobalt (Co), Indium (In), and Lead (Pb), at least 5x consecutively. The solution shall contain all required isotopes of these elements. The laboratory shall make any adjustments necessary to bring peak width within the instrument manufacturer's specifications and adjust the resolution of the mass calibration to within 0.1 µ over the range of 6-210 µ.

2. The Percent Relative Standard Deviation (%RSD) of the absolute signals for all analytes in the tuning solution must be ≤ 5%.

D. Evaluation

1. Verify, using the raw data and Form 13-IN, that the appropriate number of analyses or scans of the ICP-MS tuning solution were performed, and that the appropriate analytes were present in the solution.

2. Verify, using the raw data and Form 13-IN, that the resolution of the mass calibration falls within the limits for each isotope of each analyte.

3. Verify, using the raw data and Form 13-IN, that the %RSD is ≤ 5% for each isotope of each analyte.

4. Verify, using the raw data, that the reported average mass and %RSD on Form 13-IN was accurately calculated. Recalculate one or more of the average masses and %RSDs for an isotope using the following equations:

$$\text{Mean} = \frac{\Sigma x}{n}$$

Where,

x   =   Mass from analysis
n   =   Number of analyses

$$\text{Percent Relative Standard Deviation} = \frac{\sigma_{n-1} \times 100}{\overline{x}}$$

Where,

$\overline{x}$ =   Mean
σ_{n-1} =   Standard Deviation

E. Action

NOTE: For ICP-MS tunes that do not meet the technical criteria, apply the action to all samples reported from the analytical sequence.

1. If the ICP-MS instrument was not tuned prior to calibration, all sample data should be qualified as unusable (R).

2. If the tuning solution was not analyzed or scanned at least 5x consecutively, or the tuning solution does not contain the required analytes spanning the analytical range, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

3. If the resolution of the mass calibration is not within 0.1 u for any isotope in the tuning solution, qualify the associated analytes that are detects as estimated (J) and non-detects as estimated (UJ). Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

4. If the %RSD > 5% for any isotope in the tuning solution, qualify detects as estimated (J) and non-detects as estimated (UJ). Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

Table 12. ICP-MS Tune Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
Tune not performed	R	R
Tune not performed properly (Section II.E.2)	Use professional judgment	Use professional judgment
Resolution of mass calibration not within 0.1 u	J	UJ
%RSD > 5%	J	UJ

III.   Calibration

A. Review Items

Form 2-IN, Form 12-IN, Form 15-IN, Form 16-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on initial calibration and calibration verification.

C. Criteria

1. Initial Calibration

The instruments shall be successfully calibrated each time the instrument is set up and after Continuing Calibration Verification (CCV) failure. The calibration date and time shall be included in the raw data.

a. A blank and at least five calibration standards shall be used to establish each calibration curve. At least one standard shall be at or below the Contract Required Quantitation Limit (CRQL), but above the Method Detection Limit (MDL). All measurements shall be within the instrument working range. A minimum of three replicate scans are required for standardization for all Quality Control (QC) and sample analyses. The average result of all the multiple scans for the standardization, QC, and sample analyses shall be used. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient of ≥ 0.995. The calculated percent differences (%Ds) for all of the non-zero standards must be within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

2. Initial and Continuing Calibration Verification

The acceptance criteria for the Initial Calibration Verification (ICV) and CCV standards are presented in Table 13:

Table 13. Acceptance Criteria for ICV and CCV Standards for ICP-MS Analysis

Analytical Method	Inorganic Analytes	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
ICP-MS	Metals	90	110

a. Initial Calibration Verification

1) Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). If the ICV Percent Recovery (%R) falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

2) Only if the ICV is not available from the United States Environmental Protection Agency (EPA), analyses shall be conducted using a certified solution of the analytes from an independent commercial standard source, at a concentration level other than that used for instrument calibration, but within the calibrated range.

3) The ICV solution shall be analyzed at each analytical mass used for analysis.

b. Continuing Calibration Verification

1) To ensure accuracy during the course of each analytical sequence, the CCV shall be analyzed and reported for each mass used for the analysis of each analyte.

2) The CCV standard shall be analyzed at a frequency of every two hours during an analytical sequence. The CCV standard shall also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

3) The analyte concentration(s) in the CCV standard(s) shall be different than the concentration used for the ICV, and at a concentration equivalent to the mid-level of their respective calibration curves.

4) The same CCV standard solution shall be used throughout the analysis for an SDG.

5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation

1. Verify that the instrument was calibrated each time the instrument was set up, utilizing a blank and at least five calibration standards, one of which was at or below the CRQL, but above the MDL.

2. Confirm that the measurements were within the working calibration range, and were the average result of at least three replicate exposures.

3. Verify that the ICV and CCV standards were analyzed for each analyte at the specified frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.

4. Recalculate one or more of the ICV and CCV %Rs using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form 2-IN.

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

Where,

Found (value)	=	Concentration (in µg/L) of each analyte measured in the analysis of the ICV or CCV solution
True (value)	=	Concentration (in µg/L) of each analyte in the ICV or CCV source

E. Action

NOTE: For initial calibrations or ICV standards that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical sequence.

1. If the instrument was not calibrated each time the instrument was set up, qualify detects and non-detects as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and at least one at or below CRQL but above MDL), use professional judgment to qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

2. If the correlation coefficient is < 0.995, the %Ds are outside the ±30% limit, or the y-intercept ≥ CRQL, qualify detects as estimated (J), and non-detects as estimated (UJ).

3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:

    a. If the ICV or CCV %R is < 75%, use professional judgment to qualify detects as estimated low (J-) or unusable (R), and non-detects as unusable (R).

    b. If the ICV or CCV %R falls within the range of 75-89%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).

    c. If the ICV or CCV %R falls within the range of 90-110%, detects and non-detects should not be qualified.

    d. If the ICV or CCV %R falls within the range of 111-125%, qualify detects as estimated high (J+). Non-detects should not be qualified.

    e. If the ICV or CCV %R is > 125%, use professional judgment to qualify detects as estimated high (J+) or unusable (R). Non-detects should not be qualified.

4. If the laboratory failed to provide adequate calibration information, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory and request the necessary information. If the information is unavailable, use professional judgment to assess the data.

5. Annotate the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.

6. If calibration criteria are grossly exceeded, note this for Regional Laboratory COR action.

NOTE: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 14. Calibration Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed	R	R
Calibration incomplete	Use professional judgment J or R	Use professional judgment UJ or R
Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL	J	UJ
ICV/CCV %R < 75%	Use professional judgment J- or R	R
ICV/CCV %R 75-89%	J	UJ
ICV/CCV %R 90-110%	No qualification	No qualification
ICV/CCV %R 111-125%	J+	No qualification
ICV/CCV %R > 125%	Use professional judgment J+ or R	No qualification

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IV.   **Blanks**

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A. Review Items

Form 1-IN, Form 3-IN, Form 12-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the blank responses by determining the existence and magnitude of contamination resulting from laboratory (or field) activities or baseline drift during analysis.

C. Criteria

1. No contaminants should be found in the blank(s).

2. The Initial Calibration Blank (ICB) shall be analyzed at each mass used for analysis after the analytical standards, but not before analysis of the ICV during the initial calibration of the instrument (see Section III.C.1).

3. A Continuing Calibration Blank (CCB) shall be analyzed at each mass used for the analysis, immediately after every CCV. The CCB shall be analyzed at a frequency of every two hours during the analytical sequence. The CCB shall be analyzed at the beginning of the analytical sequence, and again after the last CCV that was analyzed after the last analytical sample of the analytical sequence. The CCB result (absolute value) shall not exceed the CRQL of each analyte for which analysis is performed.

4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every SDG, or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.

5. If the concentration of any analyte in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with the analyte's concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed for that analyte. The laboratory is not to correct the sample concentration for the blank value.

6. If the concentration of any analyte in the Preparation Blank is < (-CRQL), all associated samples with the analyte's concentration < 10x the CRQL should be redigested and reanalyzed.

D. Evaluation

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the specified frequency and sequence during the analytical sequence, and Preparation Blanks were prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).

2. Review the results reported on Form 3-IN, as well as the raw data for all blanks, and verify that the results were accurately reported.

3. Evaluate all of the associated blanks for the presence of target analytes. Verify that if the concentration of any target analyte was > CRQL in a Preparation Blank, all associated samples with analyte concentration > CRQL but < 10x the Preparation Blank concentration were redigested and reanalyzed for the analytes. Verify that if a concentration was < (-CRQL) in a Preparation Blank, all associated samples with the analyte's concentration < 10x CRQL were redigested and reanalyzed. Verify that if the absolute value of any target analytes was > CRQL in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

E. Action

>   **NOTES**:   For ICBs that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.
>
>   For CCBs that do not meet the technical criteria, apply the action to all associated samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical sequence.
>
>   For Preparation Blanks that do not meet the technical criteria, apply the action to all associated samples prepared in the same preparation batch.

1. If the appropriate blanks were not analyzed with the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.

3. Some general "technical" review actions include:

    a. For any blank (including Preparation Blanks) reported with detects ≤ CRQLs, report detects ≤ CRQLs at the CRQLs and qualify as non-detect (U). For any blank (including Preparation Blanks) reported with detects ≤ CRQLs, use professional judgment to qualify the sample results > CRQL. Non-detects should not be qualified.

    b. For any blank (including Preparation Blanks) reported with a negative result ≤ (-MDL) but ≥ (-CRQL), carefully evaluate and determine its effect on the sample data. Use professional judgment to assess the data.

    c. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form 1-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form 3-IN. It may be easier to work with the raw data and/or convert the ICB or CCB results to the same units as the soil/sediment samples for comparison purposes.

4. Specific "method" actions include:

    a. If an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, non-detects should not be qualified. Report detects ≤ CRQLs at CRQLs and qualify as non-detect (U). Report sample results that are > CRQLs but < ICB/CCB Results at ICB/CCB Results and use professional judgment to qualify as non-detect (U) or unusable (R). Use professional judgment to qualify sample results ≥ ICB/CCB Results. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

    b. If an ICB or a CCB result is < (-CRQL), the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, use professional judgment to qualify non-detects as estimated (UJ) or unusable (R). Use professional judgment to qualify detects ≤ CRQL, or qualify as estimated low (J-). Use professional judgment to qualify sample results that are > CRQLs as estimated low (J-).

    c. If the concentration of any analyte in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10x the Preparation Blank concentration. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and > CRQL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, report the sample results at Preparation Blank Results; use professional judgment to qualify the results as

estimated high (J+) or unusable (R). Report detects ≤ CRQLs in the samples associated with the Preparation Blank at CRQLs and qualify as non-detect (U). Non-detects and sample results that are ≥ 10x the Preparation Blank Results should not be qualified. If the laboratory failed to redigest and reanalyze the samples associated with the Preparation Blank, record it in the Data Review Narrative, and note it for Regional Laboratory COR action.

d. For any Preparation Blank reported with a negative result < (-CRQL), use professional judgment to qualify detects ≤ CRQLs or qualify as estimated low (J-). Qualify sample results that are ≥ CRQLs as estimated low (J-), and qualify non-detects as estimated (UJ). Sample results that are ≥ 10x CRQLs should not be qualified.

Table 15. Blank Actions for ICP-MS Analysis

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Detect ≤ CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	Detect or non-detect	Use professional judgment
ICB/CCB	> CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL but < ICB/CCB Result	Report at ICB/CCB Result and qualify as non-detect (U) or unusable (R)
		≥ ICB/CCB Result	Use professional judgment
ICB/CCB	< (-CRQL)	Non-detect	Use professional judgment to qualify as estimated (UJ) or unusable (R)
		Detect ≤ CRQL	Use professional judgment or (J-)
		> CRQL	Use professional judgment to qualify as estimated low (J-)
Preparation Blank	Detect ≤ CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
Preparation Blank	≤ (-MDL) but ≥ (-CRQL)	Detect or non-detect	Use professional judgment

Blank Type	Blank Result	Sample Result	Action
Preparation Blank	> CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and use professional judgment to qualify results as estimated high (J+) or unusable (R)
		≥ 10x the Preparation Blank Result	No qualification
Preparation Blank	< (-CRQL)	Non-detect	Qualify as estimated (UJ)
		Detect ≤ CRQL	Use professional judgment or (J-)
		< 10x CRQL	Report results ≥ CRQL as estimated low (J-)
		≥ 10x CRQL	No qualification

V.   Interference Check Sample

A. Review Items

Form 4-IN, Form 12-IN, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the instrument's ability to overcome interferences typical of those found in samples.

C. Criteria

1. The Interference Check Sample (ICS) consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A, for all masses used for each analyte or interferent reported by ICP-MS.

2. An ICS must be analyzed undiluted at the beginning of each analysis sequence. The ICS is not to be analyzed prior to the ICV, and shall be immediately followed by a CCV, followed by a CCB.

3. Results for the analysis of the ICS Solution A must fall within the control limits of ± 2x the CRQL or ± 20% of the true value (whichever is greater) for the analytes and interferents included in the solution.

4. Results for the analysis of the ICS Solution AB must fall within the control limits of ± 2x the CRQL or ± 20% of the true value (whichever is greater) for the analytes and interferents included in the solution.

5. If the value of an ICS result exceeds ± 2x the CRQL, or ± 20% of true value (whichever is greater) criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the new calibration then reverified, and all analytical samples analyzed since the last compliant ICS reanalyzed.

6. The ICS should be obtained from the EPA, if available, and analyzed according to the instructions supplied with the solutions. If the ICS is not available from the EPA, an independent ICS solution shall be prepared using certified standards with the interferent and analyte concentrations at the levels specified in the method.

D. Evaluation

1. Verify, using Form 12-IN and the raw data, that the ICS was analyzed at the specified frequency and sequence during the analytical sequence.

2. Evaluate the ICS raw data for results with an absolute value that is ≥ MDL for those analytes that are not present in the ICS solution.

3. Recalculate, using the raw data and the following equation, one or more of the analyte %Rs, and verify that the recalculated value agrees with the laboratory-reported values on Form 4-IN.

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

Where,

Found (value)	=	Concentration (in µg/L) of each analyte interferent measured in the analysis of ICS Solution A or ICS Solution AB
True (value)	=	Concentration (in µg/L) of each analyte or interferent in ICS Solution A or ICS Solution AB

4. If the value of an ICS result exceeds ± 2x the CRQL, or ± 20% of true value (whichever is greater) criteria, and the laboratory failed to terminate the analysis and take the appropriate corrective action, note this for Regional Laboratory COR action and record the situation in the Data Review Narrative. Use professional judgment to assess the data.

E. Action

NOTE: For an ICS that does not meet the technical criteria, apply the action to all samples reported from the analytical sequence.

1. If the ICS was not analyzed at the specified frequency, qualify detects and non-detects as unusable (R). If the ICS was analyzed, but not in the proper sequence, use professional judgment to qualify detects and non-detects.

2. The raw data may not contain results for interferents. In this case, use professional judgment to qualify the data. If the data contains results for interferents, apply the following actions to samples with concentrations of interferents that are within 10% of the levels of the levels of interferent in the ICS:

    a. If the ICS Solution AB %R for an analyte or interferent is < 50%, qualify detects as estimated low (J-) and non-detects as unusable (R).

    b. If the ICS %R for an analyte or interferent falls within the range of 50-79% [or the ICS found value is < (true value - 2x the CRQL), whichever is lower], qualify detects as estimated low (J-), and non-detects as estimated (UJ).

    c. If the ICS %R for an analyte or interferent falls within the range of 80-120%, detects and non-detects should not be qualified.

    d. The ICS %R for an analyte or interferent is > 120% [or the ICS found value is > (true value + 2x the CRQL), whichever is greater], qualify detects as estimated high (J+). Non-detects should not be qualified.

    e. If the ICS %R for an analyte or interferent is above 150%, use professional judgment to determine the qualifications of the associated sample data.

3. If sample results that are ≥ MDLs are observed for analytes which are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify detects as estimated high (J+). Non-detects should not be qualified.

4. If negative sample results are observed for analytes that are not present in the ICS solution, and their absolute values are ≥ MDLs, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with levels of interferents that are comparable to or higher than the levels found in the ICS, qualify detects < 10x the absolute value of the negative result as estimated low (J-), and qualify non-detects as estimated (UJ).

NOTE: The same result units should be used when comparing analyte results in samples to those in the ICS. Unit conversion may be necessary when soil/sediment samples are evaluated.

5. If the raw data does not contain results for the interferents, annotate this in the Data Review Narrative.

6. Actions regarding the interpretation and/or the subsequent qualification of Inductively Coupled Plasma (ICP) data due to the ICS analytical results can be extremely complex. Use professional judgment to determine the need for the associated sample data to be qualified; obtain additional information from the laboratory, if necessary. Record all interpretive situations in the Data Review Narrative.

7. If the ICS acceptance criteria are grossly exceeded, note the specifics for Regional Laboratory COR action.

Table 16. Interference Check Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
ICS not analyzed	R	R
ICS not analyzed in proper sequence	Use professional judgment	Use professional judgment
ICSAB %R < 50%	J-	R
ICS %R 50-79% [or ICS found value is < (true value – 2x CRQL), whichever is lower]	J-	UJ
ICS %R 80-120%	No qualification	No qualification
ICS %R > 150%	Use professional judgment	Use professional judgment
ICS %R > 120% [or ICS true value is > (true value + 2x CRQL), whichever is greater]	J+	No qualification
Sample results ≥ MDLs, but not present in ICS	J+	No qualification
Negative sample results, but not present in ICS	J- for results < 10x(|negative sample result|)	UJ

VI.   Laboratory Control Sample

A. Review Items

Form 7-IN, preparation logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the recovery of the digested Laboratory Control Sample (LCS).

C. Criteria

1. Aqueous/water and soil/sediment LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples.

   a. One LCS shall be prepared and analyzed for every group of aqueous/water or soil/sediment samples in an SDG, or with each batch of samples digested, whichever is more frequent. The LCS shall be spiked such that the final digestate contains each analyte at 2x the CRQL for the associated matrix.

   b. All LCS %Rs must fall within the control limits of 70-130%. If the %R for the LCS falls outside of the control limits, the analysis should be terminated, the problem corrected, and the samples prepared with that LCS redigested and reanalyzed.

D. Evaluation

1. Verify, using Form 7-IN, preparation logs, and raw data, that the appropriate number of required LCSs were prepared and analyzed for the SDG.

2. Verify, using Form 7-IN, that all results for each analyte fall within the established control limits.

   a. Check the raw data to verify that the %Rs on Form 7-IN were accurately transcribed. Recalculate one or more of the reported %Rs using the following equation:

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

   Where,

Found (value)	=	Concentration of each analyte (in µg/L or mg/kg) measured in the analysis of the LCS
True (value)	=	Concentration of each analyte (in µg/L or mg/kg) in the LCS

3. Verify that the LCS was prepared at the same time as the associated samples using the same procedures.

E. Action

NOTE: If the LCS criteria are not met, the laboratory performance and method accuracy are in question. Use professional judgment to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data associated with an LCS that does not meet the required criteria.

                For an LCS that does not meet the technical criteria, apply the action to all samples in the same preparation batch.

1. If the required LCS was not analyzed at the specified frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. If a laboratory fails to analyze a LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries, record the situation in the

Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ).

2. LCS for all matrices:

    a. If LCS %R is < 40%, qualify detects as estimated low (J-) and non-detects as unusable (R).

    b. If the LCS %R falls within the range of 40-69%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).

    c. If the LCS %R falls within the range of 70-130%, detects and non-detects should not be qualified.

    d. If the LCS %R is > 130%, qualify detects as estimated high (J+). Non-detects should not be qualified.

    e. If the LCS %R is > 150%, qualify detects as unusable (R). Non-detects should not be qualified.

3. Annotate the potential effects on the data due to out-of-control LCS results in the Data Review Narrative.

Table 17. LCS Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
Aqueous/Water and Soil/Sediment %R < 40%	J-	R
Aqueous/Water and Soil/Sediment %R 40-69%	J-	UJ
Aqueous/Water and Soil/Sediment %R 70-130%	No qualification	No qualification
Aqueous/Water and Soil/Sediment %R > 130%	J+	No qualification
Aqueous/Water and Soil/Sediment %R > 150%	R	No qualification

VII.   Duplicate Sample Analysis

A. Review Items

Cover Page, Form 6-IN, instrument printouts, and raw data.

B. Objective

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis.

C. Criteria

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.

2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each SDG. Duplicates cannot be averaged for reporting on Form 1-IN. Additional duplicate sample analyses may be required by EPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.

3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values $\geq$ 5x the CRQL.

4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form 6-IN. If both samples are non-detects, the RPD is not calculated for Form 6-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

D. Evaluation

1. Verify, from the Cover Page and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.

2. Verify, using Form 6-IN and the raw data, that all duplicate results for each analyte fall within the established control limits.

3. Verify that a field blank or PE sample was not used for duplicate analysis.

4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form 6-IN:

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

Where,

RPD   =   Relative Percent Difference
S   =   Sample Result (original)
D   =   Duplicate Result

E. Action

> **NOTE**:   For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. Possible determinations are: 1) only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified; or, 2) no samples are sufficiently similar to the sample used for the duplicate, and thus only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples were not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.

2. If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is > 20%, qualify detects as estimated (J), and qualify non-detects as estimated (UJ).

3. If RPD > 100%, use professional judgment to determine if the associated sample data should be qualified.

4. If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is ≤ 20%, detects and non-detects should not be qualified.

5. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate > CRQL, qualify detects as estimated (J) and non-detects as estimated (UJ).

6. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate ≤ CRQL, detects and non-detects should not be qualified.

7. If a field blank or PE sample was used for the duplicate sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Use professional judgment when evaluating the data.

8. Annotate the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 18. Duplicate Sample Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
Both original sample and duplicate sample results are ≥ 5x the CRQL and RPD > 20%*	J	UJ
Both original sample and duplicate sample results are ≥ 5x the CRQL and RPD is ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
Original sample or duplicate sample result < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	J	UJ
Original sample or duplicate sample result < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate ≤ CRQL	No qualification	No qualification

* The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, **for technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

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VIII.   **Spike Sample Analysis**

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A. Review Items

Cover Page, Form 5A-IN, Form 5B-IN, instrument printouts, and raw data.

B. Objective

The objective of the spiked sample analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Criteria

1. Samples identified as field blanks or PE samples cannot be used for spiked sample analysis.

2. At least one spiked sample (pre-digestion) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each SDG.

3. When the Matrix Spike recovery falls outside of the control limits and the sample result is < 4x the spike added, a post-digestion spike shall be performed for those analytes that do not meet the specified criteria. An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the CRQL, whichever is greater.

4. The spike %R shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.

5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample." The average of the duplicate results cannot be used for the purpose of determining %R.

NOTE: The final spike concentrations required for the various target analytes are presented in the methods described in the SOW.

D. Evaluation

1. Verify, using the Cover Page, Form 5A-IN, and raw data, that the appropriate number of required spiked samples was prepared and analyzed for the SDG.

2. Verify that a field blank or PE sample was not used for the spiked sample analysis.

3. Verify, using Form 5A-IN and the raw data, that all pre-digestion spiked sample results for each required analyte fall within the established control limits. If not, verify that a post-digestion spike was prepared and analyzed.

4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms 5A-IN & 5B-IN:

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

    Where,

    SSR   =   Spiked Sample Result
    SR   =   Sample Result
    SA   =   Spike Added

NOTE: When the sample result is < MDL or reported as a non-detect, use SR = 0 only for the purpose of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms 5A-IN & 5B-IN.

E. Action

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions). Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. Possible determinations are: 1) only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified; or, 2) no samples are sufficiently similar to the sample used for the Matrix Spike, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.

2. If a field blank or PE sample was used for the spiked sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Use professional judgment when evaluating the data. Detects should be qualified as estimated (J) and non-detects as estimated (UJ).

3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-digestion spike was not performed, note this for Regional Laboratory COR action.

4. If the Matrix Spike %R is < 30%, verify that a post-digestion spike was analyzed. If the post-digestion spike %R is < 75% or the analysis was not performed, qualify detects as estimated low (J-) and non-detects as unusable (R). If the post-digestion spike %R is ≥ 75%, qualify detects as estimated (J) and non-detects as estimated (UJ).

5. If the Matrix Spike %R falls within the range of 30-74%, verify that a post-digestion spike was analyzed (if required when sample concentration is < 4x spike added). If the post-digestion spike %R is < 75% or the analysis was not performed, qualify detects as estimated low (J-) and non-detects as estimated (UJ). If the post-digestion spike %R for is ≥ 75%, qualify detects as estimated (J) and non-detects as estimated (UJ).

6. If the Matrix Spike %R falls within the range of 75-125%, no post-digestion spike is required. Detects and non-detects should not be qualified.

7. If the Matrix Spike %R is > 125%, verify that a post-digestion spike was analyzed (if required when sample concentration is < 4x spike added). If the post-digestion spike %R is also > 125% or the analysis was not performed, qualify detects as estimated high (J+); non-detects should not be qualified. If the post-digestion spike %R is ≤ 125%, qualify detects as estimated (J); non-detects should not be qualified.

8. Annotate the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 19. Spike Sample Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike %R < 30% Post-digestion spike %R < 75%	J-	R
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R 30-74% Post-digestion spike %R < 75%	J-	UJ
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R > 125% Post-digestion spike %R > 125%	J+	No qualification
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	J	No qualification
Matrix Spike %R < 30% No post-digestion spike performed	J-	R
Matrix Spike %R 30-74% No post-digestion spike performed	J-	UJ
Matrix Spike %R 75-125% No post-digestion spike is required	No qualification	No qualification
Matrix Spike %R > 125% No post-digestion spike performed	J+	No qualification

NOTE: The above control limits are **method requirements** for spike samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 10 %R and 150 %R for the lower and upper limits) to be assessed against spike and post-digestion spike soil samples.

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IX.   Serial Dilution

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A. **Review Items**

Form 1-IN, Form 8-IN, instrument printouts, and raw data.

B. **Objective**

The objective of the serial dilution analysis is to determine whether or not significant physical or chemical interferences exist due to sample matrix.

C. **Criteria**

1. An ICP Serial Dilution analysis shall be performed on a sample from each group of samples with a similar matrix type (e.g., water or soil) or for each SDG, whichever is more frequent.

2. Samples identified as field blanks or PE samples cannot be used for the ICP Serial Dilution analysis.

3. If the analyte concentration is sufficiently high (concentration in the original sample is > 50x the MDL), the %D between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution (concentration in the serial dilution sample is $\geq$ CRQL) shall be $\leq$ 10%.

NOTE: The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., %D > 15%) to be assessed against serial dilution soil samples.

D. **Evaluation**

1. Verify that a field blank or PE sample was not used for the serial dilution analysis.

2. Check the raw data and recalculate the %D using the following equation. Verify that the serial dilution analysis results and the calculated %D results agree with the values reported by the laboratory on Form 8-IN:

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

   Where,

   I   =   Initial Sample Result
   S   =   Serial Dilution Result

3. Check the raw data for any evidence of positive or negative interference (results from the diluted sample which are significantly different than the original sample), possibly due to high levels of dissolved solids in the sample, ionization effects, etc.

E. **Action**

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions). Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. Two determinations are: 1) only some of the samples in the SDG are similar to the serial dilution sample, and that only these samples should be qualified; or, 2) no samples are

sufficiently similar to the sample used for serial dilution, and thus only the field sample used to prepare the serial dilution sample should be qualified.

1. If the appropriate number of serial dilution samples was not analyzed for each matrix, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. If a field blank or PE sample was used for the serial dilution analysis, record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ).

2. If the analyte concentration in the original sample is > 50x the MDL, its concentration in the serial dilution sample is ≥ CRQL, and the %D > 10%, qualify detects as estimated (J) and non-detects as estimated (UJ).

3. If the analyte concentration in the original sample is > 50x the MDL, its concentration in the serial dilution sample is ≥ CRQL, and the %D is ≤ 10%, detects and non-detects should not be qualified.

4. If the analyte concentration in the original sample is > 50x the MDL, its concentration in the serial dilution sample is ≥ CRQL, and %D is ≥ 100%, use professional judgment to determine if the associated sample data should be qualified.

5. If the analyte concentration in the original sample is > 5x the CRQL and its concentration in the serial dilution sample is < CRQL, detects and non-detects should not be qualified.

6. If evidence of positive or negative interference is found, use professional judgment to qualify the associated sample data. Annotate the potential effects on the reported data in the Data Review Narrative.

Table 20. Serial Dilution Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
Sample concentration > 50x MDL, serial dilution sample concentration ≥ CRQL, and %D > 10%*	J	UJ
Sample concentration > 50x MDL, serial dilution sample concentration ≥ CRQL, and %D ≤ 10%	No qualification	No qualification
Sample concentration > 50x MDL, serial dilution sample concentration ≥ CRQL, and %D ≥ 100%	Use professional judgment	Use professional judgment
Sample concentration > 5x CRQL and serial dilution sample concentration < CRQL	No qualification	No qualification
Interferences present	Use professional judgment	Use professional judgment

* The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., %D > 15%) to be assessed against serial dilution soil samples.

X. Internal Standards

A. Review Items

Form 11-IN, Form 14-IN, instrument printouts, and raw data.

B. Objective

The objective of internal standard analysis is to determine the existence and magnitude of instrument drift and physical interferences.

C. Criteria

1. All samples analyzed during an analytical sequence, with the exception of the tune, shall contain internal standards. A minimum of five internal standards from the following list shall be added to each sample: Li (the Li^6 isotope); Sc; Y; Rh; In; Tb; Ho; Lu; and Bi. If the laboratory uses lithium as an internal standard, the laboratory shall use an Li6-enriched standard. The laboratory shall monitor the same internal standards throughout the entire analytical sequence and shall assign each analyte to at least one internal standard.

2. The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in the sample shall fall within 60-125% of the response in the calibration blank.

3. If the %RI of the response in the sample falls outside of these limits, the laboratory shall reanalyze the original sample at a two-fold dilution with internal standard added.

D. Evaluation

1. Verify, using Form 14-IN and the raw data, that a minimum of five internal standards from the specified list were used for the analysis; that the same internal standards were monitored for the entire analytical sequence; and that each analyte was associated to at least one internal standard.

2. Verify, using Form 14-IN and the raw data, that these internal standards were added to each sample in the analytical sequence, including calibrations, samples, and QC samples (except tune).

3. Verify, using Form 14-IN, that the %RI between an internal standard in a sample and the internal standard in the calibration blank was reported for each sample.

4. Verify, using Form 11-IN, Form 14-IN, and the raw data, that if the %RI for a sample was outside the limits (60-125%), the sample was reanalyzed of a two-fold dilution with internal standard added.

E. Action

NOTE: Apply the action to the affected analytes for each sample that does not meet the internal standard criteria.

1. If no internal standards were analyzed with the analytical sequence, detects and non-detects should be qualified as unusable (R). Record this issue in the Data Review Narrative, and note it for Regional Laboratory COR action.

2. If less than five of the required internal standards were analyzed with the analytical sequence, or (a) target analyte(s) is (are) not associated to an internal standard, the sample results, for the analyte(s) not associated to an internal standard should be qualified as unusable (R). Record this issue in the Data Review Narrative, and note it for Regional Laboratory COR action.

3. If the %RI for the internal standards in a sample falls within the range of 60-125%, the sample results should not be qualified.

4. If the %RI for an internal standard in a sample is < 60% or > 125%, qualify the sample results of the analytes associated with the non-compliant internal standard(s) as follows:

a. If the sample was reanalyzed at a two-fold dilution with internal standard %RIs within the limits, report the result (for those analytes associated to the internal standard outside the limits in the initial analysis) from the diluted analysis without qualification. If any of the %RIs of the diluted analysis were < 60% or > 125%, report the results of the original undiluted analysis and qualify the detects as estimated (J) and non-detects as estimated (UJ) for the associated analytes.

b. If the sample was not reanalyzed at a two-fold dilution, use professional judgment to determine the reliability of the data. Detects should be qualified as estimated (J) or unusable (R) and non-detects as estimated (UJ) or unusable (R) for the associated analytes.

Table 21. Internal Standard Actions for ICP-MS Analysis

Criteria	Action	
	Detect	**Non-detect**
No internal standards	R	R
< 5 of the required internal standards	R	R
Target analyte not associated with internal standard	R	R
%RI 60-125%	No qualification	No qualification
%RI < 60% or > 125% and original sample reanalyzed at 2-fold dilution	J	UJ
Original sample not reanalyzed at 2-fold dilution	Use professional judgment J or R	Use professional judgment UJ or R

XI.   Regional Quality Assurance and Quality Control

A. Review Items

Form 1-IN, instrument printouts, and raw data.

B. Objective

The objective is to use results from the analysis of Regional QA/QC samples such as field blanks, PE samples, blind spikes, and blind blanks to determine the validity of the analytical results.

C. Criteria

Criteria are determined by each Region.

D. Evaluation

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples to the acceptance criteria for the specific PE samples if possible.

Calculate the RPD between field duplicates and provide this information in the Data Review Narrative.

E. Action

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Regional Laboratory COR action.

XII. Overall Assessment of Data

A. Review Items

Entire sample data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

B. Objective

The objective is to provide the overall assessment on data quality and usability.

C. Criteria

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method. All sample results must be within the linear calibration ranges per methods. Percent Solids (%Solids) must be properly used for all applicable matrix result calculations.

D. Evaluation

Examine the raw data to verify that the correct calculation of the sample results was reported by the laboratory. Digestion logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form 1-IN through Form 16-IN).

1. Evaluate any technical problems not previously addressed.

2. Examine the raw data for any anomalies (e.g., baseline shifts, negative response, mass dependent drift, omissions, illegibility, etc.).

3. Verify that appropriate methods and volumes were used in preparing the samples for analysis. If reduced volumes were used, verify that the laboratory had received Regional Laboratory COR approval for the use of the reduced volume.

4. Verify that there are no transcription or reduction errors (e.g., dilutions, %Solids, sample weights, etc.) on one or more samples. Recalculate %Solids for at least 10% of the samples and verify that the calculated %Solids agree with that reported by the library.

5. Verify that MDLs are properly reported and that they are not greater than the respective CRQLs.

6. Verify that results fall within the calibrated range(s) of the instrument(s) (Form 15-IN).

7. If appropriate information is available, assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the SOPs, and communication with the user concerning the intended use and desired quality of these data.

E. Action

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

2. Use professional judgment to qualify detects and non-detects if the MDL exceeds CRQL.

3. If a sample is not diluted properly when sample results exceed the upper limit of the calibration range, qualify detects as estimated (J).

4. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Annotate any discrepancies between the data and the SDG Narrative for Regional Laboratory COR action. If sufficient information on the intended use and required quality of the data is available, include an assessment of the data usability within the given context.

5. If any discrepancies are found, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory to obtain additional information for a resolution. If a discrepancy remains unresolved, use professional judgment to determine if qualification of the data is warranted.

XIII. <u>Calculations</u>

Aqueous/Water Sample Concentration by ICP-MS:

The concentrations determined in the digestate are to be reported in units of μg/L:

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

Where,

C   =   Instrument value in μg/L (the average of all replicate integrations)
V_f =   Final digestion volume (mL)
V   =   Initial Aliquot Amount (mL)
DF   =   Dilution Factor

Soil/Sediment Sample Concentration by ICP-MS:

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of mg/kg:

$$\text{Concentration (mg/kg dry weight)} = C \times \frac{V_f}{W \times S} \times DF/1000$$

Where,

C   =   Instrument value in μg/L (the average of all replicate integrations)
V_f =   Final digestion volume (mL)
W   =   Initial aliquot amount (g)
S   =   %Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)
DF   =   Dilution Factor

Adjusted MDL/Adjusted CRQL Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, substitute the value of the MDL (μg/L) or CRQL (μg/L) into the "C" term in the equation above.

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

$$\text{Adjusted MDL or CRQL} \ (mg/kg) = C \times \frac{W_M}{W \times S} \times \frac{V_f}{V_M} \times DF$$

Where,

C   =   MDL or CRQL (mg/kg)
W_M =   Minimum method required aliquot amount (g) (1.00g or 0.50g)
W   =   Initial aliquot amount (g)
V_M =   Method required final sample digestion volume (mL) (100 mL)
V_f =   Final digestion volume (mL)
S   =   %Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)
DF   =   Dilution Factor

MERCURY DATA REVIEW

The inorganic data requirements for mercury to be reviewed during validation are listed below:

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Example Analytical Sequence

This is an example of an analytical sequence:

S##
S##
S##
S##
S##
S##
ICV
ICB
CCV###
CCB###
samples
CCV###
CCB###
samples
CCV###
CCB###, etc.

*Suffix ## and ### are as specified in Exhibit B of the Statement of Work (SOW).

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I.   Preservation and Holding Times

A.   Review Items

Form 1-IN, Form 12-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; shipping container temperature; holding time; and other sample conditions.

B.   Objective

The objective is to determine the validity of the analytical results based on the sample conditions and the holding time of the sample.

C.   Criteria

1.   The technical holding time is determined from the date of collection, or the date Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction is complete, to the date of analysis.

2.   The technical holding time criteria for aqueous/water samples and leachate samples from TCLP or SPLP is 28 days, preserved (with nitric acid) to pH ≤ 2.

3.   The technical holding time criteria for soil/sediment samples is 28 days, based on the technical holding time criteria for aqueous/water samples.

4.   Soil/sediment samples shall be maintained at ≤ 6ºC (but not frozen) from the time of collection until receipt at the laboratory. All aqueous/water and soil/sediment samples must be stored at ≤ 6ºC (but not frozen) from the time of sample receipt until digestion. The TCLP and SPLP leachates must be stored at ≤ 6ºC (but not frozen) from the time of the leaching procedure completion until digestion.

5.   Samples and standards shall be analyzed with 48 hours of preparation.

D.   Evaluation

Establish technical holding times by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form 12-IN and the raw data; also consider using information in the Complete SDG File (CSF), as it may be helpful in the assessment. Verify that the analysis dates on the Form 12-IN and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication of problems with the samples, the sample integrity may be compromised. Use professional judgment to evaluate the effect of the problem on the sample results.

E.   Action

NOTE: Apply the action to each field sample for which the preservation or holding time criteria was not met.

1.   If the pH of aqueous/water samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH to ≤ 2 at the time of sample receipt. Also determine if the laboratory adjusted the pH to ≤ 2 for the TCLP and SPLP leachates after completion of the leaching procedure. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of Mercury (possible Methylation). Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

2.   If soil/sediment samples are not maintained at ≤ 6°C (but not frozen) from the time of collection until receipt at the laboratory, detects should be qualified as estimated low (J-) and non-detects as unusable (R).

3.   If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

4. Due to limited information concerning holding times for soil/sediment samples, use professional judgment when deciding whether to apply the aqueous/water holding time criteria to soil/sediment samples. If they are applied, document this action in the Data Review Narrative.

5. If samples are received with shipping container temperatures > 10ºC, use professional judgment to determine the reliability of the data, or qualify detects as estimated (J) and non-detects as estimated (UJ).

6. When shipping or storage temperatures grossly exceed the requirements, the loss of volatile mercury compounds or metallic mercury is possible. The expected bias would be low. Use professional judgment to qualify the samples and note it for Regional Laboratory Contracting Officer Representative (COR) action.

7. When the holding times are exceeded, annotate any possible consequences for the analytical results in the Data Review Narrative, and note it for Regional Laboratory COR action.

Table 22. Preservation and Holding Time Actions for Mercury Analysis

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with pH > 2 and pH not adjusted	Use professional judgment J-	Use professional judgment R
TCLP/SPLP leachate samples with pH > 2 and pH not adjusted	Use professional judgment J-	Use professional judgment R
Soil/sediment samples not maintained at ≤ 6°C (but not frozen) from time of collection until receipt at the laboratory	J-	R
Technical Holding Time: Aqueous/water and TCLP/SPLP leachate samples > 28 days	J-	R
Technical Holding Time: Soil/sediment samples > 28 days	J-	R
Samples received > 10ºC*	Use professional judgment J	Use professional judgment UJ

*   For samples received with shipping container temperatures > 10ºC, Regional policy or project Data Quality Objectives (DQO) may allow the use of higher temperature criteria before assessing any actions for the affected samples.

II.   Calibration

A. Review Items

Form 2-IN, Form 12-IN, Form 15-IN, Form 16-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on initial calibration and calibration verification.

C. Criteria

1. Initial Calibration

    The instruments shall be successfully calibrated daily (or once every 24 hours), and each time the instrument is set up. The calibration date and time shall be included in the raw data. The calibration curve shall be prepared by the same method used to prepare the samples for analysis. The curve shall be prepared with the samples that will be analyzed using this calibration curve.

    a. A blank and at least five calibration standards shall be used to establish the calibration curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL) but above the Method Detection Limit (MDL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curve must have a correlation coefficient ≥ 0.995. The calculated percent differences (%Ds) for all of the non-zero standards must fall within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

2. Initial and Continuing Calibration Verification

    The acceptance criteria for the Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) standards are presented in Table 23. These standards shall be prepared by the same method used to prepare the samples for analysis.

Table 23. Acceptance Criteria for ICV and CCV Standards for Mercury Analysis

Analytical Method	Inorganic Analyte	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
Cold Vapor AA	Mercury	85	115

a. Initial Calibration Verification

1) Immediately after the system has been calibrated, the accuracy of the initial calibration must be verified and documented by the analysis of an ICV solution(s). If the ICV Percent Recovery (%R) falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

2) Only if the ICV is not available from the United States Environmental Protection Agency (EPA), analyses shall be conducted using a certified solution of the analyte from an independent commercial standard source, at a concentration level other than that used for instrument calibration, but within the calibrated range.

b. Continuing Calibration Verification

1) To ensure accuracy during the course of each analytical sequence, the CCV shall be analyzed and reported.

2) The CCV standard shall be analyzed at a frequency of every hour during an analytical sequence. The CCV standard shall also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

3) The analyte concentration in the CCV standard shall be different than the concentration used for the ICV, and a concentration equivalent to the mid level of the calibration curve.

4) The same CCV standard solution shall be used throughout the analysis for an SDG.

5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation

1. Verify that the instrument was calibrated daily (once every 24 hours) and each time the instrument was set up, utilizing a blank and at least five calibration standards. Confirm that at least one of the calibration standards was analyzed at or below the CRQL, but above the MDL. Confirm that calibration standards and samples were prepared at the same time.

2. Verify that the ICV and CCV standards were analyzed at the specified frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.

3. Recalculate one or more of the ICV or CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form 2-IN.

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

Where,

Found (value)	=	Concentration (in µg/L) of mercury measured in the analysis of the ICV or CCV solution
True (value)	=	Concentration (in µg/L) of mercury in the ICV or CCV source

E. Action

NOTES: For initial calibrations or ICV standards that do not meet the technical criteria, apply the action to the associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the Quality Control (QC) sample and a subsequent technically acceptable analysis of the QC sample in the analytical sequence.

1. If the instrument was not calibrated daily and each time the instrument was set up, qualify detects and non-detects as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank, and at least one standard at or below the CRQL but above the MDL), or if the instrument was not calibrated with standards prepared at the same time as the samples, use professional judgment to qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

2. If the correlation coefficient is < 0.995, the %D is outside the ±30% limit, or the y-intercept is ≥ CRQL, qualify detects as estimated (J) and non-detects as estimated (UJ).

3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:

   a. If the ICV or CCV %R is < 70%, use professional judgment to qualify detects as estimated low (J-) or unusable (R), and non-detects as unusable (R).

   b. If the ICV or CCV %R falls within the range of 70-84%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).

   c. If the ICV or CCV %R falls within the range of 85-115%, detects and non-detects should not be qualified.

   d. If the ICV or CCV %R falls within the range of 116-130%, qualify detects as estimated high (J+). Non-detects should not be qualified.

   e. If the ICV or CCV %R is > 130%, use professional judgment to qualify detects as estimated high (J+) or unusable (R). Non-detects should not be qualified.

   f. If the ICV or CCV %R is > 165%, qualify detects as unusable (R). Non-detects should not be qualified.

4. If the laboratory failed to provide adequate calibration information, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory and request the necessary information. If the information is unavailable, use professional judgment to assess the data.

5. Annotate the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.

6. If calibration criteria are grossly exceeded, note this for Regional Laboratory COR action.

NOTE: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 24. Calibration Actions for Mercury Analysis

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed	R	R
Calibration incomplete	Use professional judgment J or R	Use professional judgment UJ or R
Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL	J	UJ
ICV/CCV %R < 70%	Use professional judgment J- or R	R
ICV/CCV %R 70-84%	J-	UJ
ICV/CCV %R 85-115%	No qualification	No qualification
ICV/CCV %R 116-130%	J+	No qualification
ICV/CCV %R > 130%	Use professional judgment J+ or R	No qualification
ICV/CCV %R > 165%	R	No qualification

III.   Blanks

A. Review Items

Form 1-IN, Form 3-IN, Form 12-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the blank responses by determining the existence and magnitude of contamination resulting from laboratory (or field) activities or baseline drift during analysis.

C. Criteria

1. No contaminants should be found in the blank(s).

2. The Initial Calibration Blank (ICB) shall be analyzed at each mass used for analysis after the analytical standards, but not before analysis of the ICV during the initial calibration of the instrument (see Section II.C.1). The ICB shall be prepared by the same method used to prepare the samples for analysis.

3. A Continuing Calibration Blank (CCB) shall be analyzed immediately after every CCV. The CCB shall be prepared by the same method used to prepare the samples for analysis. The CCB shall be analyzed at a frequency of every hour during the analytical sequence. The CCB shall be analyzed at the beginning of the analytical sequence, and again after the last CCV that was analyzed after the last analytical sample of the analytical sequence. The CCB result (absolute value) shall not exceed the CRQL.

4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every SDG, or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.

5. If the analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of the analyte in the associated samples must be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with the analyte's concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed. The laboratory is not to correct the sample concentration for the blank value.

6. If the analyte concentration in the Preparation Blank is < (-CRQL), all associated samples with the analyte's concentration < 10x the CRQL, should be redigested and reanalyzed.

7. At least one Leachate Extraction Blank (LEB) shall be prepared and analyzed for each batch of samples extracted by TCLP or SPLP. The LEB consists of reagent water processed through the extraction procedure. Post-extraction, the LEB shall be processed through the appropriate sample preparation and analysis procedure.

D. Evaluation

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the specified frequency and sequence during the analytical sequence, and Preparation Blanks are prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).

2. Review the results reported on Form 3-IN, as well as the raw data for all blanks, and verify that the results are accurately reported.

3. Evaluate all of the associated blanks for the presence of the target analyte. Verify that if the concentration of the target analyte was > CRQL in a Preparation Blank, all associated samples with analyte's concentration > CRQL but < 10x the Preparation Blank concentration were redigested and reanalyzed for that analyte. Verify that if the concentration was < (-CRQL) in a

Preparation Blank, all associated samples with the analyte's concentration < 10x CRQL were redigested and reanalyzed. Verify that if the absolute value of the target analyte was > CRQL in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

E. Action

> **NOTES**: For ICBs that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.
>
> For CCBs that do not meet the technical criteria, apply the action to all associated samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical sequence.
>
> For Preparation Blanks that do not meet the technical criteria, apply the action to all associated samples prepared in the same preparation batch. For LEBs that do not meet the technical criteria, apply the action to all associated samples extracted in the same extraction batch.

1. If the appropriate blanks were not analyzed with the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.

3. Some general "technical" review actions include:

    a. For any blank (including Preparation Blanks and LEBs) reported with detects ≤ CRQL, report detects ≤ CRQL at the CRQL and qualify as non-detect (U). For any blank (including Preparation Blanks and LEBs) reported with a detect ≤ CRQL, use professional judgment to qualify the sample results > CRQL. Non-detects should not be qualified.

    b. For any blank (including Preparation Blanks and LEBs) reported with a negative result ≤ (-MDL) but ≥ (- CRQL), carefully evaluate it to determine its effect on the sample data. Use professional judgment to assess the data.

    c. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form 1-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form 3-IN. It may be easier to work with the raw data and/or convert the ICB or CCB results to the same units as the soil/sediment samples for comparison purposes.

4. Specific "method" actions include:

    a. If an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, non-detects should not be qualified. Report detects ≤ CRQL at CRQL and qualify as non-detect (U). Report sample results that are > CRQL but < ICB/CCB Results at ICB/CCB Results and use professional judgment to qualify as non-detect (U) or unusable (R). Use professional judgment to qualify sample results ≥ ICB/CCB Results. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

b. If an ICB or a CCB result is < (-CRQL), the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, use professional judgment to qualify non-detects as estimated (UJ) or unusable (R). Use professional judgment to qualify detects ≤ CRQL or qualify as estimated low (J-). Use professional judgment to qualify sample results that are > CRQLs as estimated low (J-).

c. If the concentration of the analyte in the Preparation Blank/LEB is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10x the Preparation Blank/LEB concentration. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and > CRQL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, report the sample results at Preparation Blank Results; use professional judgment to qualify as estimated high (J+) or unusable (R).   Report results <10x the LEB concentration and > CRQL in the samples associated with the LEB at LEB Results; use professional judgment to qualify the results as estimated high (J+) or unusable (R).   Report detects ≤ CRQLs in the samples associated with the Preparation Blank/LEB at CRQLs and qualify as non-detect (U). Non-detects and sample results that are ≥ 10x Preparation Blank/LEB Results should not be qualified. If the laboratory failed to redigest and reanalyze the samples associated with the Preparation Blank, record it in the Data Review Narrative, and note it for Regional Laboratory COR action.

d. For any Preparation Blank or LEB reported with a negative result, < (-CRQL), use professional judgment to qualify detects ≤ CRQL or qualify as estimated low (J-). Qualify sample results that are ≥ CRQLs as estimated low (J-), and qualify non-detects as estimated (UJ). Sample results that are ≥ 10x CRQLs should not be qualified.

Table 25. Blank Actions for Mercury Analysis

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Detect ≤ CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	Detect or non-detect	Use professional judgment
ICB/CCB	> CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL but < ICB/CCB Result	Report at ICB/CCB Result as non-detect (U) or unusable (R)
		≥ ICB/CCB Result	Use professional judgment
ICB/CCB	< (-CRQL)	Non-detect	Use professional judgment to qualify as estimated (UJ) or unusable (R)
		Detect ≤ CRQL	Use professional judgment or (J-)
		> CRQL	Use professional judgment to qualify as estimated low (J-)
Preparation Blank/LEB	Detect ≤ CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
Preparation Blank/LEB	≤ (-MDL) but ≥ (-CRQL)	Detect or non-detect	Use professional judgment
Preparation Blank/LEB	> CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL but < 10x the Preparation Blank/LEB Result	Report at Preparation Blank/LEB Result and use professional judgment to qualify results as estimated high (J+) or unusable (R)
		≥ 10x the Preparation Blank/LEB Result	No qualification
Preparation Blank/LEB	< (-CRQL)	Non-detect	Qualify as estimated (UJ)
		Detect ≤ CRQL	Use professional judgment or (J-)
		< 10x CRQL	Report results ≥ CRQL as estimated low (J-)
		≥ 10x CRQL	No qualification

<center>IV.   <u>**Duplicate Sample Analysis**</u></center>

A. Review Items

Cover Page, Form 6-IN, instrument printouts, and raw data.

B. Objective

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis.

C. Criteria

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.

2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each SDG. Duplicates cannot be averaged for reporting on Form 1-IN. Additional duplicate sample analyses may be required by EPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.

3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ 5x the CRQL.

4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form 6-IN. If both samples are non-detects, the RPD is not calculated for Form 6-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

D. Evaluation

1. Verify, from the Cover Page and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.

2. Verify, using Form 6-IN and the raw data, that the duplicate results fall within the established control limits.

3. Verify that a field blank or PE sample was not used for duplicate analysis.

4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form 6-IN:

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

Where,

RPD   =   Relative Percent Difference
S   =   Sample Result (original)
D   =   Duplicate Result

E. Action

> **NOTE**: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. Two determinations are: 1) only some samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified; or 2) no samples are sufficiently similar to the sample used for the duplicate, and thus only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Associated samples that are detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.

2. If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is > 20%, qualify detects as estimated (J), and non-detects as estimated (UJ).

3. If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is ≤ 20%, detects and non-detects should not be qualified.

4. If RPD > 100%, use professional judgment to determine if the associated sample data should be qualified.

5. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate > CRQL, qualify detects as estimated (J), and non-detects as estimated (UJ).

6. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate ≤ CRQL, detects and non-detects should not be qualified.

7. If a field blank or PE sample was used for the duplicate sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Exercise professional judgment when evaluating the data.

8. Annotate the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 26. Duplicate Sample Actions for Mercury Analysis

Criteria	Action	
	Detect	**Non-detect**
Both original sample and duplicate sample results are ≥ 5x the CRQL and RPD > 20%*	J	UJ
Both original sample and duplicate sample results are ≥ 5x the CRQL and RPD is ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
Original sample or duplicate sample results < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	J	UJ
Original sample or duplicate sample result < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate ≤ CRQL	No qualification	No qualification

*   The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, **for technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

<div align="center">

V.   Spike Sample Analysis

</div>

A. Review Items

Cover Page, Form 5A-IN, instrument printouts, and raw data.

B. Objective

The objective of the spiked sample analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Criteria

1. Samples identified as field blanks or PE samples cannot be used for spiked sample analysis.

2. At least one spiked sample shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each SDG.

3. The spike %R shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.

4. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample." The average of the duplicate results cannot be used for the purpose of determining %R.

   NOTE: The final spike concentration required is presented in the method described in the SOW.

D. Evaluation

1. Verify, using the Cover Page, Form 5A-IN and raw data, that the appropriate number of required spiked samples was prepared and analyzed for the SDG.

2. Verify that a field blank or PE sample was not used for the spiked sample analysis.

3. Verify, using Form 5A-IN and the raw data, that all Matrix Spike sample results fall within the established control limits.

4. Recalculate, using the raw data, one or more of the %Rs using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Form 5A-IN:

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

    Where,

    SSR   = Spiked Sample Result
    SR   = Sample Result
    SA   = Spike Added

    NOTE: When the sample result is < MDL or reported as a non-detect, use SR = 0 only for the purpose of calculating the %R. The actual spiked sample result, sample result, and %R (positive or negative) shall still be reported on Forms 5A-IN.

E. Action

    NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions). Additionally, use the sample data (e.g., similar

concentrations of analytes) in determining similarity between samples in the SDG. Possible determinations are: 1) only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified; or, 2) no samples are sufficiently similar to the sample used for the Matrix Spike, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.

2. If a field blank or PE sample was used for the spiked sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Use professional judgment when evaluating the data. Detects should be qualified as estimated (J) and non-detects as estimated (UJ).

3. If the Matrix Spike %R is < 30%, qualify detects as estimated low (J-) and non-detects as unusable (R).

4. If the Matrix Spike %R falls within the range of 30-74%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).

5. If the Matrix Spike %R falls with the range of 75-125%, detects and non-detects should not be qualified.

6. If the Matrix Spike %R is > 125%, qualify detects as estimated high (J+). Non-detects should not be qualified.

7. Annotate the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 27. Spike Sample Actions for Mercury Analysis

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike %R < 30%	J-	R
Matrix Spike %R 30-74%	J-	UJ
Matrix Spike %R 75-125%	No qualification	No qualification
Matrix Spike %R > 125%	J+	No qualification

NOTE: The above control limits are **method requirements** for spike samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 10 %R and 150 %R for the lower and upper limits) to be assessed against spike soil samples.

VI.   Regional Quality Assurance and Quality Control

A. Review Items

Form 1-IN, instrument printouts, and raw data.

B. Objective

The objective is to use results from the analysis of Regional Quality Assurance/Quality Control (QA/QC) samples such as field blanks, PE samples, blind spikes, and blind blanks to determine the validity of the analytical results.

C. Criteria

Criteria are determined by the Region.

D. Evaluation

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples with the acceptance criteria for the specific PE samples if possible.

Calculate the RPD between field duplicates and provide his information in the Data Review Narrative.

E. Action

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Regional Laboratory COR action.

VII.   Overall Assessment of Data

A. Review Items

Entire sample data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

B. Objective

The objective is to provide the overall assessment on data quality and usability.

C. Criteria

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method. All sample results must be within the linear calibration ranges per methods. Percent Solids (%Solids) must be properly used for all applicable matrix result calculations.

D. Evaluation

Examine the raw data to verify that the correct calculation of the sample results was reported by the laboratory. Digestion logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form 1-IN through Form 16-IN).

1. Evaluate any technical problems not previously addressed.

2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).

3. Verify that the appropriate methods and amounts were used to prepare samples and standards for analysis. If reduced volumes are used, verify that the laboratory received Regional Laboratory COR approval for the use of the reduced volume.

4. Verify that there are no transcription or reduction errors (e.g., dilutions, %Solids, sample weights, etc.) on one or more samples. Recalculate %Solids for at least 10% of the samples and verify that the calculated %Solids agree with that reported by the laboratory.

5. Verify that the MDL is properly reported and that it is not greater than the CRQL.

6. Verify that results fall within the calibrated range (Form 15-IN).

7. If appropriate information is available, assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the SOPs, and communication with the user concerning the intended use and desired quality of these data.

E. Action

1. Use professional judgment to determine if there is any need to qualify data which are not qualified based on the QC criteria previously discussed.

2. Use professional judgment to qualify detects and non-detects if the MDL exceeds CRQL.

3. If a sample is not diluted properly when sample results exceed the upper limit of the calibration range, qualify detects as estimated (J).

4. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Annotate any discrepancies between the data and the SDG Narrative for Regional Laboratory COR action. If sufficient information on the intended use and required quality of the data is available, include an assessment of the data usability within the given context.

5. If any discrepancies are found, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory to obtain additional information for resolution. If a discrepancy remains unresolved, use professional judgment to determine if qualification of the data is warranted.

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VIII.   <u>**Calculations**</u>

</div>

Aqueous/Water Samples:

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

    Where,

    C   =   Instrument value in $\mu g/L$ from the calibration curve
    DF   =   Dilution Factor of the original sample

Soil/Sediment Samples:

$$\text{Hg Concentration (mg/kg dry weight)} = C \times \frac{1}{W \times S} \times DF \times 0.1$$

    Where,

    C   =   Instrument value in $\mu g/L$ from the calibration curve
    W   =   Initial aliquot amount (g)
    S   =   %Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)
    DF   =   Dilution Factor

Adjusted MDL/Adjusted CRQL Calculation:

    To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, substitute the value of the MDL ($\mu g/L$) or CRQL ($\mu g/L$) into the "C" term in the equation above.

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

$$\text{Adjusted MDL or CRQL (mg/kg)} = C \times \frac{W_m}{W \times S} \times DF$$

    Where,

    C   =   MDL or CRQL (mg/kg)
    W_m =   Method required minimum sample weight (g) (0.50 g)
    W   =   Initial aliquot amount (g)
    S   =   %Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)
    DF   =   Dilution Factor

CYANIDE DATA REVIEW

The inorganic data requirements for cyanide to be reviewed during validation are listed below:

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Example Analytical Sequence

The following is an example of an analytical sequence:

S##
S##
S##
S##
S##
S##
ICV
ICB
CCV###
CCB###
samples
CCV###
CCB###
samples
CCV###
CCB###, etc.

*Suffix ## and ### are as specified in Exhibit B of the Statement of Work (SOW).

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I.   Preservation and Holding Times

A. Review Items

Form 1-IN, Form 12-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; shipping container temperature; holding time; and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on sample conditions and the technical holding time of the sample.

C. Criteria

1. The technical holding time is determined from the date of collection, or the date Synthetic Precipitation Leaching Procedure (SPLP) extraction is complete, to the date of analysis.

2. The technical holding time criteria for aqueous/water samples and leachate samples from SPLP is 14 days, preserved (with sodium hydroxide) to pH ≥ 10.

3. The technical holding time criteria for soil/sediment samples is 14 days, based on the technical holding time criteria for aqueous/water samples.

4. Aqueous/water and soil/sediment samples shall be maintained at ≤ 6ºC (but not frozen) from the time of collection until preparation. The SPLP leachates must be stored at ≤ 6ºC (but not frozen) from the time of the leaching procedure completion until preparation.

D. Evaluation

Establish technical holding times by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form 12-IN and the raw data; also consider using information in the Complete SDG File (CSF), as it may be helpful in the assessment. Verify that the analysis dates on the Form 12-IN and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication of problems with the samples, the sample integrity may be compromised. Use professional judgment to evaluate the effect of the problem on the sample results. For aqueous/water samples, look for evidence that the samples were tested for the presence of sulfides, oxidizing agents, or nitrate/nitrite, and whether the appropriate preservation steps were taken.

E. Action

NOTE: Apply the action to each field sample for which the preservation or holding time criteria were not met.

1. If oxidizing agents were detected in aqueous/water samples at the time of sample preparation, qualify detects as estimated low (J-) and non-detects as unusable (R). If sulfides were detected in aqueous/water samples at the time of sample preparation, qualify detects as estimated (J) and non-detects as unusable (R). If there is evidence that samples were not treated with sulfamic acid prior to distillation for nitrate/nitrite interferences, qualify detects as estimated (J) and non-detects as unusable (R). If the pH of aqueous/water samples was < 10 at the time of sample receipt, use professional judgment to qualify the samples based on the pH of the sample. Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

2. If aqueous/water and soil/sediment samples are not maintained at ≤ 6°C (but not frozen) from the time of collection until receipt at the laboratory, detects should be qualified as estimated low (J-) and non-detects as unusable (R).

3. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples are properly preserved. The expected bias would be low. Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

4. Due to limited information concerning holding times for soil/sediment samples, use professional judgment in deciding whether to apply the aqueous/water holding time criteria to soil/sediment samples. If they are applied, document this in the Data Review Narrative.

5. When the holding times are exceeded, annotate any possible consequences for the analytical results in the Data Review Narrative, and note it for Regional Laboratory Contracting Officer Representative (COR) action.

Table 28. Preservation and Holding Time Actions for Cyanide Analysis

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with oxidizing agents present	J-	R
Aqueous/water samples received with sulfides present	J	R
Aqueous/water samples received with nitrate present and not treated with sulfamic acid	J	R
Aqueous/water samples received with pH < 10	Use professional judgment J-	Use professional judgment R
Aqueous/water and soil/sediment samples not maintained at ≤ 6°C (but not frozen) from time of collection until receipt at the laboratory	Use professional judgment J-	Use professional judgment R
Technical holding time: Aqueous/water and SPLP leachate samples > 14 days	J-	R
Technical holding time: Soil/sediment samples > 14 days	J-	R

II. Calibration

A. Review Items

Form 2-IN, Form 12-IN, Form 15-IN, Form 16-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on initial calibration and calibration verification.

C. Criteria

1. Initial Calibration

   The instruments shall be successfully calibrated daily (or once every 24 hours), and each time the instrument is set up. The calibration date and time shall be included in the raw data. The calibration curve standards shall be distilled by the same method used to prepare the samples for analysis.

   a. A blank and at least five calibration standards shall be employed to establish the calibration curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL), but above the Method Detection Limit (MDL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curve must have a correlation coefficient ≥ 0.995. The calculated percent differences (%Ds) for all of the non-zero standards must be within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

2. Initial and Continuing Calibration Verification

   The acceptance criteria for the Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) standards are presented in Table 29:

Table 29. Acceptance Criteria for ICV and CCV Standards for Cyanide Analysis

Analytical Method	Inorganic Analyte	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
Colorimetric	Cyanide	85	115

   a. Initial Calibration Verification

      1) Immediately after each colorimetric system has been calibrated, the accuracy of the initial calibration must be verified and documented by the analysis of an ICV solution(s). If the ICV Percent Recovery (%R) falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

      2) Only if the ICV is not available from the United States Environmental Protection Agency (EPA), analyses shall be conducted using a certified solution of the analyte from an independent commercial standard source, at a concentration level other than that used for instrument calibration, but within the calibrated range.

      3) The ICV standard solution shall be distilled by the same method used to prepare the samples for analysis.

   b. Continuing Calibration Verification

      1) To ensure accuracy during the course of each analytical sequence, the CCV shall be analyzed and reported.

2) The CCV standard shall be analyzed at a frequency of every hour during an analytical sequence. The CCV standard shall also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

3) The analyte concentration in the CCV standard shall be different from the concentration used for the ICV, and at a concentration equivalent to the mid level of the calibration curve.

4) The same CCV standard solution shall be used throughout the analysis for an SDG.

5) The CCV shall be processed and analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

6) The CCV standard solution shall be distilled by the same method used to prepare the samples for analysis.

D. Evaluation

1. Verify that the instrument was calibrated daily (once every 24 hours) and each time the instrument was set up, utilizing a blank and at least five calibration standards. Confirm that at least one of the calibration standards was analyzed at or below the CRQL, but above the MDL.

2. Verify, using the distillation log, that the calibration standards, the ICV, and the CCV standards were distilled and analyzed.

3. Verify that the ICV and CCV standards were analyzed at the specified frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.

4. Recalculate one or more of the ICV or CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form 2-IN.

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

Where,

| Found (value) | = | Concentration (in µg/L) of cyanide measured in the analysis of the ICV or CCV solution |
| True (value) | = | Concentration (in µg/L) of cyanide in the ICV or CCV source |

E. Action

NOTES: For initial calibrations or ICV standards that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the Quality Control (QC) sample and a subsequent technically acceptable analysis of the QC sample in the analytical sequence.

1. If the instrument was not calibrated daily and each time the instrument was set up, qualify detects and non-detects as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and a standard at or below the CRQL but above the MDL), use professional judgment to qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

2. If the correlation coefficient is < 0.995, the %D is outside the ±30% limit, or y-intercept ≥ CRQL, qualify detects as estimated (J) and non-detects as estimated (UJ).

3. If the ICV or the CCV standards are not distilled, qualify detects as estimated (J) and non-detects as estimated low (UJ-).

4. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:

    a. If the ICV or CCV %R is < 70%, qualify detects as estimated low (J-) or unusable (R) and non-detects as unusable (R).

    b. If the ICV or CCV %R falls within the range of 70-84%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).

    c. If the ICV or CCV %R falls within the range of 85-115%, detects and non-detects should not be qualified.

    d. If the ICV or CCV %R falls within the range of 116-130%, qualify detects as estimated high (J+). Non-detects should not be qualified.

    e. If the ICV or CCV %R is > 130%, use professional judgment to qualify detects as estimated high (J+) or unusable (R). Non-detects should not be qualified.

5. If the laboratory failed to provide adequate calibration information, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory and request the necessary information. If the information is not available, use professional judgment to assess the data.

6. Annotate the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.

7. If calibration criteria are grossly exceeded, note this for Regional Laboratory COR action.

NOTE: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 30. Calibration Actions for Cyanide Analysis

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed	R	R
Calibration incomplete	Use professional judgment J or R	Use professional judgment UJ or R
Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL	J	UJ
Standards and QC not distilled	J	UJ-
ICV/CCV %R < 70%	Use professional judgment J- or R	R
ICV/CCV %R 70-84%	J-	UJ
ICV/CCV %R 85-115%	No qualification	No qualification
ICV/CCV %R 116-130%	J+	No qualification
ICV/CCV %R > 130%	Use professional judgment J+ or R	No qualification

III.   <u>Blanks</u>

A. Review Items

Form 1-IN, Form 3-IN, Form 12-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the blank responses by determining the existence and magnitude of contamination resulting from laboratory (or field) activities or baseline drift during analysis.

C. Criteria

1. No contaminants should be found in the blank(s).

2. The Initial Calibration Blank (ICB) shall be analyzed at each mass used for analysis after the analytical standards, but not before analysis of the ICV during the initial calibration of the instrument (see Section II.C.1).

3. A Continuing Calibration Blank (CCB) shall be analyzed immediately after every CCV. The CCB shall be analyzed at a frequency of every hour during the analytical sequence. The CCB shall be analyzed at the beginning of the analytical sequence, and again after the last CCV that was analyzed after the last analytical sample of the analytical sequence. The CCB result (absolute value) shall not exceed the CRQL.

4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every SDG, or with each batch of samples distilled, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.

5. If the analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of the analyte in the associated samples must be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with the analyte's concentration < 10x the Preparation Blank concentration, and > CRQL, should be redistilled and reanalyzed. The laboratory is not to correct the sample concentration for the blank value.

6. If the analyte concentration in the Preparation Blank is < (-CRQL), all associated samples with the analyte's concentration < 10x the CRQL, should be redistilled and reanalyzed.

7. At least one Leachate Extraction Blank (LEB) shall be prepared and analyzed for each batch of samples extracted by SPLP. The LEB consists of reagent water processed through the extraction procedure. Post-extraction, the LEB shall be processed through the appropriate sample preparation and analysis procedure.

D. Evaluation

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the specified frequency and sequence during the analytical sequence, and Preparation Blanks and LEBs are prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of distillation batches, etc.).

2. Review the results reported Form 3-IN, as well as the raw data for all blanks, and verify that the results were accurately reported.

3. Evaluate all of the associated blanks for the presence of the target analyte. Verify that if the concentration of the target analyte was > CRQL in a Preparation Blank, all associated samples with the analyte's concentration > CRQL but < 10x the Preparation Blank concentration were redistilled and reanalyzed for the analyte. Verify that if a concentration was < (-CRQL) in a Preparation Blank, all associated samples with the analyte's concentration < 10x CRQL were redistilled and reanalyzed. Verify that if the absolute value of the target analyte was > CRQL in

an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

E. Action

NOTES: For ICBs that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCBs that do not meet the technical criteria, apply the action to all associated samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical sequence.

For Preparation Blanks that do not meet the technical criteria, apply the action to all associated samples prepared in the same preparation batch. For LEBs that do not meet the technical criteria, apply the action to all associated samples extracted in the same extraction batch.

1. If the appropriate blanks were not analyzed with the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.

3. Some general "technical" review actions include:

    a. For any blank (including Preparation Blanks and LEBs) reported with detects $\leq$ CRQL, report detects $\leq$ CRQL at the CRQL and qualify as a non-detect (U). For any blank (including Preparation Blanks and LEBs) reported with a detect $\leq$ CRQL, use professional judgment to qualify the sample results > CRQLs. Non-detects should not be qualified.

    b. For any blank (including Preparation Blanks and LEBs) reported with a negative result, $\leq$ (-MDL) but $\geq$ (-CRQL), carefully evaluate and determine its effect on the sample data. Use professional judgment to assess the data.

    c. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form 1-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form 3-IN. It may be easier to work with the raw data and/or convert the ICB or CCB results to the same units as the soil/sediment samples for comparison purposes.

4. Specific "method" actions include:

    a. If an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, non-detects should not be qualified. Report detects $\leq$ CRQL at the CRQL and qualify as non-detect (U). Report sample results that are > CRQL but < ICB/CCB Results at ICB/CCB Results and use professional judgment to qualify as non-detect (U) or unusable (R). Use professional judgment to qualify sample results that are $\geq$ ICB/CCB Results. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

    b. If an ICB or a CCB result is < (-CRQL), the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, use professional judgment to qualify non-detects as estimated (UJ) or unusable (R). Use professional judgment to qualify detects $\leq$ CRQL, or qualify as estimated low (J-). Use professional judgment to qualify sample results that are > CRQLs as estimated low (J-).

c. If the concentration of any analyte in the Preparation Blank/LEB is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10x the Preparation Blank/LEB concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should have been redistilled and reanalyzed. If the associated samples were not redistilled and reanalyzed, report the sample results at Preparation Blank Results; use professional judgment to qualify the results as estimated high (J+) or unusable (R). Report results < 10x the LEB concentration and > CRQL in the samples associated with the LEB at LEB Results; use professional judgment to qualify the results as estimated high (J+) or unusable (R). Report detects ≤ CRQLs in the samples associated with the Preparation Blank/LEB at CRQLs and qualify as non-detect (U). Non-detects and sample results that are ≥ 10x the Preparation Blank/LEB Results should not be qualified. If the laboratory failed to redistill and reanalyze the samples associated with the Preparation Blank, record it in the Data Review Narrative, and note it for Regional Laboratory COR action.

d. For any Preparation Blanks or LEBs reported with a negative result < (-CRQL), use professional judgment to qualify detects ≤ CRQL, or qualify as estimated low (J-). Qualify sample results that are ≥ CRQLs as estimated low (J-), and non-detects as estimated (UJ). Sample results that are ≥ 10x CRQLs should not be qualified.

Table 31. Blank Actions for Cyanide Analysis

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Detect ≤ CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	Detect or non-detect	Use professional judgment
ICB/CCB	> CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL but < ICB/CCB Result	Report at ICB/CCB Result and qualify as non-detect (U) or unusable (R)
		≥ ICB/CCB Result	Use professional judgment
ICB/CCB	< (-CRQL)	Non-detect	Use professional judgment to qualify as estimated (UJ) or unusable (R)
		Detect ≤ CRQL	Use professional judgment or (J-)
		> CRQL	Use professional judgment to qualify as estimated low (J-)
Preparation Blank/LEB	Detect ≤ CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment

Blank Type	Blank Result	Sample Result	Action
Preparation Blank/LEB	≤ (-MDL) but ≥ (-CRQL)	Detect or non-detect	Use professional judgment
Preparation Blank/LEB	> CRQL	Non-detect	No qualification
		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL but < 10x the Preparation Blank/LEB Result	Report at Preparation Blank/LEB Result and use professional judgment to qualify results as estimated high (J+) or unusable (R)
		≥ 10x the Preparation Blank/LEB Result	No qualification
Preparation Blank/LEB	< (-CRQL)	Non-detect	Qualify as estimated (UJ)
		Detect ≤ CRQL	Use professional judgment or (J-)
		< 10x CRQL	Qualify results that are ≥ CRQL as estimated low (J-)
		≥ 10x CRQL	No qualification

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IV.   **Duplicate Sample Analysis**

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A. Review Items

Cover Page, Form 6-IN, instrument printouts, and raw data.

B. Objective

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis.

C. Criteria

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.

2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each SDG. Duplicates cannot be averaged for reporting on Form 1-IN. Additional duplicate sample analyses may be required by EPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.

3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values $\geq$ 5x the CRQL.

4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form 6-IN. If both samples are non-detects, the RPD is not calculated for Form 6-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

D. Evaluation

1. Verify, from the Cover Page and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.

2. Verify, using Form 6-IN and the raw data, that the duplicate results fall within the established control limits.

3. Verify that a field blank or PE sample was not used for duplicate analysis.

4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form 6-IN:

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

    Where,

    RPD   = Relative Percent Difference
    S   = Sample result (original)
    D   = Duplicate result

E. Action

>   **NOTE**:   For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The determinations are: 1) only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified; or, 2) no samples are sufficiently similar to the sample used for the duplicate, and thus only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.

2. If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is > 20%, qualify detects as estimated (J), and qualify non-detects as estimated (UJ).

3. If RPD > 100%, use professional judgment to determine if the associated sample data should be qualified.

4. If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is ≤ 20%, detects and non-detects should not be qualified.

5. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate > CRQL, qualify detects as estimated (J) and non-detects as estimated (UJ).

6. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate ≤ CRQL, detects and non-detects should not be qualified.

7. If a field blank or PE sample was used for the duplicate sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Exercise professional judgment when evaluating the data.

8. Annotate the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 32. Duplicate Sample Actions for Cyanide Analysis

Criteria	Action	
	Detect	**Non-detect**
Both original sample and duplicate sample results are ≥ 5x the CRQL and RPD > 20%*	J	UJ
RPD > 100%	Use professional judgment	Use professional judgment
Both original sample and duplicate sample results are ≥ 5x the CRQL and RPD is ≤ 20%	No qualification	No qualification
Original sample or duplicate sample result < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	J	UJ
Original sample or duplicate sample result < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate ≤ CRQL	No qualification	No qualification

*   The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

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V.   Spike Sample Analysis

A. Review Items

Cover Page, Form 5A-IN, Form 5B-IN, instrument printouts, and raw data.

B. Objective

The objective of the spiked sample analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Criteria

1. Samples identified as field blanks or PE samples cannot be used for spiked sample analysis.

2. At least one spiked sample (pre-distillation) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each SDG.

3. When the Matrix Spike recovery falls outside of the control limits and the sample result is < 4x the spike added, a post-distillation spike shall be performed. An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the CRQL, whichever is greater.

4. The spike %R shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.

5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample." The average of the duplicate results cannot be used for the purpose of determining %R.

   NOTE: The final spike concentration required is presented in the method described in the SOW.

D. Evaluation

1. Verify, using the Cover Page, Form 5A-IN and raw data, that the appropriate number of required spiked samples was prepared and analyzed for the SDG.

2. Verify that a field blank or PE sample was not used for the spiked sample analysis.

3. Verify, using Form 5A-IN and the raw data, that all pre-distillation spiked sample results fall within the established control limits. If not, verify that a post-distillation spike was prepared and analyzed.

4. Recalculate, using the raw data, one or more of the %Rs using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms 5A-IN & 5B-IN:

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

   Where,

   SSR   =   Spiked Sample Result
   SR   =   Sample Result
   SA   =   Spike Added

   NOTE: When the sample result is < MDL or reported as a non-detect, use SR = 0 only for the purpose of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms 5A-IN & 5B-IN.

E. Action

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E_h, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions). Also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The possible determination are: 1) only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified; or 2) no samples are sufficiently similar to the sample used for the Matrix Spike, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.

2. If a field blank or PE sample was used for the spiked sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Use professional judgment when evaluating the data. Detects should be qualified as estimated (J) and non-detects as estimated (UJ).

3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-distillation spike was not performed, note this for Regional Laboratory COR action.

4. If the Matrix Spike %R is < 30%, verify that a post-distillation spike was analyzed (if required when sample concentration is < 4x spike added). If the post-distillation spike %R is < 75% or the analysis was not performed, qualify detects as estimated low (J-), and non-detects as unusable (R). If the post-distillation spike %R is ≥ 75%, qualify detects as estimated (J) and non-detects as estimated (UJ).

5. If the Matrix Spike %R falls within the range of 30-74%, verify that a post-distillation spike was analyzed (if required when sample concentration is < 4x spike added). If the post-distillation spike %R is < 75% or the analysis was not performed, qualify detects as estimated low (J-), and non-detects as estimated (UJ). If the post-distillation spike %R is ≥ 75%, qualify detects as estimated (J), and non-detects as estimated (UJ).

6. If the Matrix Spike %R falls within the range of 75-125%, no post-distillation spike is required. Detects and non-detects should not be qualified.

7. If the Matrix Spike %R is > 125%, verify that a post-distillation spike was analyzed (if required when sample concentration is < 4x spike added). If the post-distillation spike %R is > 125% or the analysis was not performed, qualify detects as estimated high (J+); non-detects should not be qualified. If the post-distillation spike %R is ≤ 125%, qualify detects as estimated (J); non-detects should not be qualified.

8. Annotate the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 33. Spike Sample Actions for Cyanide Analysis

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike %R < 30% Post-distillation spike %R < 75%	J-	R
Matrix Spike %R < 30% Post-distillation spike %R ≥ 75%	J	UJ
Matrix Spike %R 30-74% Post-distillation spike %R < 75%	J-	UJ
Matrix Spike %R 30-74% Post-distillation spike %R ≥ 75%	J	UJ
Matrix Spike %R > 125% Post-distillation spike %R > 125%	J+	No qualification
Matrix Spike %R > 125% Post-distillation spike %R ≤ 125%	J	No qualification
Matrix Spike %R < 30% No post-distillation spike performed	J-	R
Matrix Spike %R 30-74% No post-distillation spike performed	J-	UJ
Matrix Spike %R 75-125% Post-distillation not required	No qualification	No qualification
Matrix Spike %R > 125% No post-distillation spike performed	J+	No qualification

NOTE: The above control limits are **method requirements** for spike samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 10 %R and 150 %R for the lower and upper limits) to be assessed against spike soil samples.

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VI.   **Regional Quality Assurance and Quality Control**

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A. Review Items

Form 1-IN, instrument printouts, and raw data.

B. Objective

The objective is to use results from the analysis of Regional Quality Assurance/Quality Control (QA/QC) samples such as field blanks, PE samples, blind spikes, and blind blanks to determine the validity of the analytical results.

C. Criteria

Criteria are determined by each Region.

D. Evaluation

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples to the acceptance criteria for the specific PE samples if possible.

Calculate the RPD between field duplicates and provide this information in the Data Review Narrative.

E. Action

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Regional Laboratory COR action.

VII.   Overall Assessment of Data

A. Review Items

Entire data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

B. Objective

The objective is to provide the overall assessment on data quality and usability.

C. Criteria

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method. All sample results must be within the linear calibration ranges per methods. Percent Solids (%Solids) must be properly used for all applicable matrix result calculations.

D. Evaluation

Examine the raw data to verify that the correct calculation of the sample results was reported by the laboratory. Distillation logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form 1-IN through Form 16-IN).

1. Evaluate any technical problems not previously addressed.

2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).

3. Verify that the appropriate methods and amounts were used to prepare samples for analysis. If reduced volumes were used, verify that the laboratory had received Regional Laboratory COR approval for the use of the reduced volume.

4. Verify that there were no transcription or reduction errors (e.g., dilutions, %Solids, sample weights, etc.) on one or more samples. Recalculate %Solids for at least 10% of the samples and verify that the calculated %Solids agree with that reported by the laboratory.

5. Verify that the MDL is properly reported and it is not greater than the CRQL.

6. Verify that results fall within the calibrated range (Form 15-IN).

7. If appropriate information is available, assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the SOPs, and communication with user concerning the intended use and desired quality of these data.

E. Action

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

2. Use professional judgment to qualify detects and non-detects if the MDL exceeds the CRQL.

3. If a sample is not diluted properly when sample results exceed the upper limit of the calibration range, qualify detects as estimated (J).

4. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Annotate any discrepancies between the data and the SDG Narrative for Regional Laboratory COR action. If sufficient information on the intended use and required quality of the data is available, include an assessment of the data usability within the given context.

5. If any discrepancies are found, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory to obtain additional information for resolution. If a discrepancy remains unresolved, determine if qualification of the data is warranted.

VIII.   <u>Calculations</u>

Aqueous/Water Sample Concentration:

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

  Where,

C	=	Instrument response in $\mu g/L$ CN from the calibration curve
V_f	=	Final prepared (absorbing solution) volume (mL)
V	=	Initial aliquot amount (mL)
DF	=	Dilution Factor

Soil/Sediment Sample Concentration:

$$\text{CN Concentration } (\text{mg/kg dry weight}) = C \times \frac{V_f}{W \times S} \times (1/1000) \times DF$$

  Where,

C	=	Instrument response in $\mu g/L$ CN from the calibration curve
V_f	=	Final prepared (absorbing solution) volume (mL)
W	=	Initial aliquot amount (g)
S	=	%Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)
DF	=	Dilution Factor

Adjusted MDL/Adjusted CRQL Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, substitute the value of the MDL ($\mu g/L$) or CRQL ($\mu g/L$) into the "C" term in the equation above.

Calculate the adjusted MDL or adjusted CRQL for all soil/sediment as follows:

$$\text{Adjusted MDL or CRQL (mg/kg)} = C \times \frac{W_M}{W \times S} \times DF$$

  Where,

C	=	MDL or CRQL (mg/kg)
W_M	=	Minimum method required aliquot amount (1.00 g for Midi or 0.50 g for Micro)
W	=	Initial aliquot amount (g)
S	=	%Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)
DF	=	Dilution Factor

APPENDIX A: GLOSSARY

Analyte – The element or ion an analysis seeks to determine; the element of interest.

Analytical Sample – Any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), Continuing Calibration Blank (CCB), and tunes. Note that the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA); matrix spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICSs); Laboratory Control Samples (LCSs); Performance Evaluation (PE) samples; and Preparation Blanks.

Analytical Services Branch (ASB) – The division of the United States Environmental Protection Agency's (EPA) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

Associated Samples – Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples analyzed under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.

Blank – An analytical sample that has negligible or unmeasurable amounts of a substance of interest. The blank is designed to assess specific sources of contamination. Types of blanks may include calibration blanks, preparation blanks, and field blanks. See the individual definitions for types of blanks.

Calibration – A set of operations that establish under specific conditions, the relationship between values indicated by a measuring instrument and the corresponding known values. The calibration standards must be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

Calibration Blank – A blank solution containing all of the reagents, and in the same concentration, as those used in the analytical sample preparation. This blank is not subjected to the preparation method for Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS), but is digested for mercury and cyanide. Calibration blanks are used to verify that the instrument baseline is stable and the instrument is free of contamination.

Calibration Curve – A plot of instrument response versus concentration of standards.

Calibration Standards – A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

Case – A finite, usually predetermined number of samples collected over a given time period from a particular site. Case Numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

Continuing Calibration Blank (CCB) – A reagent water sample that is run 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN) and designed to detect any carryover contamination.

Continuing Calibration Verification (CCV) – A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples. The CCV should have a concentration in the middle of the calibration range and shall be analyzed at the beginning of the day prior to the analysis of samples, and every 2 hours (1 hour for Hg and CN).

Contract Compliance Screening (CCS) – A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under EPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.

Contract Laboratory Program (CLP) – Supports the EPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known and documented quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of the EPA.

Contract Required Quantitation Limit (CRQL) – Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

Contractual Holding Time – The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

Duplicate – A second aliquot of a sample that is treated the same as the original sample in order to evaluate the precision.

Field Blank – Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

Field Duplicate – A duplicate sample generated in the field, not in the laboratory.

Initial Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Initial Calibration Blank (ICB) – The first blank standard analysis to confirm the calibration curve.

Initial Calibration Verification (ICV) – Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when EPA ICV solutions are not available.

Interference Check Sample (ICS) – A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

Internal Standard – A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

Laboratory Control Sample (LCS) – A matrix spiked at a known concentration. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the EPA samples received.

Matrix – The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, and wipe.

Matrix Spike – Aliquot of a sample (aqueous/water or soil/sediment) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure to indicate the appropriateness of the method for the matrix by measuring recovery.

Method Detection Limit (MDL) – The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

Percent Difference (%D) – As used in this document and the Statement of Work (SOW), is used to compare two values. It is the difference between the two values divided by one of the values multiplied by 100.

Performance Evaluation (PE) Sample – A sample of known composition to the EPA; however, unknown to the Contractor that is provided to evaluate Contractor performance.

Post-Digestion Spike/Post-Distillation Spike – The addition of a known amount of standard after digestion or distillation (also identified as an analytical spike).

Preparation Blank – An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Regional Laboratory Contracting Officer Representative (Regional Laboratory COR) – The EPA official who monitors assigned CLP laboratories (either inside or outside of the Regional Laboratory COR's respective Region), responds to and identifies problems in laboratory operations, and participants in on-site laboratory programs.

Relative Percent Difference (RPD) – As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

Relative Standard Deviation (RSD) – As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.

Sample – A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.

Sample Delivery Group (SDG) – A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or

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

- All samples scheduled with the same level of deliverables.

- In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG.

Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory. Laboratories shall take all precautions to meet the 20 sample per SDG criteria.

Sample Management Office (SMO) – A contractor-operated facility operated under the SMO contract, awarded and administered by the EPA. SMO provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

SDG Narrative – Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Serial Dilution – The dilution of a sample by a factor of five. When corrected by the Dilution Factor (DF), the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents [Inductively Coupled Plasma (ICP) only].

Statement of Work (SOW) – A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

Technical Holding Time – The maximum amount of time that samples may be held from the collection date until analysis.

Tune – A solution containing a range of isotope masses to establish ICP-MS accuracy, resolution, and precision prior to calibration. May also be called Instrument Performance Check sample (IPC).

APPENDIX B: INORGANIC DATA REVIEW SUMMARY

CASE NO. _____   SITE _____

LABORATORY_____   NO. OF SAMPLES/MATRIX _____

MA NO. _____   SDG NO. _____

SOW NO._____   REGION _____

REVIEWER NAME _____   COMPLETION DATE _____

REGIONAL LABORATORY COR
ACTION _____   FYI _____

REVIEW CRITERIA	**METHOD/ANALYTE**			
	ICP-AES	ICP-MS	Mercury	Cyanide
1. Preservation and Holding Time	_____	_____	_____	_____
2. Tune Analysis		_____		
3. Calibration	_____	_____	_____	_____
4. Blanks	_____	_____	_____	_____
5. Interference Check Sample	_____	_____		
6. Laboratory Control Sample	_____	_____		
7. Duplicate Sample Analysis	_____	_____	_____	_____
8. Spike Sample Analysis	_____	_____	_____	_____
9. Serial Dilution	_____	_____		
10. Internal Standards		_____		
11. Regional Quality Assurance and Quality Control	_____	_____	_____	_____
12. Overall Assessment of Data	_____	_____	_____	_____

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