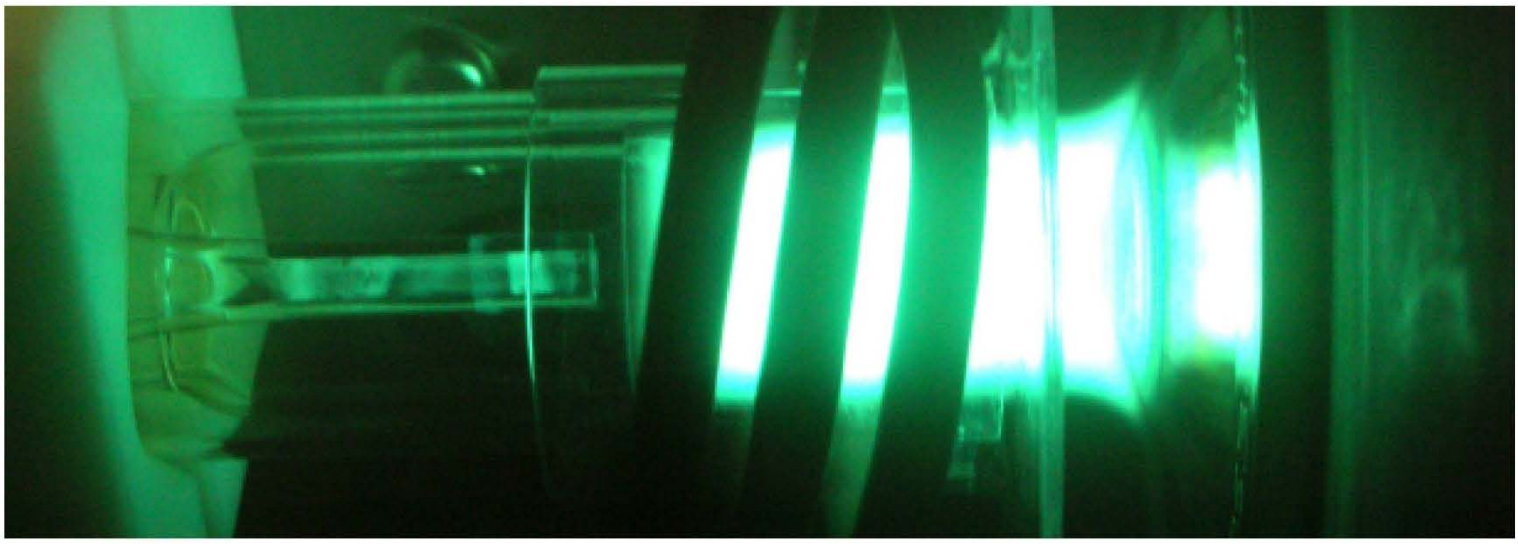
OLEM 9240.1-66
EPA 542-R-20-006
November 2020

NATIONAL FUNCTIONAL GUIDELINES

for Inorganic Superfund Methods Data Review



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NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (EPA) and other governmental employees. They do not constitute rule making by the EPA, and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at variance with the policies and procedures in this manual.

This document can be obtained from the EPA's Superfund Analytical Services and Contract Laboratory Program website at:

https://www.epa.gov/clp/contract-laboratory-program-national-functional-guidelines-data-review

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ACRONYMS AND ABBREVIATIONS

I. Terminology

The following acronyms and abbreviations are applicable to this document. For definitions, see Appendix A: Glossary at the end of the document.

%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%RSD	Percent Relative Standard Deviation
%Solids	Percent Solids
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CLPSS	Contract Laboratory Program Support System
COC	Chain of Custody
DF	Dilution Factor
DL	Detection Limit
DQA	Data Quality Assessment
DQO	Data Quality Objectives
EDM	EXES Data Manager
EPA	United States Environmental Protection Agency
EXES	Electronic Data Exchange and Evaluation System
IC	Ion Chromatography
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
OSRTI	Office of Superfund Remediation and Technology Innovation
PE	Performance Evaluation
PDF	Portable Document Format
QA	Quality Assurance

QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SEDD	Staged Electronic Data Deliverable
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SPLP	Synthetic Precipitation Leaching Procedure
TCLP	Toxicity Characteristic Leaching Procedure
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TSS	Total Suspended Solids

INTRODUCTION

I. Purpose of Document

This document provides guidance to aid in the evaluation and documentation of the quality of analytical data generated for metals by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES), metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS), mercury, cyanide, anions by Ion Chromatography (IC), hexavalent chromium by IC, and Total Organic Carbon (TOC).

The guidelines presented in this document have been designed to assist United States Environmental Protection Agency (EPA) Regional offices in evaluating (a) whether the analytical data meet the technical and Quality Control (QC) criteria established in the project-specific Quality Assurance Project Plan (QAPP) or in the EPA Superfund Contract Laboratory Program (CLP) Statement of Work (SOW), and (b) the uncertainty and extent of bias of any data that do not meet these criteria. These guidance documents have also been used by many outside the CLP community and outside EPA who evaluate analytical chemistry data, because of the attention to detail, and the decision matrices in each section.

The specific criteria and QC limits, on which the National Functional Guidelines (NFG) data qualification recommendations are based, are from the EPA CLP SOW due to the fact that these guidelines are primarily used for the review and validation of CLP data, both electronically and manually. The criteria provided in a project-specific QAPP will take precedence over those in the EPA CLP SOW. It is recognized that some criteria may have become standard for a particular analytical method. However, when utilizing the NFG for non-CLP data review, the criteria used should come from the project-specific QAPP (if available), reference method, or applicable Standard Operating Procedures (SOPs). Therefore, the source of the criteria used for the review should be clearly documented in the Data Review Narrative.

This document contains guidance for evaluating data quality in areas such as blanks, calibration and verification, instrument performance checks and performance evaluation samples, in which performance is fully under a laboratory's control, as well as more general guidance to aid in making subjective judgments regarding the quality of data for their use in making site decisions.

II. Data Reviewer Considerations

The guidance provided herein does not eliminate the need to consult other sources of information or to use professional judgment. Professional judgment is not frequently called for in this guidance document, but it is essential, in consideration of the intended use of the data. It is frequently necessary for making the best decision regarding data quality when multiple factors are involved and two qualifiers are presented. Reliable professional judgment comes from experience gained as a result of extensive training received from experts, having performed the subject analyses, and from having reviewed other analysts' and/or laboratories' data generated with similar procedures. The Action section, in each data element subchapter, provides guidance to assist the reviewer to make the most appropriate decision on how to represent data quality.

Data quality is impacted by many factors including procedures and events that may have occurred before the samples arrived at the laboratory. The reviewer would need to have knowledge of these factors, as well as a complete understanding of the project goals in order to make appropriate judgments about data usability. Ultimately, these decisions should be made by project management personnel, using the data review reports which are the product of following this guidance document, in addition to other information available to them.

Effective use of this guidance document requires the reviewer to understand the cited reference method(s) and underlying chemistry, the data quality requirements of the project, and the data provided by the laboratory. The reviewer is advised to evaluate all information provided by the laboratory to gain a complete understanding of data quality issues. Additional information may be

needed from the laboratory that was not included in the data package and may be requested as needed. Findings from the review should be thoroughly documented in the Data Review Narrative, including additional explanation as needed where professional judgment was applied.

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, the review procedures are addressed for each method in a stand-alone format. A complete list of acronyms used in this document appears preceding this Introduction, and a Glossary is included as Appendix A. An Inorganic Data Review Summary is included as Appendix B.

IV. Additional Information

For additional information about EPA methods and guidance, refer to the links below.

Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8	https://www.epa.gov/quality/guidance-environmental-data-verification-and-data-validation
EPA's Contract Laboratory Program (CLP)	https://www.epa.gov/clp
EPA CLP Statement of Work for Superfund Analytical Methods (SOW)	https://www.epa.gov/clp/epa-contract-laboratory-program-statement-work-superfund-analytical-methods-multi-media-multi-0
Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use	https://www.epa.gov/clp/superfund-clp-analytical-services-guidance-documents
Hazardous Waste Test Methods (SW-846)	https://www.epa.gov/hw-sw846
Clean Water Act Analytical Methods	https://www.epa.gov/cwa-methods

PART A: GENERAL DATA REVIEW

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I. Preliminary Review

A preliminary review of the data should be performed, prior to performing the method-specific review (Part B). During this process, the necessary elements should be compiled to ensure all information needed for validation is available and to obtain an overview of the data.

This preliminary review should include, but is not limited to, the verification of the exact number of samples, their matrix type(s), assigned identifiers (IDs) and analyses. It should take into consideration all the documentation specific to the sample data package, which may include any modifications to the project-specific Quality Assurance Project Plan (QAPP), Standard Operating Procedures (SOPs), or United States Environmental Protection Agency (EPA) Superfund Contract Laboratory Program (CLP) Statement of Work (SOW) used to generate the data, the sampling documentation [e.g., Chain of Custody (COC) Records], the associated data package narrative, and other applicable documents.

Sampling events and data packages routinely contain unique field quality control (QC) samples that may affect the outcome of the review. These samples include field blanks (e.g., equipment blanks, rinse blanks), field duplicates, and Performance Evaluation (PE) samples that should be identified in the sampling records. The reviewer should verify that the following information is identified in the sampling records (e.g., COC Records, field logs, and/or applicable tables):

1. The party responsible for collecting the samples,
2. The complete list of samples with information on:
    a. Sample ID
    b. Sample matrix
    c. Field blanks (if applicable)
    d. Field duplicates (if applicable)
    e. Field spikes (if applicable)
    f. PE samples (if applicable)
    g. Sampling dates
    h. Sampling times
    i. Shipping dates
    j. Preservatives
    k. Types of analysis
    l. Laboratory

The laboratory's data package narrative is another source of general information which may include notable problems with matrices; insufficient sample volume for analysis or reanalysis; samples received in broken containers; preservation information, verified by the laboratory; example calculation(s) used to produce the results; and unusual events. The reviewer should also inspect email or telephone/communication logs in the data package detailing any discussion of sample logistics, preparation and/or analysis issues between the laboratory and project manager or other point of contact. The reviewer should also have a copy of the QAPP, or similar document for the project for which samples were analyzed, to assist in the validation.

For data obtained through the EPA 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 compliance with the technical and QC criteria in the applicable EPA CLP SOW, and 2) automated data validation based on the criteria in the EPA CLP National Functional Guidelines (NFG) for the applicable Superfund methods. When a choice of data qualifiers is presented during the data validation process, the qualifier that is more

protective of human health is selected. For example, the "J" qualifier, which designates a value as estimated, would be selected over the "R" qualifier, which designates a value as rejected. In addition, completeness checks are manually performed on the data in the Portable Document Format (PDF) version of the hardcopy. The results of the SEDD and PDF data review issues are subsequently included in a method compliance defect report that is provided to the laboratory and the data requester. The laboratory may then submit a reconciliation package for any missing items or to correct non-compliant data identified in the method compliance report. The automated data validation results are summarized in criteria-based NFG reports, which consist of various data summary reports (e.g., Initial Calibration Data Summary) generated from the SEDD, that are provided to the data users. The method compliance review and NFG reports can be accessed through the EXES Data Manager (EDM) via the Superfund Analytical Services Sample Management Office (SMO) Contract Laboratory Program Support System (CLPSS) Portal and may be used to assist with the validation process. EXES and EDM can be accessed via the Superfund Analytical Services SMO CLPSS Portal at: https://www.smoclpss.com.

II. Data Qualifier Definitions

The following table provides brief explanations of the 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 should accompany the data review in the Data Review Narrative.

General Table 1. Data Qualifiers and Definitions

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the adjusted detection limit or quantitation limit, as appropriate.
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.

NOTE: With familiarity of project data objectives and/or consultation with project staff, the reviewer should be able to refine the use of data qualifiers to avoid ambiguity. For example, if critical site decisions are to be made based on the data, the reviewer may decide to apply an "R" qualifier rather than a "UJ".

Although a "J+" or a "J-" may be seen as less ambiguous than a "J", the reviewer should reserve the application of directional bias indicators to those situations when there is an overwhelming influence in one direction. The exercise of professional judgment is critical, especially in situations where ambiguity exists due to opposing factors, to objectively interpret the effects of all factors.

III. Data Review Narrative

The reviewer should complete a Data Review Narrative, to include comments that address the problems identified during the review process and state the limitations of the data related to meeting project Data Quality Objectives (DQO). The sample identifiers, analytical methods, extent of the problem(s), and any assigned qualifiers should also be listed in the document. Note that QAPP, reference method or SOP-specified acceptance criteria may differ from the EPA CLP SOW-specified acceptance criteria on which the NFG data qualification recommendations are based. Therefore, the source of the criteria used for the data review and qualification should be clearly indicated. Additional information in the Data Review Narrative should include, but not be limited to, calculation checks, documentation of any approved deviations from the reference method and an explanation of any laboratory-assigned data qualifiers in the data. Finally, the process of reviewing and qualifying the data should be documented for future reference (i.e., using the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use) including the use of any professional judgment.

The Data Review Narrative, potentially including a summary form like the Inorganic Data Review Summary form (see Appendix B), should be provided with the laboratory data, marked with data qualifiers as necessary, to the appropriate recipient(s), including the designated project management personnel.

IV. Performance Evaluation (PE) Sample

A. Review Items

Laboratory Results Reports, sampling documentation (e.g., COC Records), sample receipt forms, preparation logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the recoveries of analytes of known concentrations in the PE sample(s). Data associated with PE samples can be used as an additional evaluation of measurement uncertainty or bias for field samples prepared along with PE samples.

C. Criteria

Matrix-specific PE samples should be analyzed utilizing the same analytical methods and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples, at a frequency to be determined by the data user or QAPP. PE samples should be prepared and analyzed together with the field samples in the data package for the sampling event, using the same procedures, reagents, and instrumentation. Measured concentrations in PE samples are compared to pre-defined acceptance criteria developed and supplied by the PE provider or otherwise appropriate acceptance criteria for the project.

D. Evaluation

1. Verify that the PE samples were prepared and analyzed with the field samples and/or field blanks in the data package, using the Laboratory Results Reports, preparation logs, and raw data.
2. Verify that the PE sample results are within the specified concentration or recovery limits using Laboratory Results Reports and any raw data.
3. If a significant number (e.g., half or more) of the analytes or any specific target analytes critical to the project in the PE samples fall outside of the acceptance limits in the PE sample(s), or if a number of false positive results are reported, evaluate the overall impact on the data. Consider all possible reasons for this finding, including laboratory procedures, changes in the analytical system, and the PE samples themselves.

E. Action

Refer to General Table 2 for the evaluation criteria and corresponding actions for detected and non-detected target analytes in the samples associated with deficient PE sample(s).

1. Obtain additional information from the laboratory if the PE sample was not prepared and analyzed with the field samples and/or field blanks. If a laboratory did not prepare or analyze the PE sample(s) provided with field samples and field blanks, or if a laboratory repeatedly fails to generate acceptable PE sample results for the same method and analyte(s), record the situation in the Data Review Narrative, and note it for designated project management personnel action.

NOTE: If the PE sample acceptance criteria are not met, the laboratory performance and measurement accuracy may be in question. For a PE sample that does not meet the technical acceptance criteria, the reviewer should consider applying the same interpretation to all samples prepared together. Qualification of field sample data based on PE sample performance may be most appropriate for those samples in which the analyte concentration is comparable to the PE sample concentration. Actions should apply only to specified target analytes that did not meet the PE sample acceptance criteria unless the failures indicate a problem with a broader scope.

2. Note the potential effects on the data due to out-of-control PE sample results in the Data Review Narrative.

General Table 2. PE Sample Actions

Criteria	Action	
	Detect	**Non-detect**
PE sample not prepared and analyzed with assigned field samples	Use professional judgment	Use professional judgment
PE sample results outside lower action limits provided with the PE sample or specified for the project	J-	R
PE sample results outside lower warning limits but inside lower action limits provided with the PE sample or specified for the project	J-	UJ
PE sample results within limits provided with the PE sample or specified for the project	No qualification	No qualification
PE sample results outside upper warning limits but inside upper action limits provided with the PE sample or specified for the project	J+	No qualification
PE sample results outside upper action limits provided with the PE sample or specified for the project	J+	No qualification

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

V. Field Quality Assurance and Quality Control (QA/QC)

A. Review Items

Laboratory Results Reports, sampling documentation (e.g., COC Records), instrument printouts, and other raw data from QA/QC samples in data package.

B. Objective

The objective is to use results from the analysis of field and project QA/QC samples such as field blanks and field duplicates to determine the validity of the analytical results.

C. Criteria

Criteria are determined by the data user or QAPP.

1. The frequency of these field and project QA/QC samples should be defined in the QAPP.
2. Performance criteria for these field and project QA/QC samples should also be defined in the QAPP.
3. The Relative Percent Difference (RPD) between field duplicates should fall within the specific limits in the QAPP or in the project-specific SOPs for data review. The limits may not apply when the sample and duplicate concentrations are less than 5x the Quantitation Limit (QL) or limit in the QAPP.
4. In the absence of other guidance, qualify associated samples for contaminants found in field blanks based on the criteria for Method Blanks (see the applicable method sections for blank actions).

D. Evaluation

1. Determine whether any non-conforming field QA/QC sample results may impact all samples in the project or only those directly associated (e.g., in the same data package, collected on the same day, prepared together, or contained in the same analytical sequence).
2. Verify precision by recalculating at least one RPD between field duplicates and provide this information in the Data Review Narrative. Also verify that the RPDs fall within the limits specified in the QAPP or project-specific SOPs for data review.
3. Determine whether RPD limits exceedance (poor precision) is the responsibility of the laboratory or may have resulted from sample non-homogeneity in the field. Laboratory observations of sample appearance, in the data package narrative, may become important in these situations.

E. Action

1. Any action should be in accordance with the project specifications and the criteria for acceptable field duplicate sample results.
2. Note where RPDs exceed criteria for field duplicate samples in the Data Review Narrative and for designated project management personnel action.
3. Note results greater than or equal to QLs in field blanks for designated project management personnel action.
4. In general, for QA/QC performance not within QAPP specification, qualify detects as estimated (J) and non-detects as estimated (UJ). The impact on overall data quality should be assessed after consultation with the data user and/or field personnel.

VI. Overall Assessment of Data

A. Review Items

Entire data package, data review results, and (if available) the QAPP and Sampling and Analysis Plan (SAP).

B. Objective

The objective is to provide the overall assessment on data quality, uncertainty, and bias.

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 should be quantitated according to the appropriate equations, as listed in the reference method. All sample results should be measured within the calibration range. Percent Solids (%Solids) should be properly used for all applicable matrix result calculations.

D. Evaluation

Examine the raw data to verify that the calculated sample results were correctly reported by the laboratory. Preparation logs, instrument printouts, etc., should be used to evaluate the final results reported in the data package.

1. Evaluate any technical problems that were not previously addressed.
2. Examine the raw data for anomalies (e.g., baseline shifts, omissions, illegibility).
3. Verify that the appropriate methods and amounts were used to prepare the samples for analysis. If reduced sample aliquot amounts were used, verify that any project-required sensitivity was not compromised and that the laboratory received prior approval.
4. Verify that there are no transcription or reduction errors (e.g., dilutions, %Solids, sample weights) on one or more samples. Recalculate the %Solids for one or more of the samples and verify that the calculated %Solids agree with that reported by the laboratory.
5. Verify that Detection Limits (DLs) are properly reported and that they are not greater than or equal to the respective QLs.
6. Verify that reported target analyte results fall within the calibrated range(s) of the instrument(s).
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 QAPP, focusing specifically on the acceptance or performance criteria, the SOPs, and communication with the project manager concerning the intended use and desired quality of these data.

NOTE: For data obtained from the EPA CLP, information regarding noncompliant analyses and data can be obtained from the NFG reports and may be used as part of the evaluation.

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 discussed in Data Review Part A and Data Review Part B.
2. Use professional judgment to qualify detects and non-detects if the Method Detection Limit (MDL) or DL is greater than or equal to the QL.
3. If a sample is not diluted properly when sample results exceeded the upper limit of the calibration range, qualify affected detects as estimated (J).
4. If the required analyses were not performed at the specified frequency and sequence and/or sufficient information was not provided for an analysis, notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. In the event that a reanalysis cannot be performed (e.g., sample holding times have expired, insufficient amount of remaining sample) or the relevant information is not available, use professional judgment to assess the existing data.
5. Write a brief Data Review Narrative (see Part A, Section III) to give the user an indication of the limitations of the analytical data. Note the issues reported in the data package narrative, calculation errors (if any), and the General Data Review (Part A) and Method-Specific Data

Review (Part B) performance criteria that are exceeded in this report. Also include the potential effects of such discrepancies on the data for designated project management personnel action.

6. If sufficient information on the intended use and required quality of the data is available, include an assessment of the usability of the data within the given context. This evaluation may be used as part of a formal Data Quality Assessment (DQA).
7. Document the process used for the data review and qualification in accordance with the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (see table in Section IV of Part A, titled Additional Information).

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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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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, raw data, and narrative in the data package, 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 shipping and storage conditions and the holding time of the sample.

C. Criteria

1. Samples received with pH ≥ 2 may be adjusted to pH < 2 by the laboratory. The laboratory must allow the sample to set for at least 16 hours after acid addition before rechecking the pH. Samples adjusted to pH < 2 by the laboratory do not require qualification.
2. The technical holding time is determined from the date of field sample collection, or the date that Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction is completed, to the date of analysis.
3. The technical holding time criteria for aqueous/water samples and TCLP/SPLP aqueous filtrate and leachate samples is 180 days or as specified in the Quality Assurance Project Plan (QAPP), preserved (with nitric acid) to pH < 2.
4. The technical holding time criteria for soil/sediment and waste samples is 180 days or as specified in the QAPP.
5. The technical holding time criteria for wipe samples is 180 days.

D. Evaluation

1. Review the data package narrative, sampling documentation, and sample receipt forms to determine if the samples were properly preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperature at receipt, pH), or if the pH was adjusted upon receipt. If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples were properly stored at the laboratory.
2. Verify that the analysis dates on the Laboratory Results Reports and the raw data are identical.
3. Establish the technical holding times by comparing the sample collection dates on the sampling documentation and the TCLP/SPLP extraction dates with the dates of analysis on the Laboratory Results Reports and the raw data.

E. Action

Refer to ICP-AES Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

If a discrepancy is found between the sample analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

ICP-AES Table 1. Preservation and Holding Time Actions

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with pH ≥ 2 and pH adjusted by laboratory	No qualification	No qualification
Aqueous/water samples received with pH ≥ 2 and pH not adjusted	J-	R
TCLP/SPLP leachates with pH ≥ 2 and pH not adjusted	J-	R
Technical Holding Time: Aqueous/water and TCLP/SPLP leachates > 180 days	J-	R
Technical Holding Time: Soil/sediment/waste/wipe samples > 180 days	J-	R
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

II. Calibration

A. Review Items

Laboratory initial calibration and calibration verification reports (if available), preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data in the data package.

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 should be successfully calibrated each time the instrument is set up, or as specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). The calibration date and time should be included in the raw data.

NOTE: A blank and the number of calibration standards specified in the QAPP or in the SOW should be used to establish each calibration curve. At least one of these standards should be at or below the Quantitation Limit (QL) in the QAPP or SOW but above the Detection Limit or the Method Detection Limit (MDL). Calibration standards at and above the QL should be continuous with none excluded to satisfy Quality Control (QC) requirements. All measurements should be within the instrument working range where the interelement correction factors are valid. A minimum of three replicate exposures or the number specified in the QAPP are required for standardization, for all QC samples, and for sample analyses. The average result of all the multiple exposures for the standardization, QC, and sample analyses should be used. The calibration curve may be fitted using linear regression or weighted linear regression, or other fits as specified in the QAPP. The curve may be forced through zero. For linear fits, the calibration curve should have a correlation coefficient greater than the value specified in the QAPP or in the SOW. The calculated percent differences (%Ds) or other specified statistical test values for all non-zero standards should fall within the limits in the QAPP or in the SOW.

2. Initial and Continuing Calibration Verification

   a. Initial Calibration Verification (ICV)

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

      ii. Analyses of the ICV should be conducted using a certified solution of the analytes from an independent standard source, at a concentration level other than that used for instrument calibration, and near the middle of the calibrated range (within ±30%).

      iii. The ICV solution should be analyzed at each analytical wavelength used for analysis.

      iv. The Percent Relative Standard Deviation (%RSD) of the replicate measurements of the ICV should not exceed 5% or the limit specified in the QAPP.

   b. Continuing Calibration Verification (CCV)

      i. To ensure accuracy during each analytical sequence, the CCV standard should be analyzed and reported for each wavelength used for the analysis of each analyte.

      ii. The CCV standard should be analyzed at the frequency specified in the QAPP, or every two hours during an analytical sequence. The CCV standard should also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

iii. The CCV standard should be prepared using the same source and in the same acid matrix as the calibration standards by combining compatible analytes at a concentration at or near the mid-level (within ±30%) of the respective calibration curve.

iv. The same CCV standard solution should be used throughout the analysis for a data package.

v. The %RSD of the replicate measurements of a CCV should not exceed 5% or the limit specified in the QAPP.

vi. The CCV should 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 and documented in the data package narrative, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

vii. An instrument blank should not be analyzed before the CCV.

D. Evaluation

1. Verify that the instrument was calibrated as specified in the QAPP or in the SOW and each time the instrument was set up, utilizing a blank and at least the minimum number of standards specified in the QAPP or in the SOW. Confirm that at least one of the calibration standards was analyzed at or below the QL in the QAPP or in the SOW but above the Detection Limit or MDL and that all subsequent calibration standards are consecutive with none removed to satisfy QC requirements. For linear fits, verify that the correlation coefficient of the calibration curve is greater than the value specified in the QAPP or in the SOW. Verify that the %Ds for all non-zero standards are within the SOW limits or that other statistical test values are within the limits specified in the QAPP.
2. Confirm that the measurements were within the working calibration range, and were the average result of at least the specified minimum number of replicate exposures.
3. Confirm that an instrument blank was not analyzed before the CCV.
4. 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.
5. Verify that the ICV/CCV %RSD does not exceed 5% or the limit specified in the QAPP.
6. Verify that the ICV and CCV %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

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

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

E. Action

Refer to ICP-AES Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient initial calibrations or calibration verification standards.

1. For initial calibrations or ICV standard analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCV standard analyses that do not meet the technical criteria, apply the actions 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.

3. 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 the QL but above the MDL), qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

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

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

ICP-AES Table 2. Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed or not performed at specified frequency	R	R
Calibration incomplete (insufficient number of standards or required concentrations missing)	J or R	UJ or R
For linear fits, correlation coefficient < 0.995	J	UJ
%D outside ±30% or other specified statistical test values outside limits	J	UJ
ICV/CCV not performed at specified frequency	J	UJ
ICV/CCV %R < 75%	J- or R	UJ or 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%	J+ or R	No qualification
ICV/CCV %RSD > 5%	J	UJ
Instrument blank analyzed prior to CCV	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

III. Blanks

A. Review Items

Laboratory blanks reports (if available), preparation logs, calibration standard logs, instrument logs, and raw data in the data package.

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) should be analyzed at each wavelength used for analysis after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) standard during the initial calibration of the instrument. The ICB result (absolute value) should not be greater than or equal to the Quantitation Limit (QL) of each analyte for which analysis is performed.
3. A Continuing Calibration Blank (CCB) should be analyzed at each wavelength used for the analysis, immediately after every Continuing Calibration Verification (CCV) standard. The CCB should be analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) during the analytical sequence. The CCB should 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) should not be greater than or equal to the QL of each analyte for which analysis is performed.
4. At least one Preparation Blank should be prepared and analyzed for each matrix, with every data package, 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 greater than or equal to the QL, the lowest concentration of that analyte in the associated samples should be $\geq$ 10x the Preparation Blank concentration. Otherwise, all associated samples with the analyte's concentration < 10x the Preparation Blank concentration, and $\geq$ the QL, 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 $\leq$ (-QL), all associated samples with the analyte's concentration < 10x the QL should be redigested and reanalyzed.
7. At least one Leachate Extraction Blank (LEB) should be prepared and analyzed for each batch of samples extracted by Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP). The LEB consists of reagent water processed through the extraction procedure. Post-extraction, the LEB should 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 and LEBs were prepared and analyzed as appropriate for the data package (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. For an ICB or a CCB, verify that if the absolute value of any target analyte was greater than or equal to the QL, the analysis was terminated, the problem corrected and documented in the data

package narrative, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

3. For a Preparation Blank, verify that if the concentration of any target analyte was greater than or equal to the QL, all associated samples with the analyte's concentration ≥ the QL but < 10x the Preparation Blank concentration were redigested and reanalyzed for that analyte. Verify that if a concentration was ≤ (-QL) in a Preparation Blank, all associated samples with the analyte's concentration < 10x the QL were redigested and reanalyzed.
4. Evaluation of field and equipment blanks should also be performed according to the QAPP or appropriate guidance.

E. Action

Refer to ICP-AES Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient blanks.

1. For ICB analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCB analyses that do not meet the technical criteria, apply the actions 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.
3. For Preparation Blank analyses that do not meet the technical criteria, apply the actions to all associated samples prepared in the same preparation batch. For LEBs that do not meet the technical criteria, apply the actions to all associated samples extracted in the same extraction batch.
4. Actions 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.
5. If the absolute value of an ICB or a CCB result is ≥ QL, the analysis should have been terminated and the affected samples re-analyzed. If samples were not re-analyzed, qualify as described in Table 3 below.
6. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and ≥ QL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, qualify as described in Table 3 below.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute analysis, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. For blank results ≤ (-MDL) but > (-QL), the possibility of false negatives exists.

NOTE: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment or waste sample results reported in the Laboratory Results Reports will not be on the same basis (units, dilution) as the calibration blank data. 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 or waste samples for comparison purposes.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

ICP-AES Table 3. Blank Actions

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Not analyzed at the specified frequency	Non-detect	UJ
		Detect	J
ICB/CCB	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
ICB/CCB	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification
ICB/CCB	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < ICB/CCB Result	Report at ICB/CCB Result and qualify U
		≥ ICB/CCB Result	J+ or no qualification
ICB/CCB	≤ (-QL)	Non-detect	UJ or R
		Detect < QL	J-
		≥ QL	J-
Preparation Blank/LEB	Not analyzed at specified frequency	Non-detect	UJ
		Detect	J
Preparation Blank/LEB/ Field Blank	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
Preparation Blank/LEB/ Field Blank	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification
Preparation Blank/LEB/ Field Blank	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < 10x the Preparation Blank/LEB Result	Report at Preparation Blank/LEB Result and qualify J+ or R
		≥ 10x the Preparation Blank/LEB Result	No qualification
Preparation Blank/LEB/ Field Blank	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-
		≥ QL but < 10x QL	J-

Blank Type	Blank Result	Sample Result	Action
		≥ 10x QL	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Interference Check Sample

A. Review Items

Laboratory interference checks reports (if available), instrument printouts and raw data in the data package.

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 should be analyzed undiluted at the beginning of each sample analysis sequence. The ICS is not to be analyzed prior to the Initial Calibration Verification (ICV) standard, and should be immediately followed by a Continuing Calibration Verification (CCV) standard, followed by a Continuing Calibration Blank (CCB).
3. Results for the analysis of the ICS Solution A should fall within the control limits specified in the Quality Assurance Project Plan (QAPP), or ±QL [or ±15% 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 should fall within the control limits specified in the QAPP or in the SOW.
5. If the value of an ICS result exceeds the limit in the QAPP or in the Statement of Work (SOW), the analysis should be terminated, the problem corrected and documented in the data package narrative, the instrument recalibrated, the new calibration then reverified, and all analytical samples since the last compliant ICS reanalyzed.
6. The ICS solutions should be prepared using certified standards with the interferent and analyte concentrations at the levels specified in the method.

D. Evaluation

1. Verify, using 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 greater than the Detection Limit or the Method Detection Limit (MDL) for those analytes that are not present in the ICS solution.
3. Verify that the %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

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

True (value) = Concentration of each analyte or interferent in ICS Solution A or ICS Solution AB

4. If the value of an ICS result exceeds the limits specified in the QAPP, the ±QL, or ±15% of the true value (whichever is greater) criteria, and the laboratory failed to terminate the analysis and

take the appropriate corrective action, note this and record the situation in the Data Review Narrative. Use professional judgment to assess the data.

E. Action

Refer to ICP-AES Table 4 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient ICSs.

1. For an ICS analysis that does not meet the technical criteria, apply the actions to all samples reported from the analytical sequence.

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/waste or wipe samples are evaluated.

2. 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 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 QL and > 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).

3. If the raw data does not contain results for the interferents, note this in the Data Review Narrative. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be 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.

ICP-AES Table 4. Interference Check Actions

Criteria	Action	
	Detect	**Non-detect**
ICS not analyzed	R	R
ICS not analyzed in the specified sequence	J	UJ
ICSAB %R < 50%	J-	R
ICS %R 50-84% [or ICS found value is < (true value – QL), whichever is lower]	J-	UJ
ICS %R 85-115%	No qualification	No qualification
ICS %R 116-150% [or ICS found value is > (true value + QL), whichever is greater]	J+	No qualification
ICS %R > 150%	J+	No qualification
ICSA results ≥ DLs or MDLs, but not present in ICS (potential false positive)	J+	No qualification
Negative ICSA results, but not present in ICS (potential false negative)	J- for results < 10x (|negative sample result|)	UJ

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

V. Laboratory Control Sample

A. Review Items

Laboratory LCS reports (if available), preparation logs, instrument printouts, and raw data in the data package.

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/waste, and wipe LCSs should 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.
2. One LCS should be prepared and analyzed for every group of aqueous/water or soil/sediment/waste samples in a data package, or with each batch of samples digested, whichever is more frequent. The LCS should be spiked such that the final digestate contains each analyte at the level specified in the Quality Assurance Project Plan (QAPP) or at 2x the Quantitation Limit (QL) for the associated matrix.
3. One LCS should be prepared and analyzed for each group of wipe samples in a data package, or with each batch of wipe samples digested, whichever is more frequent. The wipe LCS should be spiked such that the final digestate contains each analyte at the level specified in the QAPP or at 2x the QL.
4. All LCS Percent Recoveries (%Rs) should fall within the control limits in the QAPP or in the Statement of Work (SOW). If the %R for the aqueous/water and soil/sediment/waste LCS falls outside of the control limits, the analysis should be terminated, the problem corrected and documented in the data package narrative, 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 the laboratory reports, preparation logs, and raw data, that the appropriate number of required LCSs were prepared and analyzed for the data package.
2. Verify that all results for each analyte fall within the established control limits.
3. Verify that the %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of each analyte measured in the analysis of the LCS
True (value) = Concentration of each analyte in the LCS

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

E. Action

Refer to ICP-AES Table 5 for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient LCSs. For an LCS analysis that does not meet the technical criteria, apply the actions to all samples in the same preparation batch.

Matrix spike data can be reviewed to determine batch quality if an LCS was not prepared and analyzed with the samples.

ICP-AES Table 5. LCS Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not prepared with samples	J	UJ
LCS not prepared at specified concentrations	J	UJ
Aqueous/water and soil/sediment/waste %R < 40% (< 20% Ag, Sb)	J-	R
Aqueous/water and soil/sediment/waste %R 40-69% (20-49% Ag, Sb)	J-	UJ
Aqueous/water and soil/sediment/waste %R 70-130% (50-150% Ag, Sb)	No qualification	No qualification
Aqueous/water and soil/sediment/waste %R 131-150% (151-170% Ag, Sb)	J+	No qualification
Aqueous/water and soil/sediment/waste %R > 150% (170% Ag, Sb)	R	No qualification
Wipe %R < 40% (< 20% Ag, Sb)	J-	R
Wipe %R 40-69% (20-49% Ag, Sb)	J-	UJ
Wipe %R 70-130% (50-150% Ag, Sb)	No qualification	No qualification
Wipe %R > 130% (>150% Ag, Sb)	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VI. Duplicate Sample Analysis

A. Review Items

Data package Cover Page, laboratory duplicate reports (if available), instrument printouts, preparation logs, and raw data in the data package.

B. Objective

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

C. Criteria

1. Field samples should be used as source samples for duplicate analysis.
2. At least one duplicate sample should be prepared and analyzed from each group of samples of a similar matrix type (e.g., aqueous/water or soil/sediment/waste) or for each data package. Duplicates are not required for wipe samples. Duplicates cannot be averaged for reporting on the Laboratory Results Report. Additional duplicate sample analyses may be required. Alternately, the data user may require that a specific sample be used for the duplicate sample analysis.
3. The Relative Percent Difference (RPD) control limit specified in the Quality Assurance Project Plan (QAPP) or of 20% should be used for original and duplicate sample values ≥ 5x the QL.
4. For samples analyzed under the Statement of Work (SOW), a control limit of the Quantitation Limit (QL) should be used if either the sample or duplicate value is < 5x the QL.

D. Evaluation

1. Verify, from the data package Cover Page, laboratory report, preparation log and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed.
2. Verify, using the raw data, that all duplicate results for each analyte fall within the established control limits.
3. Verify that the duplicate analysis was performed on a field sample.
4. Verify that the RPD values are correct by recalculating one or more of the RPDs using the raw data and the following equation:

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

Where,

S = Sample Result (original)
D = Duplicate Result

NOTE: When the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only for calculating the RPD. This will always yield an RPD of 200%.

E. Action

Refer to ICP-AES Table 6 for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient duplicates.

1. For a duplicate sample analysis that does not meet the technical criteria, apply the actions 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 data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the duplicate sample should be qualified.
2. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.
3. For high RPDs (i.e., > 100%), use professional judgment to qualify the data as this may be indicative of a sampling problem.

ICP-AES Table 6. Duplicate Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Duplicate analysis not performed at the specified frequency.	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%*	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
For samples analyzed under the SOW, original sample or duplicate sample result < 5x the QL (including non-detects) and absolute difference between sample and duplicate > QL*	J	UJ
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* 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**, the QAPP or project-specific SOPs for data review may allow the use of less restrictive criteria (e.g., 35% RPD, 2x QL) to be assessed against duplicate soil samples.

VII. Spike Sample Analysis

A. Review Items

Data package Cover Page, laboratory matrix spike reports (if available), instrument printouts, preparation logs, and raw data in the data package.

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. Field samples should be used as source samples for matrix spike analysis.
2. At least one spiked sample (pre-digestion) should be prepared and analyzed from each group of samples with a similar matrix type (e.g., aqueous/water or soil/sediment/waste), or for each data package. Matrix Spikes are not required for wipe samples. Additional matrix spike sample analyses may be required. Alternately, the data user may require that a specific sample be used for the matrix spike sample analysis.
3. The spike Percent Recovery (%R) should be within the established acceptance limits. However, for samples analyzed under the Statement of Work (SOW), spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data should be reported unqualified, even if the %R does not meet the acceptance criteria.
4. For sample analyzed under the SOW, when the spike recovery falls outside of the control limits and the sample result is < 4x the spike added, a post-digestion spike analysis should be performed for those analytes that do not meet the specified criteria. An aliquot of the remaining unspiked sample should be spiked at 2x the indigenous level or 2x the Quantitation Limit (QL), whichever is greater.

NOTE: Post-digestion spikes are not required for Antimony (Sb) and Silver (Ag).

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

NOTE: The final spike concentrations required for the various target analytes are presented in the methods described in the Quality Assurance Project Plan (QAPP) or in the SOW.

D. Evaluation

1. Verify, using the data package Cover Page, laboratory reports, preparation log and raw data, that the appropriate number of required spiked samples was prepared and analyzed.
2. Verify the matrix spike analysis was performed on a field sample.
3. Verify, using the raw data, that all matrix spike 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. Verify that the %R values for the matrix spike are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

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

NOTE: When the Sample Result is reported as a non-detect, use SR = 0 only for calculating the %R.

E. Action

Refer ICP-AES Table 7 for the evaluation criteria and corresponding actions for detected and non-detected target and spike analyte results in the samples associated with deficient matrix spikes.

1. For a matrix spike sample analysis that does not meet the technical criteria, apply the actions 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 data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.
2. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

ICP-AES Table 7. Spike Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike analysis not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
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

Criteria	Action	
	Detect	**Non-detect**
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 (not required for Ag and Sb)	J-	R
Matrix Spike %R 30-74% No post-digestion spike performed (not required for Ag and Sb)	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 (not required for Ag and Sb)	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

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**, the QAPP or the project-specific SOPs for data review 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

Laboratory serial dilution reports (if available), instrument printouts, and raw data in the data package.

B. Objective

The objective of the serial dilution analysis is to determine if significant physical or chemical interferences exist due to sample matrix.

C. Criteria

1. An Inductively Coupled Plasma (ICP) Serial Dilution analysis should be performed on a sample from each group of samples with a similar matrix type (e.g., aqueous/water or soil/sediment/waste) or for each data package, whichever is more frequent.
2. An ICP Serial Dilution analysis is not required for wipe samples.
3. Field samples should be used as source samples for the ICP Serial Dilution analysis.
4. If the analyte concentration is sufficiently high [concentration in the original sample is > 50x the Method Detection Limit (MDL) that is calculated for the sample or the limit in the Quality Assurance Project Plan (QAPP)], the Percent Difference (%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 ≥ Quantitation Limit (QL)] should be ≤ 20%.

D. Evaluation

1. Verify that the serial dilution analysis was performed on a field sample.
2. Verify that the %D values are correct by recalculating one or more of the %Ds using the raw data and the following equation:

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

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 from the original sample), possibly due to high levels of dissolved solids in the sample, ionization effects, etc.

E. Action

Refer to ICP-AES Table 8 for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient serial dilution analyses.

1. For a serial dilution sample analysis that does not meet the technical criteria, apply the actions 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 data package. Two possible determinations are: 1) only some of the samples in the data package 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.
2. Note the potential effects on the reported data in the Data Review Narrative.

ICP-AES Table 8. Serial Dilution Actions

Criteria	Action	
	Detect	**Non-detect**
Serial Dilution not performed at the specified frequency	J	UJ
Sample concentration > 50x MDL, serial dilution sample concentration ≥ QL, and %D > 20%*	J	No qualification
Sample concentration >50x MDL, serial dilution sample concentration ≥ QL, and %D ≤ 20%	No qualification	No qualification
Sample concentration > 5x QL and serial dilution sample concentration < QL	No qualification	No qualification
Interferences present	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, the QAPP or the project-specific SOPs for data review may allow the use of less restrictive criteria (e.g., %D > 25%) to be assessed against serial dilution soil samples.

IX. Target Analyte Quantitation

A. Review Items

Laboratory Result Reports, sample preparation sheets, data package narrative, instrument printouts and raw data.

B. Objective

The objective is to ensure that the reported results and quantitation limits for target analytes reported by the laboratory are accurate and sufficient to meet requirements.

C. Criteria

Final target analyte results and quantitation limits should be calculated according to the correct equations, taking into account amount of sample prepared, final digestate volume, dilution factor, and percent solids, as appropriate.

D. Evaluation

1. Verify that the results for all positively identified target analytes are calculated and reported by the laboratory according to the equations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Verify that the reported Quantitation Limits (QLs) for non-detected target analytes are calculated and reported by the laboratory according to the equations in the QAPP or in the SOW.
3. Verify that all reported results and QLs have been adjusted to reflect percent solids, original sample mass/volume, and any applicable dilutions.

E. Action

1. If sample results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).
2. If the sample percent solids is < 30%, check if the sample was prepared at greater mass to maintain the QLs. Use professional judgment when this was not completed.

F. Example Equations

1. **Aqueous/Water and TCLP/SPLP Sample Concentration**

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

Where,

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

2. **Soil/Sediment and Waste Sample Concentration**

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

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
DF = Dilution Factor

3. **Wipe Mass**

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

Where,

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

4. **Adjusted DL (or MDL)/Adjusted QL**

To calculate the adjusted Detection Limit (DL) or adjusted Quantitation Limit (QL) for aqueous/water or TCLP/SPLP leachate samples, substitute the value of the DL or QL, in the appropriate units, into the "C" term in the equation above.

Calculate the adjusted DL or adjusted QL for soil/sediment samples as follows:

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

Where,

C = Detection Limit (DL) or Quantitation Limit (QL) (mg/kg)
W_M = Minimum method required aliquot amount (g)
W = Initial aliquot amount (g)
V_M = Method required final sample digestion volume (mL)
V_f = Final digestion volume (mL)
S = % Solids/100
DF = Dilution Factor

5. **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

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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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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, raw data, and narrative in the data package, 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 shipping and storage conditions and the holding time of the sample.

C. Criteria

1. Samples received with pH ≥ 2 may be adjusted to pH < 2 by the laboratory. The laboratory must allow the sample to set for at least 16 hours after acid addition before rechecking the pH. Samples adjusted to pH < 2 by the laboratory do not require qualification.
2. The technical holding time is determined from the date of sample collection to the date of analysis.
3. The technical holding time criteria for aqueous/water samples is 180 days or as specified in the Quality Assurance Project Plan (QAPP), preserved (with nitric acid) to pH < 2.
4. The technical holding time criteria for soil/sediment/waste samples is 180 days or as specified in the QAPP.

D. Evaluation

1. Review the data package narrative, sampling documentation, and sample receipt forms to determine if the samples were properly preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperature at receipt, pH), or if the pH was adjusted upon receipt. If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples were properly stored at the laboratory.
2. Verify that the analysis dates on the Laboratory Results Reports and the raw data are identical.

   Establish the technical holding times by comparing the sample collection dates on the sampling documentation with the dates of analysis on the Laboratory Results Reports and the raw data.

E. Action

Refer to ICP-MS Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

If a discrepancy is found between the sample analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

ICP-MS Table 1. Preservation and Holding Time Actions

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with pH ≥ 2 and pH adjusted by laboratory	No qualification	No qualification
Aqueous/water samples received with pH ≥ 2 and pH not adjusted	J-	R
Technical Holding Time: Aqueous/water samples > 180 days	J-	R
Technical Holding Time: Soil/sediment/waste samples > 180 days	J-	R
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

II. Tune Analysis

A. Review Items

Laboratory instrument performance check (Tune) reports (if available), instrument printouts and raw data in the data package.

B. Objective

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

C. Criteria

1. Prior to calibration, the laboratory should analyze or scan the ICP-MS tuning solution, containing the elements and concentrations specified in the Quality Assurance Project Plan (QAPP) or 100 µg/L of Beryllium (Be), Magnesium (Mg), Cobalt (Co), Indium (In), and Lead (Pb), at least five times consecutively. The solution should contain all required isotopes of the specified elements. The laboratory should 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 u over the range of 6-210 u.
2. The Percent Relative Standard Deviation (%RSD) of the absolute signals for all analytes in the tuning solution should be less than the value specified in the QAPP or in the Statement of Work (SOW).

D. Evaluation

1. Verify, using the raw data 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, that the resolution of the mass calibration falls within the limits for each isotope of each analyte.
3. Verify, using the raw data, that the %RSD is less than or equal to the limit for each isotope of each analyte.
4. Verify that the average mass and %RSD values are correct by recalculating one or more of the average masses and %RSDs for an isotope using the raw data and the following equations:

Mean Value

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

Where,

$\bar{X}$ = Mean Value
X_i = Individual replicate mass reading
n = Number of replicates

Percent Relative Standard Deviation

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

Where,

%RSD = Percent RSD
SD = Standard Deviation of replicates
$\bar{X}$ = Mean value of replicates

E. Action

Refer to ICP-MS Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient ICP-MS Tunes. For ICP-MS tunes that do not meet the technical criteria, apply the actions to all samples reported from the analytical sequence.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

ICP-MS Table 2. Tune Actions

Criteria	Action	
	Detect	**Non-detect**
Tune not performed	R	R
Tune not performed with required isotopes and/or number of scans	J or R	UJ or R
Resolution of mass calibration not within 0.1 u	J	UJ
%RSD > 5%	J	UJ
Tune properly analyzed with required isotopes, mass resolution and %RSD within specified limits	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

III. Calibration

A. Review Items

Laboratory initial calibration and calibration verification reports (if available), preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data in the data package.

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 should be successfully calibrated each time the instrument is set up, or as specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). The calibration date and time should be included in the raw data.

NOTE: A blank and the number of calibration standards specified in the QAPP or in the SOW should be used to establish each calibration curve. At least one of these standards should be at or below the Quantitation Limit (QL) in the QAPP or in the SOW, but above the Detection Limit or the Method Detection Limit (MDL). Calibration standards at and above the QL should be continuous with none excluded to satisfy QC requirements. All measurements should be within the instrument working range. A minimum of three replicate scans or the number specified in the QAPP are required for standardization, for all Quality Control (QC) samples, and for sample analyses. The average result of all the multiple scans for the standardization, QC, and sample analyses should be used. The calibration curve may be fitted using linear regression or weighted linear regression, or other fits as specified in the QAPP. The curve may be forced through zero. For linear fits, the calibration curve should have a correlation coefficient greater than the value specified in the QAPP or in the SOW. The calculated percent differences (%Ds) or other specified statistical test values for all non-zero standards should fall within the limits in the QAPP or in the SOW.

2. Initial and Continuing Calibration Verification

   a. Initial Calibration Verification (ICV)

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

      ii. Analyses of the ICV should be conducted using a certified solution of the analytes from an independent standard source, at a concentration level other than that used for instrument calibration, and near the middle of the calibrated range (within ±30%).

      iii. The ICV solution should be analyzed at each analytical mass used for analysis.

      iv. The Percent Relative Standard Deviation (%RSD) of the replicate measurements of the ICV should not exceed 5% or the limit specified in the QAPP.

   b. Continuing Calibration Verification (CCV)

      i. To ensure accuracy during each analytical sequence, the CCV should be analyzed and reported for each mass used for the analysis of each analyte.

ii. The CCV standard should be analyzed at the frequency specified in the QAPP or every two hours during an analytical sequence. The CCV standard should also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

iii. The CCV standard should be prepared using the same source and in the same acid matrix as the calibration standards by combining compatible analytes at a concentration at or near the mid-level (within ±30%) of the respective calibration curve.

iv. The same CCV standard solution should be used throughout the analysis for a data package.

v. The %RSD of the replicate measurements of a CCV should not exceed 5% or the limit specified in the QAPP.

vi. The CCV should 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 and documented in the data package narrative, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

vii. An instrument blank should not be analyzed before the CCV.

D. Evaluation

1. Verify that the instrument was calibrated as specified in the QAPP or in the SOW and each time the instrument was set up, utilizing a blank and at least the minimum number of standards specified in the QAPP or in the SOW. Confirm that at least one of the calibration standards was analyzed at or below the QL in the QAPP or in the SOW but above the Detection Limit or MDL and that all subsequent calibration standards are consecutive with none removed to satisfy QC requirements. For linear fits, verify that the correlation coefficient of the calibration curve is greater than the value specified in the QAPP or in the SOW. Verify that the %Ds for all non-zero standards are within the SOW limits or that other statistical test values are within the limits specified in the QAPP.
2. Confirm that the measurements were within the working calibration range, and were the average result of at least the specified minimum number of 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. Verify that the ICV/CCV %RSD does not exceed 5% or the limit specified in the QAPP.
5. Verify that the ICV or CCV %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

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

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

E. Action

Refer to ICP-MS Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient initial calibrations or calibration verification standards.

1. For initial calibrations or ICV standard analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

2. For CCV standards analyses that do not meet the technical criteria, apply the actions 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.
3. 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 the QL but above the MDL), qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

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

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

ICP-MS Table 3. Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed or not performed at specified frequency	R	R
Calibration incomplete (insufficient number of standards or required concentrations missing)	J or R	UJ or R
For linear fits, the correlation coefficient < 0.995	J	UJ
%D outside ±30% or other specified statistical test values outside limits	J	UJ
ICV/CCV not performed at specified frequency	J	UJ
ICV/CCV %R < 75%	J- or R	UJ or 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%	J+ or R	No qualification
ICV/CCV %RSD > 5%	J	UJ
Instrument blank analyzed prior to CCV	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Blanks

A. Review Items

Laboratory blanks reports (if available), preparation logs, calibration standard logs, instrument logs, and raw data in the data package.

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) should be analyzed at each mass used for analysis after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) standard during the initial calibration of the instrument. The ICB result (absolute value) should not be greater than or equal to the Quantitation Limit (QL) of each analyte for which analysis is performed.
3. A Continuing Calibration Blank (CCB) should be analyzed at each mass used for the analysis, immediately after every Continuing Calibration Verification (CCV) standard. The CCB should be analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) during the analytical sequence. The CCB should 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) should not be greater than or equal to the QL of each analyte for which analysis is performed.
4. At least one Preparation Blank should be prepared and analyzed for each matrix, with every data package, 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 greater than or equal to the QL, the lowest concentration of that analyte in the associated samples should be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with the analyte's concentration < 10x the Preparation Blank concentration and ≥ the QL 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 ≤ (-QL), all associated samples with the analyte's concentration < 10x the QL 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 analysis; and Preparation Blanks were prepared and analyzed as appropriate for the data package (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. For an ICB or a CCB, verify that if the absolute value of any target analyte was greater than or equal to the QL, the analysis was terminated, the problem corrected and documented in the data package narrative, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.
3. For a Preparation Blank, verify that if the concentration of any target analyte was greater than or equal to the QL, all associated samples with the analyte's concentration ≥ the QL but < 10x the Preparation Blank concentration were redigested and reanalyzed for that analyte. Verify that if a

concentration was ≤ (-QL) in a Preparation Blank, all associated samples with the analyte's concentration < 10x the QL were redigested and reanalyzed.

4. Evaluation of field and equipment blanks should be performed according to the QAPP or appropriate guidance.

E. Action

Refer to ICP-MS Table 4 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient blanks.

1. For ICB analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCB analyses that do not meet the technical criteria, apply the actions 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.
3. For Preparation Blank analyses that do not meet the technical criteria, apply the actions to all associated samples prepared in the same preparation batch.
4. 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.
5. If the absolute value of an ICB or a CCB result is ≥ QL, the analysis should have been terminated and the affected samples re-analyzed. If samples were not re-analyzed, qualify as described in Table 4 below.
6. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and ≥ QL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, qualify as described in Table 4 below.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute run, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. For blank results ≤ (-MDL) but > (-QL), the possibility of false negative exists.

NOTE: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment or waste sample results reported Laboratory Results Reports will not be on the same basis (units, dilution) as the calibration blank data. 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 or waste samples for comparison purposes.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

ICP-MS Table 4. Blank Actions

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Not analyzed at the specified frequency	Non-detect	UJ
		Detect	J
ICB/CCB	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U

Blank Type	Blank Result	Sample Result	Action
		≥ QL	J+ or no qualification
ICB/CCB	≤ (-MDL) but > (-QL)	Non-Detect	UJ
		Detect	J- or no qualification
ICB/CCB	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < ICB/CCB Result	Report at ICB/CCB Result and qualify U
		≥ ICB/CCB Result	J+ or no qualification
ICB/CCB	≤ (-QL)	Non-detect	UJ or R
		Detect < QL	J-
		≥ QL	J-
Preparation Blank	Not analyzed at specified frequency	Non-detect	UJ
		Detect	J
Preparation Blank/Field Blank	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
Preparation Blank/Field Blank	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification
Preparation Blank/Field Blank	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and qualify J+ or R
		≥ 10x the Preparation Blank Result	No qualification
Preparation Blank/Field Blank	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-
		≥ QL but < 10x QL	J-
		≥ 10x QL	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifier.

V. Interference Check Sample

A. Review Items

Laboratory interference checks reports (if available), instrument printouts and raw data in the data package.

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 should be analyzed undiluted at the beginning of each sample analysis sequence and every 12 hours during the analytical sequence. The ICS is not to be analyzed prior to the Initial Calibration Verification (ICV) standard, and should be immediately followed by a Continuing Calibration Verification (CCV) standard, followed by a Continuing Calibration Blank (CCB).
3. Results for the analysis of the ICS Solution A should fall within the control limits specified in the QAPP, or ±2x the Quantitation Limit (QL) [or ±15% 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 should fall within the control limits specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
5. If the value of an ICS result exceeds the limit in the QAPP or in the SOW, the analysis should be terminated, the problem corrected and documented in the data package narrative, the instrument recalibrated, the new calibration then reverified, and all analytical samples analyzed since the last compliant ICS reanalyzed.
6. The ICS solutions should be prepared using certified standards with the interferent and analyte concentrations at the levels specified in the method.

D. Evaluation

1. Verify, using 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 greater than the Detection Limit or the Method Detection Limit (MDL) for those analytes that are not present in the ICS solution.
3. Verify that the ICS Percent Recovery (%R) values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

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

True (value) = Concentration of each analyte or interferent in ICS Solution A or ICS Solution AB

4. If the value of an ICS result exceeds the limits specified in the QAPP or ±2x the QL, or ±15% of the true value (whichever is greater) criteria, and the laboratory failed to terminate the analysis and take the appropriate corrective action, note this and record the situation in the Data Review Narrative. Use professional judgment to assess the data.

E. Action

Refer to ICP-MS Table 5 below for the evaluation criteria and corresponding actions for detected and non-detected target analytes results in the samples associated with deficient ICSs.

1. For an ICS analysis that does not meet the technical criteria, apply the actions to all samples reported from the analytical sequence.

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/waste samples are evaluated.

2. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be 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.

ICP-MS Table 5. Interference Check Actions

Criteria	Action	
	Detect	**Non-detect**
ICS not analyzed	R	R
ICS not analyzed in specified sequence	J	UJ
ICSAB %R < 50%	J-	R
ICS %R 50-84% [or ICS found value is < (true value – 2x QL), whichever is lower]	J-	UJ
ICS %R 85-115%	No qualification	No qualification
ICS %R 116-150% [or ICS true value is > (true value + 2x QL), whichever is greater]	J+	No qualification
ICS %R > 150%	J+	No qualification
ICSA results ≥ DLs or MDLs, but not present in ICS (potential false positives)	J+	No qualification
Negative ICSA results, but not present in ICS (potential false negatives)	J- for results < 10x (|negative sample result|)	UJ

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VI. Laboratory Control Sample

A. Review Items

Laboratory LCS reports (if available), preparation logs, instrument printouts, and raw data in the data package.

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/waste LCSs should 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 should be prepared and analyzed for every group of aqueous/water or soil/sediment/waste samples in a data package, or with each batch of samples digested, whichever is more frequent. The LCS should be spiked such that the final digestate contains each analyte at the level specified in the Quality Assurance Project Plan (QAPP) or at 2x the Quantitation Limit (QL) for the associated matrix.
   b. All LCS Percent Recoveries (%Rs) should fall within the control limits in the QAPP or in the Statement of Work (SOW). If the %R falls outside of the control limits, the analysis should be terminated, the problem corrected and documented in the data package narrative, and the samples prepared with that LCS redigested and reanalyzed.

D. Evaluation

1. Verify, using the laboratory reports, preparation logs, and raw data, that the appropriate number of required LCSs were prepared and analyzed for the data package.
2. Verify that all results for each analyte fall within the established control limits.
3. Verify that the %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of each analyte measured in the analysis of the LCS

True (value) = Concentration of each analyte in the LCS

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

E. Action

Refer to ICP-MS Table 6 for the evaluation criteria and corresponding actions for detected and non-detected target analytes in the samples associated with deficient LCSs. For an LCS analysis that does not meet the technical criteria, apply the actions to all samples in the same preparation batch.

Matrix spike data can be reviewed to determine batch quality if an LCS was not prepared and analyzed with the samples.

ICP-MS Table 6. LCS Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not prepared with samples	J	UJ
LCS not prepared at specified concentrations	J	UJ
Aqueous/water and soil/sediment/waste %R < 40%	J-	R
Aqueous/water and soil/sediment/waste %R 40-69%	J-	UJ
Aqueous/water and soil/sediment/waste %R 70-130%	No qualification	No qualification
Aqueous/water and soil/sediment/waste %R 131-150%	J+	No qualification
Aqueous/water and soil/sediment/waste %R > 150%	R	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VII. Duplicate Sample Analysis

A. Review Items

Data Package Cover Page, laboratory duplicate reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Field samples should be used as source samples for duplicate analysis.
2. At least one duplicate sample should be prepared and analyzed from each group of samples of a similar matrix type (e.g., aqueous/water or soil/sediment/waste) or for each data package. Duplicates cannot be averaged for reporting on the Laboratory Results Report. Additional duplicate sample analyses may be required. Alternately, the data user may require that a specific sample be used for the duplicate sample analysis.
3. The Relative Percent Difference (RPD) control limit specified in the Quality Assurance Project Plan (QAPP) or of 20% should be used for original and duplicate sample values ≥ 5x the QL.
4. For samples analyzed under the Statement of Work (SOW), a control limit of the Quantitation Limit (QL) should be used if either the sample or duplicate value is < 5x the QL.

D. Evaluation

1. Verify, from the data package Cover Page, laboratory reports, preparation log and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed.
2. Verify, using the raw data, that all duplicate results for each analyte fall within the established control limits.
3. Verify that the duplicate analysis was performed on a field sample.
4. Verify that the RPD values are correct by recalculating one or more of the RPDs using the raw data and the following equation:

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

Where,

S = Sample Result (original)
D = Duplicate Result

NOTE: When the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only for calculating the RPD. This will always yield an RPD of 200%.

E. Action

Refer to ICP-MS Table 7 for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient duplicates.

1. For a duplicate sample analysis that does not meet the technical criteria, apply the actions 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 data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the duplicate sample should be qualified.

2. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.
3. For high RPDs (i.e., > 100%), use professional judgment to qualify the data as this may be indicative of a sampling problem.

ICP-MS Table 7. Duplicate Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Duplicate analysis not performed at the specified frequency	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%*	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL*	J	UJ
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* 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**, the QAPP or project-specific SOPs for data review may allow the use of less restrictive criteria (e.g., 35% RPD, 2x QL) to be assessed against duplicate soil samples.

VIII. Spike Sample Analysis

A. Review Items

Data package Cover Page, laboratory matrix spike reports (if available), preparation logs, instrument printouts, and raw data in the data package.

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. Field samples should be used as source samples for matrix spike analysis.
2. At least one spiked sample (pre-digestion) should be prepared and analyzed from each group of samples with a similar matrix type (e.g., aqueous/water or soil/sediment/waste), or for each data package. Additional matrix spike sample analyses may be required. Alternately, the data user may require that a specific sample be used for the matrix spike sample analysis.
3. The spike Percent Recovery (%R) should be within the established acceptance limits. However, for samples analyzed under the Statement of Work (SOW), spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data should be reported unqualified, even if the %R does not meet the acceptance criteria.
4. For samples analyzed under the SOW, when the spike recovery falls outside of the control limits and the sample result is < 4x the spike added, a post-digestion spike analysis should be performed for those analytes that do not meet the specified criteria. An aliquot of the remaining unspiked sample should be spiked at 2x the indigenous level or 2x the Quantitation Limit (QL), whichever is greater.
5. If the spiked sample analysis was performed on the same sample that was selected for the duplicate sample analysis, spike calculations should be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for determining the %R.

NOTE: The final spike concentrations required for the various target analytes are presented in the methods described in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).

D. Evaluation

1. Verify, using the data package Cover Page, laboratory reports, preparation log and raw data, that the appropriate number of required spiked samples was prepared and analyzed.
2. Verify that the matrix spike analysis was performed on a field sample.
3. Verify, using the raw data, that all matrix spike 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. Verify that the %R values for the matrix spike are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

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

NOTE: When the Sample Result is reported as a non-detect, use SR = 0 only for calculating the %R.

E. Action

Refer to ICP-MS Table 8 for the evaluation criteria and corresponding actions for detected and non-detected target and spike analyte results in the samples associated with deficient matrix spikes.

1. For a matrix spike sample analysis that does not meet the technical criteria, apply the actions 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 data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.
2. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

ICP-MS Table 8. Spike Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike analysis not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
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

Criteria	Action	
	Detect	**Non-detect**
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

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

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**, the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review 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.

IX. Serial Dilution

A. Review Items

Laboratory serial dilution reports (if available), instrument printouts, and raw data in the data package.

B. Objective

The objective of the serial dilution analysis is to determine if significant physical or chemical interferences exist due to sample matrix.

C. Criteria

1. An ICP Serial Dilution analysis should be performed on a sample from each group of samples with a similar matrix type (e.g., aqueous/water or soil/sediment/waste) or for each data package, whichever is more frequent.
2. Field samples should be used as source samples for the ICP Serial Dilution analysis.
3. If the analyte concentration is sufficiently high [concentration in the original sample is > 50x the Method Detection Limit (MDL) that is calculated for the sample or the limit in the Quality Assurance Project Plan (QAPP)], the Percent Difference (%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 ≥ Quantitation Limit (QL)] should be ≤ 20%.

D. Evaluation

1. Verify that the serial dilution analysis was performed on a field sample.
2. Verify that the %D values are correct by recalculating one or more of the %Ds using the raw data and the following equation:

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

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 from the original sample), possibly due to high levels of dissolved solids in the sample, ionization effects, etc.

E. Action

Refer to ICP-MS Table 9 for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient serial dilution analyses.

1. For a serial dilution sample analysis that does not meet the technical criteria, apply the actions 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 data package. Two possible determinations are: 1) only some of the samples in the data package 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.

2. Note the potential effects on the reported data in the Data Review Narrative.

ICP-MS Table 9. Serial Dilution Actions

Criteria	Action	
	Detect	**Non-detect**
Serial Dilution analysis not performed at the specified frequency	J	UJ
Sample concentration > 50x MDL, serial dilution sample concentration ≥ QL, and %D > 20%*	J	No qualification
Sample concentration > 50x MDL, serial dilution sample concentration ≥ QL, and %D ≤ 20%	No qualification	No qualification
Sample concentration > 5x QL and serial dilution sample concentration < QL	No qualification	No qualification
Interferences present	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, the QAPP or the project-specific SOPs for data review may allow the use of less restrictive criteria (e.g., %D > 25%) to be assessed against serial dilution soil samples.

X. Internal Standards

A. Review Items

Laboratory internal standard reports (if available), instrument printouts and raw data in the data package.

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, should contain internal standards. The minimum number of internal standards specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) from the list specified in the QAPP or in the SOW should be added to each sample. If the laboratory uses lithium as an internal standard, the laboratory should use a Li^6-enriched standard. The laboratory should monitor the same internal standards throughout the entire analytical sequence and should assign each analyte to at least one internal standard or the number specified in the QAPP.
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 should fall within the limits specified in the QAPP or in the SOW of the response in the calibration blank. When collision or reaction cells are used, both the target analyte and associated internal standard responses should be monitored in the same mode.
3. If the %RI of the response in the sample falls outside of these limits, the laboratory should reanalyze the original sample at the specified dilution with internal standard added.

D. Evaluation

1. Verify, using the raw data, that the minimum number of 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 the specified number of internal standard(s).
2. Verify, using the raw data, that these internal standards were added to each sample in the analytical sequence, including calibrations, samples, and Quality Control (QC) samples (except tune).
3. Verify 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 the raw data, that if the %RI for a sample was outside the limits in the QAPP or in the SOW, the sample was reanalyzed at the specified dilution with internal standard added.

E. Action

Refer to ICP-MS Table 10 for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples with deficient internal standards. Apply the actions to the affected analytes for each sample that does not meet the internal standard criteria.

If the Internal Standard %RI grossly exceeds the limits in both the original analysis and the diluted re-analysis, qualify the data based on the following considerations:

a. If the %RI is greater than 200%, high recoveries are generally due to the natural presence of the internal standard isotope in the sample(s). This occurrence may have been detected in earlier sampling of the site. Apply another appropriate internal standard to the affected analytes, do not qualify the analytes based on the high internal standard.

b. If the Internal Standard %RI is less than 30%, it is possible that some form of signal suppression is taking place.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

ICP-MS Table 10. Internal Standard Actions

Criteria	Action	
	Detect	**Non-detect**
Internal standards not analyzed	R	R
Less than the required number of internal standards analyzed	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 specified dilution with %RI 60-125%	No qualification	No qualification
%RI < 60% or > 125% and original sample reanalyzed at specified dilution with %RI < 60% or > 125%	J	UJ
Original sample not reanalyzed at specified dilution	J or R	UJ or R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

XI. Target Analyte Quantitation

A. Review Items

Laboratory Result Reports, sample preparation sheets, data package narrative, instrument printouts and raw data.

B. Objective

The objective is to ensure that the reported results and quantitation limits for target analytes reported by the laboratory are accurate and sufficient to meet requirements.

C. Criteria

Final target analyte results and quantitation limits (QLs) should be calculated according to the correct equations, taking into account amount of sample prepared, final digestate volume, dilution factor, and percent solids, as appropriate.

D. Evaluation

1. Verify that the results for all positively identified target analytes are calculated and reported by the laboratory according to the equations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Verify that the reported QLs for non-detected target analytes are calculated and reported by the laboratory according to the equations in the QAPP or in the SOW.
3. Verify that all reported results and QLs have been adjusted to reflect percent solids, original sample mass/volume, and any applicable dilutions.

E. Action

1. If sample results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).
2. If the sample percent solids is < 30%, check if the sample was prepared at greater mass to maintain the QLs. Use professional judgment when this was not completed.

F. Example Equations

1. **Aqueous/Water Sample Concentration**

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

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

2. **Soil/Sediment and Waste Sample Concentration**

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

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
DF = Dilution Factor

3. **Adjusted DL (or MDL) /Adjusted QL**

   To calculate the adjusted Detection Limit (DL) or adjusted Quantitation Limit (QL) for aqueous/water samples, substitute the value of the DL or QL into the "C" term in the equation above.

   Calculate the adjusted DL or adjusted QL for soil/sediment samples as follows:

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

   Where,

C	=	Detection Limit (DL) or Quantitation Limit (QL) (mg/kg)
W_M	=	Minimum method required aliquot amount (g)
W	=	Initial aliquot amount (g)
V_M	=	Method required final sample digestion volume (mL)
V_f	=	Final digestion volume (mL)
S	=	%Solids/100
DF	=	Dilution Factor

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MERCURY DATA REVIEW

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

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, raw data, and narrative in the data package, 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 shipping and storage conditions and the holding time of the sample.

C. Criteria

1. Samples received with pH ≥ 2 may be adjusted to pH < 2 by the laboratory. The laboratory must allow the sample to set for at least 16 hours after acid addition before rechecking the pH. Samples adjusted to pH < 2 by the laboratory do not require qualification.
2. The technical holding time is determined from the date of sample collection, or the date that Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction is complete, to the date of analysis.
3. The technical holding time criteria for aqueous/water samples and TCLP/SPLP aqueous filtrates and leachate samples is 28 days or as specified in the Quality Assurance Project Plan (QAPP), preserved (with nitric acid) to pH < 2.
4. The technical holding time criteria for soil/sediment and waste samples is 28 days or as specified in the QAPP.
5. Soil/sediment and waste samples should be maintained at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of collection until receipt at the laboratory and should be stored at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of sample receipt until digestion.

D. Evaluation

1. Review the data package narrative, sampling documentation, and sample receipt forms, to determine if the samples were properly preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperature at receipt, pH), or if the pH was adjusted upon receipt. If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples were properly stored at the laboratory.
2. Verify that the analysis dates on the Laboratory Results Reports and the raw data are identical.
3. Establish the technical holding times by comparing the sample collection dates on the sampling documentation and the TCLP/SPLP extraction dates with the dates of analysis on the Laboratory Results Reports and the raw data.

E. Action

Refer to Mercury Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected mercury results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

If a discrepancy is found between the sample analysis date on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

Mercury Table 1. Preservation and Holding Time Actions

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with pH ≥ 2 and pH adjusted by laboratory	No qualification	No qualification
Aqueous/water samples received with pH ≥ 2 and pH not adjusted	J-	R
TCLP/SPLP leachates with pH ≥ 2 and pH not adjusted	J-	R
Soil/sediment and waste samples received or stored at a temperature > 6°C but ≤ 10°C	J	UJ
Soil/sediment/waste samples received at a temperature >10°C*	J-	R
Technical Holding Time: Aqueous/water and TCLP/SPLP leachates > 28 days	J-	R
Technical Holding Time: Soil/sediment and waste samples > 28 days	J-	R
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

* For samples received with shipping container temperatures > 10ºC, the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review may allow the use of higher temperature criteria before assessing any actions for the affected samples.

II. Calibration

A. Review Items

Laboratory initial calibration and calibration verification reports (if available), preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Critical

1. Initial Calibration

   The instruments should be successfully calibrated daily, or as specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW), and each time the instrument is set up. The calibration date and time should be included in the raw data. For samples analyzed under the SOW, the calibration curve standards should be prepared by the same method used to prepare the samples for analysis. The curve should be prepared with the samples that will be analyzed using this calibration curve.

NOTE: A blank and the number of calibration standards specified in the QAPP or in the SOW should be used to establish the calibration curve. At least one of the calibration standards should be at or below the Quantitation Limit (QL) in the QAPP or in the SOW but above the Detection Limit or the Method Detection Limit (MDL). Calibration standards at and above the QL should be continuous with none excluded to satisfy Quality Control (QC) requirements. The calibration curve may be fitted using linear regression or weighted linear regression, or other fits as specified in the QAPP. The curve may be forced through zero. For linear fits, the calibration curve should have a correlation coefficient greater than the value specified in the QAPP or in the SOW. The calculated percent differences (%Ds) or other specified statistical test values for all non-zero standards should fall within the limits in the QAPP or in the SOW.

2. Initial and Continuing Calibration Verification

   For samples analyzed under the SOW, these standards should be prepared by the same method used to prepare the samples for analysis.

   a. Initial Calibration Verification (ICV)

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

      ii. Analyses of the ICV should be conducted using a certified solution of the analyte from an independent standard source, at a concentration level other than that used for instrument calibration and near the middle of the calibrated range (within ±30%).

   b. Continuing Calibration Verification (CCV)

      i. To ensure accuracy during each analytical sequence, the CCV should be analyzed and reported.

      ii. The CCV standard should be analyzed at the frequency specified in the QAPP, or every hour during an analytical sequence. The CCV standard should also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

iii. The CCV standard should be prepared using the same source and in the same acid matrix as the calibration standards at a concentration at or near the mid-level (within ±30%) of the respective calibration curve.

iv. The same CCV standard solution should be used throughout the analysis for a data package.

v. The CCV should 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 and documented in the data package narrative, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

vi. An instrument blank should not be analyzed before the CCV.

D. Evaluation

1. Verify that the instrument was calibrated as specified in the QAPP or in the SOW and each time the instrument was set up, utilizing a blank and at least the minimum number of standards specified by the QAPP or in the SOW. Confirm that at least one of the calibration standards was analyzed at or below the QL in the QAPP or in the SOW, but above the Detection Limit or MDL and that all subsequent calibration standards are consecutive with none removed to satisfy QC requirements. For linear fits, verify that the correlation coefficient of the calibration curve is greater than the value specified in the QAPP or in the SOW. Verify that the %Ds for all non-zero standards are within the SOW limits or that other statistical test values are within the limits specified in the QAPP. 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. Confirm that an instrument blank was not analyzed before the CCV.
4. Verify that the ICV and CCV %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

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

E. Action

Refer to Mercury Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected mercury results in the samples associated with deficient initial calibrations or calibration verification standards.

1. For initial calibrations or ICV standard analyses that do not meet the technical criteria, apply the actions to the associated samples reported from the analytical sequence.
2. For CCV standard analyses that do not meet the technical criteria, apply the actions 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.
3. 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 the QL but above the MDL), qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

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

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

Mercury Table 2. Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed or not performed at specified frequency	R	R
Calibration incomplete (insufficient number of standards or required concentrations missing)	J or R	UJ or R
For linear fits, correlation coefficient < 0.995	J	UJ
%D outside ±30%, or other specified statistical test values outside limits	J	UJ
Calibration Standards and/or ICV/CCV not prepared with samples	J	UJ
ICV/CCV not performed at specified frequency	J	UJ
ICV/CCV %R < 70%	J- or R	UJ or 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%	J+ or R	No qualification
Instrument blank analyzed prior to CCV	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

III. Blanks

A. Review Items

Laboratory blanks reports (if available), preparation logs, calibration standard logs, instrument logs, and raw data in the data package.

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) should be analyzed at the wavelength used for analysis after the analytical standards, but not before analysis of the ICV standard during the initial calibration of the instrument. The ICB should be prepared by the same method used to prepare the samples for analysis. The ICB result (absolute value) should not be greater than or equal to the Quantitation Limit (QL).
3. A Continuing Calibration Blank (CCB) should be analyzed immediately after every Continuing Calibration Verification (CCV) standard. The CCB should be prepared by the same method used to prepare the samples for analysis. The CCB should be analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) during the analytical sequence. The CCB should 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) should not be greater than or equal to the QL.
4. At least one Preparation Blank should be prepared and analyzed for each matrix, with every data package, 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 mercury concentration in the Preparation Blank is greater than or equal to the QL, the lowest concentration of mercury in the associated samples should be $\geq$ 10x the Preparation Blank concentration. Otherwise, all associated samples with a mercury concentration < 10x the Preparation Blank concentration and $\geq$ the QL should be redigested and reanalyzed. The laboratory is not to correct the sample concentration for the blank value.
6. If the mercury concentration in the Preparation Blank is $\leq$ (-QL), all associated samples with mercury concentrations < 10x the QL should be redigested and reanalyzed.
7. At least one Leachate Extraction Blank (LEB) should be prepared and analyzed for each batch of samples extracted by Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP). The LEB consists of reagent water processed through the extraction procedure. Post-extraction, the LEB should 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 and LEBs are prepared and analyzed as appropriate for the data package (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Verify, using the digestion log, that the ICB and CCB were digested by the same method used to prepare the samples.

3. For an ICB or a CCB, verify that if the absolute value of mercury was greater than or equal to the QL, the analysis was terminated, the problem corrected and documented in the data package narrative, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.
4. For a Preparation Blank, verify that if the concentration of mercury was greater than or equal to the QL, all associated samples with mercury concentration ≥ the QL but < 10x the Preparation Blank concentration were redigested and reanalyzed. Verify that if the mercury concentration was ≤ (-QL) in a Preparation Blank, all associated samples with mercury concentration < 10x the QL were redigested and reanalyzed.
5. Evaluation of field and equipment blanks should also be performed according to the QAPP or appropriate guidance.

E. Action

Refer to Mercury Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected mercury results in the samples associated with deficient blanks.

1. For ICB analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCB analyses that do not meet the technical criteria, apply the actions 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.
3. For Preparation Blank analyses that do not meet the technical criteria, apply the actions to all associated samples prepared in the same preparation batch. For LEBs that do not meet the technical criteria, apply the actions to all associated samples extracted in the same extraction batch.
4. 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.
5. If the absolute value of an ICB or a CCB result is ≥ QL, the analysis should have been terminated and the affected samples re-analyzed. If samples were not re-analyzed, qualify as described in Table 3 below.
6. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and ≥ QL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, qualify as described in Table 3 below.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute run, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. For blank results ≤ (-MDL) but > (-QL), the possibility of false negatives exists.

NOTE: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment or waste sample results reported in the Laboratory Results Reports will not be on the same basis (units, dilution) as the calibration blank data. 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 or waste samples for comparison purposes.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

Mercury Table 3. Blank Actions

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Not analyzed at the specified frequency	Non-detect	UJ
		Detect	J
ICB/CCB	Not digested	Detect or non-detect	Use professional judgment
ICB/CCB	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
ICB/CCB	≤ (-MDL) but > (-QL)	Non-Detect	UJ
		Detect	J- or no qualification
ICB/CCB	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < ICB/CCB Result	Report at ICB/CCB Result and qualify U
		≥ ICB/CCB Result	J+ or no qualification
ICB/CCB	≤ (-QL)	Non-detect	UJ or R
		Detect < QL	J-
		≥ QL	J-
Preparation Blank/LEB	Not analyzed at specified frequency	Non-detect	UJ
		Detect	J
Preparation Blank/LEB/ Field Blank	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
Preparation Blank/LEB/ Field Blank	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification
Preparation Blank/LEB/ Field Blank	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < 10x the Preparation Blank/LEB Result	Report at Preparation Blank/LEB Result and qualify J+ or R
		≥ 10x the Preparation Blank/LEB Result	No qualification

Blank Type	Blank Result	Sample Result	Action
Preparation Blank/LEB/ Field Blank	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-
		≥ QL but < 10x QL	J-
		≥ 10x QL	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Duplicate Sample Analysis

A. Review Items

Data package Cover Page, laboratory duplicate reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Field samples should be used as source samples for duplicate analysis.
2. At least one duplicate sample should be prepared and analyzed from each group of samples of a similar matrix type (e.g., aqueous/water or soil/sediment/waste) or for each data package. Duplicates cannot be averaged for reporting on the Laboratory Results Report. Additional duplicate sample analyses may be required. Alternately, the data user may require that a specific sample be used for the duplicate sample analysis.
3. The Relative Percent Difference (RPD) control limit specified in the Quality Assurance Project Plan (QAPP) or of 20% should be used for original and duplicate sample values ≥ 5x the Quantitation Limit (QL).
4. For samples analyzed under the SOW, a control limit of the QL should be used if either the sample or duplicate value is < 5x the QL.

D. Evaluation

1. Verify, from the data package Cover Page, laboratory reports, preparation log and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed.
2. Verify, using the raw data, that the duplicate results fall within the established control limits.
3. Verify that the duplicate analysis was performed on a field sample.
4. Verify that the RPD values are correct by recalculating one or more of the RPDs using the raw data and the following equation:

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

Where,

S = Sample Result (original)
D = Duplicate Result

NOTE: When the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only for calculating the RPD. This will always yield an RPD of 200%.

E. Action

Refer to Mercury Table 4 below for the evaluation criteria and corresponding actions for detected and non-detected mercury results in the samples associated with deficient duplicates.

1. For a duplicate sample analysis that does not meet the technical criteria, apply the actions 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 mercury) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the duplicate sample should be qualified.
2. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.
3. For high RPDs (i.e., > 100%), use professional judgment to qualify the data as this may be indicative of a sampling problem.

Mercury Table 4. Duplicate Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Duplicate analysis not performed at the specified frequency	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%*	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
For samples analyzed under the SOW, original sample or duplicate sample results < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL*	J	UJ
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* 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**, the QAPP or the project-specific SOPs for data review may allow the use of less restrictive criteria (e.g., 35% RPD, 2x QL) to be assessed against duplicate soil samples.

V. Spike Sample Analysis

A. Review Items

Data package Cover Page, laboratory matrix spike reports (if available), preparation logs, instrument printouts, and raw data in the data package.

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. Field samples should be used as source samples for matrix spike analysis.
2. At least one spiked sample should be prepared and analyzed from each group of samples with a similar matrix type (e.g., aqueous/water or soil/sediment/waste), or for each data package. Additional matrix spike sample analyses may be required. Alternately, the data user may require that a specific sample be used for the matrix spike sample analysis.
3. The spike Percent Recovery (%R) should be within the established acceptance limits. However, for samples analyzed under the Statement of Work (SOW), spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data should be reported unqualified, even if the %R does not meet the acceptance criteria.
4. If the spiked sample analysis was performed on the same sample that was selected for the duplicate sample analysis, spike calculations should be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for determining the %R.

NOTE: The final spike concentration required is presented in the method described in the Quality Assurance Project Plan (QAPP) or in the SOW.

D. Evaluation

1. Verify, using the data package Cover Page, laboratory reports, preparation log, and raw data, that the appropriate number of required spiked samples was prepared and analyzed.
2. Verify that the matrix spike analysis was performed on a field sample.
3. Verify, using the raw data, that all Matrix Spike sample results fall within the established control limits.
4. Verify that the %R values for the matrix spike are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where**,**

SSR = Spiking analyte result in the spiked sample
SR = Result of the same analyte in the original sample
SA = Spike added in the spiked sample

NOTE: When the Sample Result is reported as a non-detect, use SR = 0 only for calculating the %R.

E. Action

Refer to Mercury Table 5 below for the evaluation criteria and corresponding actions for detected and non-detected mercury results in the samples associated with deficient matrix spikes.

1. For a Matrix Spike sample analysis that does not meet the technical criteria, apply the actions 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 mercury) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.
2. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Mercury Table 5. Spike Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike analysis not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
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

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

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**, the QAPP or the project-specific SOPs for data review 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. Target Analyte Quantitation

A. Review Items

Laboratory Result Reports, sample preparation sheets, data package narrative, instrument printouts and raw data.

B. Objective

The objective is to ensure that the reported results and quantitation limits for target analytes reported by the laboratory are accurate and sufficient to meet requirements.

C. Criteria

Final target analyte results and quantitation limits should be calculated according to the correct equations, taking into account amount of sample prepared, final digestate volume, dilution factor, and percent solids, as appropriate.

D. Evaluation

1. Verify that the results for all positively identified target analytes are calculated and reported by the laboratory according to the equations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Verify that the reported Quantitation Limits (QLs) for non-detected target analytes are calculated and reported by the laboratory according to the equations in the QAPP or in the SOW.
3. Verify that all reported results and QLs have been adjusted to reflect percent solids, original sample mass/volume, and any applicable dilutions.

E. Action

1. If sample results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).
2. If the sample percent solids is < 30%, check if the sample was prepared at greater mass to maintain the QLs. Use professional judgment when this was not completed.

F. Example Equations

1. **Aqueous/Water and TCLP/SPLP Leachate Sample Concentration**

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

Where,

C = Instrument value in µg/L from the calibration curve
DF = Dilution Factor

2. **Soil/Sediment and Waste Sample Concentration**

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

Where,

Concentration = Analyte/Result (mg/kg)
C = Instrument value in µg/L from the calibration curve
V_f = Final digestion volume (mL)
W = Initial aliquot amount (g)
S = %Solids/100
DF = Dilution Factor

3. **Adjusted DL (or MDL)/Adjusted QL**

   To calculate the adjusted Detection Limit (DL) or adjusted Quantitation Limit (QL) for aqueous/water or Total Characteristic Leaching Procedure (TCLP)/Synthetic Precipitation Leaching Procedures (SPLP) leachate samples, substitute the value of the DL or QL, in the appropriate units, into the "C" term in the equation above.

   Calculate the adjusted DL or adjusted QL for soil/sediment and waste samples as follows:

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

   Where,

C	=	Detection Limit (DL) or Quantitation Limit (QL) (mg/kg)
W_m	=	Method required minimum sample weight (g)
W	=	Initial aliquot amount (g)
S	=	%Solids/100
DF	=	Dilution Factor

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CYANIDE DATA REVIEW

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

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, raw data, and narrative in the data package, 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 shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The technical holding time is determined from the date of sample collection, or the date that Synthetic Precipitation Leaching Procedure (SPLP) extraction is complete, to the date of analysis.
2. The technical holding time criteria for aqueous/water samples, and SPLP aqueous filtrate and leachate samples is 14 days or as specified in the Quality Assurance Project Plan (QAPP), preserved (with sodium hydroxide) to pH > 10.
3. The technical holding time criteria for soil/sediment and waste samples is 14 days or as specified in the QAPP.
4. Aqueous/water and soil/sediment/waste samples should be maintained at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of collection until receipt at the laboratory and be stored at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of sample receipt until distillation. The SPLP leachates should be stored at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of the leaching procedure completion until preparation.

D. Evaluation

1. Review the data package narrative, sampling documentation, and sample receipt forms to determine if the samples were properly preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperature at receipt, pH). If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples were properly stored at the laboratory For aqueous/water samples, look for evidence that the samples were tested for the presence of sulfides, oxidizing agents, and nitrate/nitrite, and whether the appropriate preservation steps were taken.
2. Verify that the analysis dates on the Laboratory Results Reports and the raw data are identical.
3. Establish the technical holding times by comparing the sample collection dates on the sampling documentation and the SPLP extraction dates with the dates of analysis on the Laboratory Results Reports.

E. Action

Refer to Cyanide Table 1 for the evaluation criteria and corresponding actions for detected and non-detected cyanide results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

If a discrepancy is found between the sample analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

Cyanide Table 1. Preservation and Holding Time Actions

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/nitrite present and not treated with sulfamic acid	J	R
Aqueous/water samples received with pH ≤ 10	J*	R
Aqueous/water and soil/sediment/waste samples received or stored at a temperature > 6°C but ≤ 10°C **	J	UJ
Aqueous/water and soil/sediment/waste samples received or stored at a temperature > 10°C**	J-	R
Technical Holding Time: Aqueous/water and SPLP leachates > 14 days	J*	R
Technical Holding Time: Soil/sediment/waste samples > 14 days	J*	R
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* The true direction of any bias may be unknown in this case. Use caution in determining whether some detected analytes should be qualified as estimated low (J-) or as estimated high (J+), based on knowledge of the potential presence of other compounds that may react with cyanide or related compounds (e.g., thiocyanate). Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for additional guidance on the use of the J+ and J- qualifiers.

** For samples received with shipping container temperatures > 10ºC, the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review may allow the use of higher temperature criteria before assessing any actions for the affected samples.

II. Calibration

A. Review Items

Laboratory initial calibration and calibration verification reports (if available), preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data in the data package.

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 should be successfully calibrated daily or as specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) and each time the instrument is set up. The calibration date and time should be included in the raw data. For samples analyzed under the SOW, the calibration curve standards should be distilled by the same method used to prepare the samples for analysis.

NOTE: A blank and the number of calibration standards specified in the QAPP or in the SOW should be used to establish the calibration curve. At least one of the calibration standards should be at or below the Quantitation Limit (QL) in the QAPP or in the SOW but above the Detection Limit or the Method Detection Limit (MDL). Calibration standards at and above the QL should be continuous with none excluded to satisfy QC requirements. The calibration curve may be fitted using linear regression or weighted linear regression, or other fits as specified in the QAPP. The curve may be forced through zero. For linear fits, the calibration curve should have a correlation coefficient greater than the value specified in the QAPP or in the SOW. The calculated percent differences (%Ds) or other specified statistical test values for all non-zero standards should be within the limits in the QAPP or in the SOW.

2. Initial and Continuing Calibration Verification

   For samples analyzed under the SOW, these standards should be distilled by the same method used to prepare the samples for analysis.

   a. Initial Calibration Verification (ICV)

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

      ii. Analyses of the ICV should be conducted using a certified solution of the analyte from an independent standard source, at a concentration level other than that used for instrument calibration and near the middle of the of the calibrated range (within ±30%).

   b. Continuing Calibration Verification (CCV)

      i. To ensure accuracy during each analytical sequence, the CCV standard should be analyzed and reported.

      ii. The CCV standard should be analyzed at the frequency specified in the QAPP, or every hour during an analytical sequence. The CCV standard should also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

iii. The CCV standard should be prepared using the same source and in the same base matrix as the calibration standards at a concentration at or near the mid-level (within ±30%) of the respective calibration curve.

iv. The same CCV standard solution should be used throughout the analysis for a data package.

v. The CCV should 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 and documented in the data package narrative, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

vi. An instrument blank should not be analyzed before the CCV.

D. Evaluation

1. Verify that the instrument was calibrated as specified in the QAPP or in the SOW and each time the instrument was set up, utilizing a blank and at least the minimum number of standards specified in the QAPP or in the SOW. Confirm that at least one of the calibration standards was analyzed at or below the QL in the QAPP or in the SOW, but above the Detection Limit or MDL and that all subsequent calibration standards are consecutive with none removed to satisfy QC requirements.. For linear fits, verify that the correlation coefficient of the calibration curve is greater than the value specified in the QAPP or in the SOW. Verify that the %Ds for all non-zero standards are within the SOW limits or that other statistical test values are with the limits specified in the QAPP.
2. Verify, using the distillation log, that the calibration standards, the ICV, and the CCV standards were distilled.
3. Confirm that an instrument blank was not analyzed before the CCV.
4. 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.
5. Verify that the ICV or CCV %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of cyanide measured in the analysis of the ICV or CCV solution

True (value) = Concentration of cyanide in the ICV or CCV source

E. Action

Refer to Cyanide Table 2 for the evaluation criteria and corresponding actions for detected and non-detects cyanide results in the samples associated with deficient initial calibrations or calibration verification standards.

1. For initial calibrations or ICV standard analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCV standard analyses that do not meet the technical criteria, apply the actions 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.
3. 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 the QL but above the MDL), qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

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

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

Cyanide Table 2. Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed or not performed at specified frequency	R	R
Calibration incomplete (insufficient number of standards or required concentrations missing)	J or R	UJ or R
For linear fits, the correlation coefficient < 0.995	J	UJ
%D outside 30% or other specified statistical test values outside limits	J	UJ
Calibration Standards and ICV/CCV not distilled	J	UJ
ICV/CCV not performed at specified frequency	J	UJ
ICV/CCV %R < 70%	J- or R	UJ or 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%	J+ or R	No qualification
Instrument blank analyzed prior to CCV	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

III. Blanks

A. Review Items

Laboratory blanks reports (if available), preparation logs, calibration standard logs, instrument logs, and raw data in the data package.

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) should be analyzed at the wavelength used for analysis after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) standard during the initial calibration of the instrument. The ICB should be distilled by the same method used to prepare the samples for analysis. The ICB result (absolute value) should not be greater than or equal to the Quantitation Limit (QL).
3. A Continuing Calibration Blank (CCB) should be analyzed immediately after every Continuing Calibration Verification (CCV) standard. The CCB should be distilled by the same method used to prepare the samples for analysis. The CCB should be analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) during the analytical sequence. The CCB should 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) should not be greater than or equal to the QL.
4. At least one Preparation Blank should be prepared and analyzed for each matrix, with every data package, 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 cyanide concentration in the Preparation Blank is greater than or equal to the QL, the lowest concentration of cyanide in the associated samples should be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with a cyanide concentration < 10x the Preparation Blank concentration and ≥ the QL should be redistilled and reanalyzed. The laboratory is not to correct the sample concentration for the blank value.
6. If the cyanide concentration in the Preparation Blank is ≤ (-QL), all associated samples with a cyanide concentration < 10x the QL should be redistilled and reanalyzed.
7. At least one Leachate Extraction Blank (LEB) should be prepared and analyzed for each batch of samples extracted by Synthetic Precipitation Leaching Procedure (SPLP). The LEB consists of reagent water processed through the extraction procedure. Post-extraction, the LEB should 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 and LEBs are prepared and analyzed as appropriate for the data package (e.g., total number of samples, various types of matrices present, number of distillation batches, etc.).
2. Verify, using the distillation log, that the ICB and CCB were distilled by the same method used to prepare the samples.
3. For an ICB or a CCB, verify that if the absolute value of cyanide was greater than or equal to the QL, the analysis was terminated, the problem corrected and documented in the data package

narrative, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

4. For a Preparation Blank, verify that if the concentration of cyanide was greater than or equal to the QL, all associated samples with a cyanide concentration ≥ the QL but < 10x the Preparation Blank concentration were redistilled and reanalyzed. Verify that if the cyanide concentration was ≤ (-QL) in a Preparation Blank, all associated samples with a cyanide concentration < 10x the QL were redistilled and reanalyzed.
5. Evaluation of field and equipment blanks should also be performed according to the QAPP or appropriate guidance.

E. Action

Refer to Cyanide Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected cyanide results in the samples associated with deficient blanks.

1. For ICB analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCB analyses that do not meet the technical criteria, apply the actions 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.
3. For Preparation Blank analyses that do not meet the technical criteria, apply the actions to all associated samples prepared in the same preparation batch. For LEBs that do not meet the technical criteria, apply the actions to all associated samples extracted in the same extraction batch.
4. 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.
5. If the absolute value of an ICB or a CCB result is ≥ QL, the analysis should have been terminated and the affected samples re-analyzed. If samples were not re-analyzed, qualify as described in Table 3 below.
6. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and ≥ QL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, qualify as described in Table 3 below.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute run, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. For blank results ≤ (-MDL) but > (-QL), the possibility of false negatives exists.

NOTE: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment or waste sample results reported in the Laboratory Results Reports will not be on the same basis (units, dilution) as the calibration blank data. 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 or waste samples for comparison purposes.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

Cyanide Table 3. Blank Actions

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Not analyzed at the specified frequency	Non-detect	UJ
		Detect	J
ICB/CCB	Not distilled	Detect or non-detect	Use professional judgment
ICB/CCB	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
ICB/CCB	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification
ICB/CCB	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < ICB/CCB Result	Report at ICB/CCB Result and qualify U
		≥ ICB/CCB Result	J+ or no qualification
ICB/CCB	≤ (-QL)	Non-detect	UJ or R
		Detect < QL	J-
		≥ QL	J-
Preparation Blank/LEB	Not analyzed at specified frequency	Non-detect	UJ
		Detect	J
Preparation Blank/LEB/ Field Blank	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
Preparation Blank/LEB/ Field Blank	≤ (-MDL) but > (-QL)	Non-detect	No qualification
		Detect	J- or no qualification
Preparation Blank/LEB/ Field Blank	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < 10x the Preparation Blank/LEB Result	Report at Preparation Blank/LEB Result and qualify J+ or R
		≥ 10x the Preparation Blank/LEB Result	No qualification
	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-

Blank Type	Blank Result	Sample Result	Action
Preparation Blank/LEB/ Field Blank		≥ QL but < 10x QL	J-
		≥ 10x QL	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Duplicate Sample Analysis

A. Review Items

Data package Cover Page, laboratory duplicate reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Field samples should be used as source samples for duplicate analysis.
2. At least one duplicate sample should be prepared and analyzed from each group of samples of a similar matrix type (e.g., aqueous/water or soil/sediment/waste) or for each data package. Duplicates cannot be averaged for reporting on the Laboratory Results Report. Additional duplicate sample analyses may be required. Alternately, the data user may require that a specific sample be used for the duplicate sample analysis.
3. The Relative Percent Difference (RPD) control limit specified in the Quality Assurance Project Plan (QAPP) or of 20% should be used for original and duplicate sample values ≥ 5x the Quantitation Limit (QL).
4. For samples analyzed under the Statement of Work (SOW), a control limit of the QL should be used if either the sample or duplicate value is < 5x the QL.

D. Evaluation

1. Verify, from the data package Cover Page, laboratory reports, preparation log and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed for the data package.
2. Verify, using the raw data, that the duplicate results fall within the established control limits.
3. Verify that the duplicate analysis was performed on a field sample.
4. Verify that the RPD values are correct by recalculating one or more of the RPDs using the raw data and the following equation:

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

Where,

S = Sample result (original)
D = Duplicate result

NOTE: When the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only for calculating the RPD. This will always yield an RPD of 200%.

E. Action

Refer to Cyanide Table 4 for the evaluation criteria and corresponding actions for detected and non-detected cyanide results in the samples associated with deficient duplicates.

1. For a duplicate sample analysis that does not meet the technical criteria, apply the actions 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 data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the duplicate sample should be qualified.
2. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.
3. For high RPDs (i.e., > 100%), use professional judgment to qualify the data as this may be indicative of a sampling problem.

Cyanide Table 4. Duplicate Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Duplicate analysis not performed at the specified frequency	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%*	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%*	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL*	J	UJ
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* 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**, the QAPP or the project-specific SOPs for data review may allow the use of less restrictive criteria (e.g., 35% RPD, 2x QL) to be assessed against duplicate soil samples.

V. Spike Sample Analysis

A. Review Items

Data package Cover Page, laboratory matrix spike reports (if available) preparation logs, instrument printouts, and raw data in the data package.

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. Field samples should be used as source samples for matrix spike analysis.
2. At least one spiked sample (pre-distillation) should be prepared and analyzed from each group of samples with a similar matrix type (e.g., aqueous/water or soil/sediment/waste), or for each data package. Additional matrix spike sample analyses may be required. Alternately, the data user may require that a specific sample be used for the matrix spike sample analysis.
3. The spike Percent Recovery (%R) should be within the established acceptance limits. However, for samples analyzed under the Statement of Work (SOW), spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data should be reported unqualified, even if the %R does not meet the acceptance criteria.
4. For samples analyzed under the SOW, when the spike recovery falls outside of the control limits and the sample result is < 4x the spike added, a post-distillation spike analysis should be performed. An aliquot of the remaining unspiked sample should be spiked at 2x the indigenous level or 2x the Quantitation Limit (QL), whichever is greater.
5. If the spiked sample analysis was performed on the same sample that was selected for the duplicate sample analysis, spike calculations should be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for determining the %R.

NOTE: The final spike concentration required is presented in the method described in the Quality Assurance Project Plan (QAPP) or in the SOW.

D. Evaluation

1. Verify, using the data package Cover Page, laboratory reports, preparation log and raw data, that the appropriate number of required spiked samples was prepared and analyzed.
2. Verify that the matrix spike analysis was performed on a field sample.
3. Verify, using the raw data, that all pre-distillation matrix spike sample results fall within the established control limits. If not, verify that a post-distillation spike was prepared and analyzed.
4. Verify that the %R values for the matrix spike are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

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

NOTE: When the Sample Result is reported as a non-detect, use SR = 0 only for calculating the %R.

E. Action

Refer to Cyanide Table 5 for the evaluation criteria and corresponding actions for detected and non-detected cyanide results in the samples associated with deficient matrix spikes.

1. For a matrix spike sample analysis that does not meet the technical criteria, apply the actions 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 cyanide) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.
2. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Cyanide Table 5. Spike Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
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

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike %R 75-125% No post-distillation is required	No qualification	No qualification
Matrix Spike %R > 125% No post-distillation spike performed	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

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**, the QAPP or the project-specific SOPs for data review 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. Target Analyte Quantitation

A. Review Items

Laboratory Result Reports, sample preparation sheets, data package narrative, instrument printouts and raw data.

B. Objective

The objective is to ensure that the reported results and quantitation limits for target analytes reported by the laboratory are accurate and sufficient to meet requirements.

C. Criteria

Final target analyte results and quantitation limits should be calculated according to the correct equations, taking into account amount of sample prepared, final distillate volume, dilution factor, and percent solids, as appropriate.

D. Evaluation

1. Verify that the results for all positively identified target analytes are calculated and reported by the laboratory according to the equations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Verify that the reported Quantitation Limits (QLs) for non-detected target analytes are calculated and reported by the laboratory according to the equations in the QAPP or in the SOW.
3. Verify that all reported results and QLs have been adjusted to reflect percent solids, original sample mass/volume, and any applicable dilutions.

E. Action

1. If sample results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).
2. If the sample percent solids is < 30%, check if the sample was prepared at greater mass to maintain the QLs. Use professional judgment when this was not completed.

F. Example Equations

1. **Aqueous/Water and SPLP Sample Concentration**

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

Where,

C	=	Instrument response in µg/L CN from the calibration curve
V_f	=	Final prepared (absorbing solution) volume (mL)
V	=	Initial aliquot amount (mL)
DF	=	Dilution Factor

2. **Soil/Sediment and Waste Sample Concentration**

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

Where,

Concentration	=	Analyte/Result (mg/kg)
C	=	Instrument response in µg/L CN from the calibration curve
V_f	=	Final prepared (absorbing solution) volume (mL)
W	=	Initial aliquot amount (g)

S = %Solids/100
DF = Dilution Factor

3. **Adjusted DL (or MDL)/Adjusted QL:**

   To calculate the adjusted Detection Limit (DL) or adjusted Quantitation Limit (QL) for aqueous/water or SPLP leachate samples, substitute the value of the DL or QL into the "C" term in the equation above.

   Calculate the adjusted DL or adjusted QL for all soil/sediment and waste samples as follows:

$$\text{Adjusted DL or QL } (mg/kg) = C \times \frac{W_M}{W \times S} \times DF$$

   Where,

C = Detection Limit (DL) or Quantitation Limit (QL) (mg/kg)
W_M = Minimum method required aliquot amount (g)
W = Initial aliquot amount (g)
S = %Solids/100
DF = Dilution Factor

ANIONS DATA REVIEW

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

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, raw data, and narrative in the data package, checking for: 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 shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The technical holding time is determined from the date and time of sample collection to the date and time of analysis.
2. The technical holding time criteria for aqueous/water samples for nitrate, nitrite, and orthophosphate is 48 hours or as specified in the Quality Assurance Project Plan (QAPP). For all other analytes the holding time is 28 days or as specified in the QAPP.
3. The technical holding time criteria for soil/sediment samples is 48 hours for nitrate, nitrite, and orthophosphate, and 28 days for all other analytes, or as specified in the QAPP.
4. Aqueous/water samples and soil/sediment samples should be maintained at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of collection until receipt at the laboratory. Aqueous/water samples to be analyzed for nitrate, nitrite, orthophosphate, or sulfate, and all soil/sediment samples should be stored at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of sample receipt until analysis or extraction. Samples for orthophosphate analysis should not be held at room temperature for more than 12 cumulative hours.

D. Evaluation

1. Review the data package narrative, sampling documentation, and sample receipt forms to determine if the samples arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperature at receipt). If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples were properly stored at the laboratory. Use professional judgment to evaluate the effect of the problem on the sample results.
2. Verify that the analysis dates and times on the Laboratory Results Reports and the raw data are identical.
3. Establish the technical holding times by comparing the sample collection dates and times on the sampling documentation with the dates and times of analysis on the Laboratory Results Reports and the raw data.

E. Action

Refer to Anions Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

If a discrepancy is found between the sample analysis dates and times on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

Anions Table 1. Preservation and Holding Time Actions

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples for nitrate, nitrite, orthophosphate, or sulfate and soil/sediment samples received at a temperature > 6°C but ≤ 10°C	J	UJ
Aqueous/water samples for nitrate, nitrite, orthophosphate, or sulfate and soil/sediment samples received at a temperature > 10°C*	J-	R
Technical Holding Time: Aqueous/water samples for nitrate, nitrite, or orthophosphate > 48 hours	J-	R
Technical Holding Time: Aqueous/water samples for all other analytes > 28 days	J-	R
Technical Holding Time: Soil/sediment samples for nitrate, nitrite, or orthophosphate > 48 hours	J-	R
Technical Holding Time: Soil/sediment samples for all other analytes > 28 days	J-	R
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

* For samples received with shipping container temperatures > 10ºC, the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review may allow the use of higher temperature criteria before assessing any actions for the affected samples.

II. Calibration

A. Review Items

Laboratory initial calibration and calibration verification reports (if available), preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data in the data package.

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 should be successfully calibrated weekly or as specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW), and each time the instrument is set up. The calibration date and time should be included in the raw data.

NOTE: A blank and the number of calibration standards specified in the QAPP or in the SOW) should be used to establish the calibration curves. At least one of the calibration standards should be at or below the Quantitation Limit (QL) in the QAPP or in the SOW but above the Detection Limit or the Method Detection Limit (MDL). Calibration standards at and above the QL should be continuous with none excluded to satisfy QC requirements. The calibration curves may be fitted using linear regression or weighted linear regression, or other fits as specified in the QAPP. The curves may be forced through zero. For linear fits, the calibration curves should have a correlation coefficient greater than the value specified in the QAPP or in the SOW. The calculated percent differences (%Ds) or other specified statistical test values for all non-zero standards should fall within the limits in the QAPP or in the SOW.

2. Initial and Continuing Calibration Verification

   a. Initial Calibration Verification (ICV)

      i. Immediately after the system has been calibrated as well as each day prior to the analysis of the opening Continuing Calibration Verification (CCV) and Continuing Calibration blank (CCB), the accuracy of the initial calibration should be verified and documented by the analysis of an ICV standard. If the ICV Percent Recovery (%R) falls outside of the control limits, the analysis should be terminated, the problem corrected and documented in the data package narrative, the instrument recalibrated, and all affected samples reanalyzed.

      ii. Analyses of the ICV should be conducted using a certified solution of the target analytes from an independent standard source, at concentration levels other than that used for instrument calibration and near the middle of the calibrated range (within ±30%).

   b. Continuing Calibration Verification (CCV)

      i. To ensure accuracy during each analytical sequence, the CCV should be analyzed and reported.

      ii. The CCV standard should be analyzed at the frequency specified in the QAPP, or every 10 samples during an analytical sequence. The CCV standard should also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

      iii. The CCV standard should be prepared using the same source and in the same matrix as the calibration standards by combining compatible analytes at a concentration at or near the mid-level (within ±30%) of the respective calibration curve.

iv. The same CCV standard solution should be used throughout the analysis for a data package.

v. The CCV should 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 and documented in the data package narrative, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

vi. An instrument blank should not be analyzed before the CCV.

D. Evaluation

1. Verify that the instrument was calibrated as specified in the QAPP or in the SOW and each time the instrument was set up, utilizing a blank and at least the minimum number of standards specified by the QAPP or in the SOW. Confirm that at least one of the calibration standards was analyzed at or below the QL in the QAPP or in the SOW, but above the Detection Limit or MDL and that all subsequent calibration standards are consecutive with none removed to satisfy QC requirements. For linear fits, verify that the correlation coefficient of the calibration curve is greater than the value specified in the QAPP or in the SOW. Verify that the %Ds for all non-zero standards are within the SOW limits or that other statistical test values are within the limits specified in the QAPP.
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. Confirm that an instrument blank was not analyzed before the CCV.
4. Verify that the ICV and CCV %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of the target analyte measured in the analysis of the ICV or CCV solution

True (value) = Concentration of the target analyte in the ICV or CCV source

E. Action

Refer to Anions Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient initial calibrations or calibration verification standards.

1. For initial calibration or ICV standard analyses that do not meet the technical criteria, apply the action to the associated samples reported from the analytical sequence.
2. For CCV standard analyses that do not meet the technical criteria, apply the actions 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.
3. 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 the QL but above the MDL), qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

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

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

Anions Table 2. Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed or not performed at specified frequency	R	R
Calibration incomplete (insufficient number of standards or required concentrations missing)	J or R	UJ or R
For linear fits, the correlation coefficient < 0.995	J	UJ
%D outside ±30%, or other specified statistical test values outside limits	J	UJ
ICV/CCV not performed at specified frequency	J	UJ
ICV/CCV %R < 75%	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%	J+ or R	No qualification
Instrument blank analyzed prior to CCV	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

III. Blanks

A. Review Items

Laboratory blanks reports (if available), preparation logs, calibration standard logs, instrument logs, and raw data in the data package.

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) should be analyzed after the analytical standards, but not before analysis of the ICV standard during the initial calibration of the instrument. The ICB result (absolute value) for the target analytes should not be greater than or equal to the respective Quantitation Limits (QLs).
3. A Continuing Calibration Blank (CCB) should be analyzed immediately after every Continuing Calibration Verification (CCV) standard. The CCB should be analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) during the analytical sequence. The CCB should 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) for the target analytes should not be greater than or equal to the respective QL.
4. At least one Preparation Blank should be prepared and analyzed for each matrix, with every data package, or with each batch of samples prepared, 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 greater than or equal to the QL, the lowest concentration of that analyte in the associated samples should be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with the analyte's concentration < 10x the Preparation Blank concentration and ≥ the QL should be reprepared and reanalyzed. 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 ≤ (-QL), all associated samples with the analyte's concentration < 10x the QL should be reprepared 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 analysis; and Preparation Blanks are prepared and analyzed as appropriate for the data package (e.g., total number of samples, various types of matrices present, number of preparation batches, etc.).
2. For an ICB or a CCB, verify that if the absolute value of any target analyte was greater than or equal to the QL, the analysis was terminated, the problem corrected and documented in the data package narrative, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.
3. For a Preparation Blank, verify that if the concentration of any target analyte was greater than or equal to the QL, all associated samples with the analyte's concentration ≥ the QL but < 10x the Preparation Blank concentration were reprepared and reanalyzed for that analyte. Verify that if a concentration was ≤ (-QL) in a Preparation Blank, all associated samples with the analyte's concentration < 10x the QL were reprepared and reanalyzed.

4. Evaluation of field and equipment blanks should also be performed according to the QAPP or appropriate guidance.

E. Action

Refer to Anions Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient blanks.

1. For ICB analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCB analyses that do not meet the technical criteria, apply the actions 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.
3. For Preparation Blank analyses that do not meet the technical criteria, apply the actions to all associated samples prepared in the same preparation batch.
4. 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.
5. If the absolute value of an ICB or a CCB result is ≥ QL, the analysis should have been terminated and the affected samples re-analyzed. If samples were not re-analyzed, qualify as described in Table 3 below.
6. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and ≥ QL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, qualify as described in Table 3 below.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute run, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. For blank results ≤ (-MDL) but > (-QL), the possibility of false negatives exists.

NOTE: 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 the Laboratory Results Reports will not be on the same basis (units, dilution) as the calibration blank data. 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.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

Anions Table 3. Blank Actions

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Not analyzed at the specified frequency	Non-detect	UJ
		Detect	J
ICB/CCB	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
ICB/CCB		Non-detect	UJ

Blank Type	Blank Result	Sample Result	Action
	≤ (-MDL) but > (-QL)	Detect	J- or no qualification
ICB/CCB	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < ICB/CCB Result	Report at ICB/CCB Result and qualify U
		≥ ICB/CCB Result	J+ or no qualification
ICB/CCB	≤ (-QL)	Non-detect	UJ or R
		Detect < QL	J-
		≥ QL	J-
Preparation Blank	Not analyzed at specified frequency	Non-detect	UJ
		Detect	J
Preparation Blank/Field Blank	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
Preparation Blank/Field Blank	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification
Preparation Blank/Field Blank	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and qualify J+ or R
		≥ 10x the Preparation Blank Result	No qualification
Preparation Blank/Field Blank	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-
		≥ QL but < 10x QL	J-
		≥ 10x QL	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Laboratory Control Sample

A. Review Items

Laboratory LCS reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Aqueous/water and soil/sediment LCSs should 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.
2. One LCS should be prepared and analyzed for every group of aqueous/water or soil/sediment samples in a data package or with each batch of samples prepared, whichever is more frequent. The LCS should be spiked such that it contains each analyte at the level specified in the QAPP or at 2x the Quantitation Limit (QL) for the associated matrix.
3. All LCS %Rs should fall within the control limits of in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). If the Percent Recovery (%R) for the aqueous/water and soil/sediment LCS falls outside of the control limits, the analysis should be terminated, the problem corrected and documented in the data package narrative, and the samples prepared with that LCS reprepared and reanalyzed.

D. Evaluation

1. Verify, using the laboratory reports, preparation logs, and raw data, that the appropriate number of required LCSs were prepared and analyzed for the data package.
2. Verify that all results for each analyte fall within the established control limits.
3. Verify that the %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of each analyte measured in the analysis of the LCS
True (value) = Concentration of each analyte in the LCS

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

E. Action

Refer to Anions Table 4 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient LCSs. For an LCS analysis that does not meet the technical criteria, apply the actions to all samples in the same preparation batch.

Matrix spike data can be reviewed to determine batch quality if an LCS was not prepared and analyzed with the samples.

Anions Table 4. LCS Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not prepared with samples	J	UJ
LCS not prepared at specified concentrations	J	UJ
Aqueous/water and soil/sediment %R < 50%	J-	R
Aqueous/water and soil/sediment %R 50-79%	J-	UJ
Aqueous/water and soil/sediment %R 80-120%	No qualification	No qualification
Aqueous/water and soil/sediment %R 121-140%	J+	No qualification
Aqueous/water and soil/sediment %R > 140%	R	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

V. Duplicate Sample Analysis

A. Review Items

Data package Cover Page, laboratory duplicate reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Field samples should be used as source samples for duplicate analysis.
2. At least one duplicate sample should be prepared and analyzed from each group of samples of a similar matrix type (e.g., aqueous/water or soil/sediment) or for each data package. Duplicates cannot be averaged for reporting on the Laboratory Results Reports. Additional duplicate sample analyses may be required. Alternately, the data user may require that a specific sample be used for the duplicate sample analysis.
3. The Relative Percent Difference (RPD) control limit specified in the Quality Assurance Project Plan (QAPP) or of 20% should be used for original and duplicate sample values ≥ 5x the Quantitation Limit (QL).
4. For samples analyzed under the Statement of Work (SOW), a control limit of the QL should be used if either the sample or duplicate value is < 5x the QL.

D. Evaluation

1. Verify, from the data package Cover Page, laboratory reports, preparation log and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed.
2. Verify, using the raw data, that the duplicate results fall within the established control limits.
3. Verify that the duplicate analysis was performed on a field sample.
4. Verify that the RPD values are correct by recalculating one or more of the RPDs using the raw data and the following equation::

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

Where,

S = Sample Result (original)
D = Duplicate Result

NOTE: When the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only calculating the RPD. This will always yield an RPD of 200%.

E. Action

Refer to Anions Table 5 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient duplicates.

1. For a duplicate sample analysis that does not meet the technical criteria, apply the actions 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]. Additionally, use the

sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the duplicate sample should be qualified.

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

3. For high RPDs (i.e., > 100%), use professional judgment to qualify the data as this may be indicative of a sampling problem.

Anions Table 5. Duplicate Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Duplicate analysis not performed at the specified frequency	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%*	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
For samples analyzed under the SOW, original sample or duplicate sample results < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL*	J	UJ
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* 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**, The QAPP or the project-specific SOPs for data review may allow the use of less restrictive criteria (e.g., 35% RPD, 2x QL) to be assessed against duplicate soil samples.

VI. Spike Sample Analysis

A. Review Items

Data package Cover Page, laboratory matrix spike reports (if available), preparation logs, instrument printouts, and raw data in the data package.

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. Field samples should be used as source samples for matrix spike analysis.
2. At least one spiked sample should be prepared and analyzed from each group of samples with a similar matrix type (e.g., aqueous/water or soil/sediment), or for each data package. Additional matrix spike samples may be required. Alternately, the data user may require that a specific sample be used for the matrix spike analysis.
3. The spike Percent Recovery (%R) should be within the established acceptance limits. However, for samples analyzed under the Statement of Work (SOW), spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data should be reported unqualified, even if the %R does not meet the acceptance criteria.
4. If the spiked sample analysis was performed on the same sample that was selected for the duplicate sample analysis, spike calculations should be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for determining the %R.

NOTE: The final spike concentration required is presented in the method described in the Quality Assurance Project Plan (QAPP) or in the SOW.

D. Evaluation

1. Verify, using the data package Cover Page, laboratory reports, preparation log and raw data, that the appropriate number of required spiked samples was prepared and analyzed.
2. Verify that the matrix spike analysis was performed on field sample.
3. Verify, using the raw data, that all Matrix Spike sample results fall within the established control limits.
4. Verify that the %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation::

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

Where,

SSR = Spiking analyte result in the spiked sample
SR = Result of the same analyte in the original sample
SA = Spike added in the spiked sample

NOTE: When the Sample Result is reported as a non-detect, use SR = 0 only for calculating the %R.

E. Action

Refer to Anions Table 6 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient matrix spikes.

1. For a matrix spike sample analysis that does not meet the technical criteria, apply the actions 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]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.
2. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Anions Table 6. Spike Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike analysis not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
Matrix Spike %R < 35%	J-	R
Matrix Spike %R 35-79%	J-	UJ
Matrix Spike %R 80-120%	No qualification	No qualification
Matrix Spike %R > 120%	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

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**, the QAPP or the project-specific SOPs for data review 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.

VII. Target Analyte Quantitation

A. Review Items

Laboratory Result Reports, sample preparation sheets, data package narrative, instrument printouts and raw data.

B. Objective

The objective is to ensure that the reported results and quantitation limits for target analytes reported by the laboratory are accurate and sufficient to meet requirements.

C. Criteria

Final target analyte results and quantitation limits should be calculated according to the correct equations, taking into account amount of sample prepared, final sample volume, dilution factor, and percent solids, as appropriate.

D. Evaluation

1. Verify that the results for all positively identified target analytes are calculated and reported by the laboratory according to the equations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Verify that the reported Quantitation Limits (QLs) for non-detected target analytes are calculated and reported by the laboratory according to the equations in the Quality Assurance Project Plan (QAPP) or in the SOW.
3. Verify that all reported results and QLs have been adjusted to reflect percent solids, original sample mass/volume, and any applicable dilutions.

E. Action

1. If sample results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).
2. If the sample percent solids is < 30%, check if the sample was prepared at greater mass to maintain the QLs. Use professional judgment when this was not completed.

F. Example Equations

1. **Aqueous/Water Sample Concentration**

$$\text{Anion Concentration (mg/L)} = C \times DF$$

Where,

C = Instrument value in mg/L from the calibration curve
DF = Dilution Factor

2. **Soil/Sediment Sample Concentration**

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

Where,

Concentration = Analyte/Result (mg/kg)
C = Analyte Result from analysis (mg/L)
V_f = Final extraction volume (mL)
W = Initial aliquot amount (g)
S = %Solids/100
DF = Dilution Factor

3. **Adjusted DL (or MDL)/Adjusted QL**

To calculate the adjusted Detection Limit (DL) or adjusted Quantitation Limit (QL) for aqueous/water substitute the value of the DL or QL, in the appropriate units, into the "C" term in the equation above.

Calculate the adjusted DL or adjusted QL for soil/sediment samples as follows:

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

Where,

C	=	Detection Limit (DL) or Quantitation Limit (QL) (mg/kg)
W_m	=	Method required minimum sample weight (g)
W	=	Initial aliquot amount (g)
S	=	%Solids/100
DF	=	Dilution Factor

HEXAVALENT CHROMIUM DATA REVIEW

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

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, raw data, and narrative in the data package, 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 shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The technical holding time is determined from the date of sample collection to the date of analysis.
2. The technical holding time criteria for properly preserved (pH > 8, free chlorine < 0.1 mg/L) aqueous/water samples is 14 days or as specified in the Quality Assurance Project Plan (QAPP).
3. Aqueous/water samples may be maintained at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of collection until receipt at the laboratory, and should be stored at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of sample receipt until analysis.

D. Evaluation

1. Review the data package narrative, sampling documentation, and sample receipt forms to determine if the samples were properly preserved and arrived at the laboratory in proper condition (e.g., received intact, pH, free chlorine). If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples were properly stored at the laboratory. Use professional judgment to evaluate the effect of the problem on the sample results.
2. Verify that the analysis dates on the Laboratory Results Reports and the raw data are identical.
3. Establish the technical holding times by comparing the sample collection dates) on the sampling documentation with the dates of analysis on the Laboratory Results Reports and the raw data.

E. Action

Refer to Hexavalent Chromium Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected hexavalent chromium results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

If a discrepancy is found between the sample analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

Hexavalent Chromium Table 1. Preservation and Holding Time Actions

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with chlorine present	J*	R
Aqueous/water samples received with pH ≤ 8	J*	R
Aqueous/water samples received at a temperature > 6°C but ≤ 10°C	J	UJ
Aqueous/water samples received at a temperature > 10°C**	J-	R
Technical Holding Time: Aqueous/water samples > 14 days	J-	R
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* The true direction of any bias may be unknown in this case. Use caution in determining whether some detected analytes should be qualified as estimated low (J-) or as estimated high (J+), based on knowledge of the potential presence of other compounds that may react with the CrO_4^{2-} ion form. Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

** For samples received with shipping container temperatures > 10ºC, the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review may allow the use of higher temperature criteria before assessing any actions for the affected samples.

II. Calibration

A. Review Items

Laboratory initial calibration and calibration verification reports (if available), preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data in the data package.

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 should be successfully calibrated weekly or as specified in the Quality Assurance Project Plan (QAPP) or Statement of Work (SOW), and each time the instrument is set up. The calibration date and time should be included in the raw data.

NOTE: A blank and the number of calibration standards specified in the QAPP or in the SOW should be used to establish the calibration curve. At least one of the calibration standards should be at or below the Quantitation Limit (QL) in the QAPP or in the SOW but above the Detection Limit or the Method Detection Limit (MDL). Calibration standards at and above the QL should be continuous with none excluded to satisfy QC requirements. The calibration curve may be fitted using linear regression or weighted linear regression, or other fits as specified in the QAPP. Forcing the curve through zero is not recommended. For linear fits, the calibration curve should have a correlation coefficient greater than the value specified in the QAPP or in the SOW. The calculated percent differences (%Ds) or other specified statistical test values for all non-zero standards should fall within the limits in the QAPP or in the SOW.

2. Initial and Continuing Calibration Verification

   a. Initial Calibration Verification (ICV)

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

      ii. Analyses of the ICV should be conducted using a certified solution(s) of hexavalent chromium from an independent standard source, at concentration levels other than that used for instrument calibration and near the middle of the calibrated range (within ±30%).

   b. Continuing Calibration Verification (CCV)

      i. To ensure accuracy during each analytical sequence, the CCV should be analyzed and reported.

      ii. The CCV standard should be analyzed at the frequency specified in the QAPP, or every 10 samples during an analytical sequence. The CCV standard should also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

      iii. The CCV standard should be prepared using the same source and in the same matrix as the calibration standards at a concentration at or near the mid-level (within ±30%) of the respective calibration curve.

      iv. The same CCV standard solution should be used throughout the analysis for a data package.

v. The CCV should 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 and documented in the data package narrative, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

vi. An instrument blank should not be analyzed before the CCV.

D. Evaluation

1. Verify that the instrument was calibrated as specified in the QAPP or in the SOW and each time the instrument was set up, utilizing a blank and at least the minimum number of standards specified by the QAPP or in the SOW. Confirm that at least one of the calibration standards was analyzed at or below the QL in the QAPP or in the SOW, but above the Detection Limit or MDL and that all subsequent calibration standards are consecutive with none removed to satisfy QC requirements. For linear fits, verify that the correlation coefficient of the calibration curve is greater than the value specified in the QAPP or in the SOW. Verify that the %Ds for all non-zero standards are within the SOW limits or that other statistical test values are within the limits specified in the QAPP.
2. Verify that the ICV and CCV standards were analyzed at the specified frequency and at the appropriate concentration. Verify that acceptable Percent Recovery (%R) results were obtained.
3. Verify that the ICV and CCV %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of hexavalent chromium measured in the analysis of the ICV or CCV solution

True (value) = Concentration of hexavalent chromium in the ICV or CCV source

E. Action

Refer to Hexavalent Chromium Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected hexavalent chromium results in the samples associated with deficient initial calibrations or calibration verification standards.

1. For initial calibrations or ICV standard analyses that do not meet the technical criteria, apply the actions to the associated samples reported from the analytical sequence.
2. For CCV standard analyses that do not meet the technical criteria, apply the actions 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.
3. 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 the QL but above the MDL), qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

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

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

Hexavalent Chromium Table 2. Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed or not performed at specified frequency	R	R
Calibration incomplete (insufficient number of standards or required concentrations missing)	J or R	UJ or R
For linear fits, the correlation coefficient < 0.995	J	UJ
%D outside ±30%, or other specified statistical test values outside limits	J	UJ
ICV/CCV not performed at specified frequency	J	UJ
ICV/CCV %R < 70%	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%	J+ or R	No qualification
Instrument blank analyzed prior to CCV	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

III. Blanks

A. Review Items

Laboratory blanks reports (if available), preparation logs, calibration standard logs, instrument logs, and raw data in the data package.

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) should be analyzed after the analytical standards, but not before analysis of the ICV standard during the initial calibration of the instrument. The ICB result (absolute value) should not be greater than or equal to the Quantitation Limit (QL).
3. A Continuing Calibration Blank (CCB) should be analyzed immediately after every Continuing Calibration Verification (CCV) standard. The CCB should be analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) during the analytical sequence. The CCB should 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) should not be greater than or equal to the QL.
4. At least one Preparation Blank should be prepared and analyzed with every data package, or with each batch of samples prepared, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
5. If the hexavalent chromium concentration in the Preparation Blank is greater than or equal to the QL, the lowest concentration of hexavalent chromium in the associated samples should be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with a hexavalent chromium concentration < 10x the Preparation Blank concentration and ≥ the QL should be reprepared and reanalyzed. The laboratory is not to correct the sample concentration for the blank value.
6. If the hexavalent chromium concentration in the Preparation Blank is ≤ (-QL), all associated samples with a hexavalent chromium concentration < 10x the QL should be reprepared 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 analysis; and Preparation Blanks are prepared and analyzed as appropriate for the data package (e.g., total number of samples, various types of matrices present, number of preparation batches, etc.).
2. For an ICB or a CCB, verify that if the absolute value of the hexavalent chromium concentration was greater than or equal to the QL, the analysis was terminated, the problem corrected and documented in the data package narrative, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.
3. For a Preparation Blank, verify that if the concentration of hexavalent chromium was greater than or equal to the QL, all associated samples with a hexavalent chromium concentration ≥ the QL but < 10x the Preparation Blank concentration were reprepared and reanalyzed. Verify that if the hexavalent chromium concentration was ≤ (-QL) in a Preparation Blank, all associated samples with hexavalent chromium concentration < 10x the QL were reprepared and reanalyzed.

4. Evaluation of field and equipment blanks should also be performed according to the QAPP or appropriate guidance.

E. Action

Refer to Hexavalent Chromium Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected hexavalent chromium results in the samples associated with deficient blanks.

1. For ICB analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCB analyses that do not meet the technical criteria, apply the actions 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.
3. For Preparation Blank analyses that do not meet the technical criteria, apply the actions to all associated samples prepared in the same preparation batch.
4. 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.
5. If the absolute value of an ICB or a CCB result is ≥ QL, the analysis should have been terminated and the affected samples re-analyzed. If samples were not re-analyzed, qualify as described in Table 3 below.
6. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and ≥ QL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, qualify as described in Table 3 below.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute run, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. For blank results ≤ (-MDL) but > (-QL), the possibility of false negatives exists.

NOTE: The blank analyses may not involve the same volumes or dilution factors as the associated samples. It may be easier to work with the raw data for comparison purposes.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

Hexavalent Chromium Table 3. Blank Actions

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Not analyzed at the specified frequency	Non-detect	UJ
		Detect	J
ICB/CCB	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
ICB/CCB	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < ICB/CCB Result	Report at ICB/CCB Result and qualify U
		≥ ICB/CCB Result	J+ or no qualification
ICB/CCB	≤ (-QL)	Non-detect	UJ or R
		Detect < QL	J-
		≥ QL	J-
Preparation Blank	Not analyzed at specified frequency	Non-detect	UJ
		Detect	J
Preparation Blank/Field Blank	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
Preparation Blank/Field Blank	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification
Preparation Blank/Field Blank	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and qualify J+ or R
		≥ 10x the Preparation Blank Result	No qualification
Preparation Blank/Field Blank	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-
		≥ QL but < 10x QL	J-
		≥ 10x QL	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Laboratory Control Sample

A. Review Items

Laboratory LCS reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Aqueous/water LCSs should be analyzed for hexavalent chromium utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples.
2. One LCS should be prepared and analyzed for every group of aqueous/water samples in a data package or with each batch of samples prepared, whichever is more frequent. The LCS should be spiked such that it contains hexavalent chromium at the levels specified in the Quality Assurance Project Plan (QAPP) or at 2x the Quantitation Limit (QL).
3. All LCS Percent Recoveries (%Rs) should fall within the control limits of in the QAPP or in the Statement of Work (SOW). If the %R for the aqueous/water LCS falls outside of the control limits, the analysis should be terminated, the problem corrected and documented in the data package narrative, and the samples prepared with that LCS reprepared and reanalyzed.

D. Evaluation

1. Verify, using the laboratory reports, preparation logs, and raw data, that the appropriate number of required LCSs were prepared and analyzed for the data package.
2. Verify that all results for each analyte fall within the established control limits.
3. Verify that the %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of each analyte measured in the analysis of the LCS
True (value) = Concentration of each analyte in the LCS

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

E. Action

Refer to Hexavalent Chromium Table 4 below for the evaluation criteria and corresponding actions for detected and non-detected hexavalent chromium results in the samples associated with deficient LCSs. For an LCS analysis that does not meet the technical criteria, apply the actions to all samples in the same preparation batch.

Matrix spike data can be reviewed to determine batch quality if an LCS was not prepared and analyzed with the samples.

Hexavalent Chromium Table 4. LCS Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not prepared with samples	J	UJ
LCS not prepared at specified concentration	J	UJ
Aqueous/water %R < 40%	J-	R
Aqueous/water %R 40-69%	J-	UJ
Aqueous/water %R 70-130%	No qualification	No qualification
Aqueous/water %R 131-150%	J+	No qualification
Aqueous/water %R > 150%	R	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

V. Duplicate Sample Analysis

A. Review Items

Data package Cover Page, laboratory duplicate reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Field samples should be used as source samples for duplicate analysis.
2. At least one duplicate sample should be prepared and analyzed for each data package. Duplicates cannot be averaged for reporting on the Laboratory Results Report. Additional duplicate sample analyses may be required. Alternately, the data user may require that a specific sample be used for the duplicate sample analysis.
3. The Relative Percent Difference (RPD) control limit specified in the Quality Assurance Project Plan (QAPP) or of 20% should be used for original and duplicate sample values ≥ 5x the Quantitation Limit (QL).
4. For samples analyzed under the Statement of Work (SOW), a control limit of the QL should be used if either the sample or duplicate value is < 5x the QL.

D. Evaluation

1. Verify, from the data package Cover Page, laboratory reports, preparation log and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed.
2. Verify, using the raw data, that the duplicate results fall within the established control limits.
3. Verify that the duplicate analysis was performed on a field sample.
4. Verify that the RPD values are correct by recalculating one or more of the RPDs using the raw data and the following equation::

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

Where,

S = Sample Result (original)

D = Duplicate Result

NOTE: When the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only for calculating the RPD. This will always yield an RPD of 200%.

E. Action

Refer to Hexavalent Chromium Table 5 below for the evaluation criteria and corresponding actions for detected and non-detected hexavalent chromium results in the samples associated with deficient duplicates.

1. For a duplicate sample analysis that does not meet the technical criteria, apply the actions to all samples if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity. Use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the duplicate sample should be qualified.

2. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.
3. For high RPDs (i.e., > 100%), use professional judgment to qualify the data as this may be indicative of a sampling problem.

Hexavalent Chromium Table 5. Duplicate Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Duplicate analysis not performed at the specified frequency	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
For samples analyzed under the SOW, original sample or duplicate sample results < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL	J	UJ
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

VI. Spike Sample Analysis

A. Review Items

Data package Cover Page, laboratory matrix spike reports (if available), preparation logs, instrument printouts, and raw data in the data package.

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. Field samples should be used as source samples for matrix spike analysis.
2. At least one spiked sample should be prepared and analyzed for each data package. Additional matrix spike sample analyses may be required. Alternately, the data user may require that a specific sample be used for the matrix spike sample analysis.
3. The spike Percent Recovery (%R) should be within the established acceptance limits. However, for samples analyzed under the Statement of Work (SOW), spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data should be reported unqualified, even if the %R does not meet the acceptance criteria.
4. If the spiked sample analysis was performed on the same sample that was selected for the duplicate sample analysis, spike calculations should be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for determining the %R.

NOTE: The final spike concentration required is presented in the method described in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).

D. Evaluation

1. Verify, using the data package Cover Page, laboratory reports, and raw data, that the appropriate number of required spiked samples was prepared and analyzed.
2. Verify that the matrix spike analysis was performed on a field sample.
3. Verify, using the raw data, that all Matrix Spike sample results fall within the established control limits.
4. Verify that the %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation::

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

Where,

SSR = Spiking analyte result in the spiked sample
SR = Result of the same analyte in the original sample
SA = Spike added in the spiked sample

NOTE: When the Sample Result is reported as a non-detect, use SR = 0 only for calculating the %R.

E. Action

Refer to Hexavalent Chromium Table 6 below for the evaluation criteria and corresponding actions for detected and non-detected hexavalent chromium results in the samples associated with deficient matrix spikes.

1. For a matrix spike sample analysis that does not meet the technical criteria, apply the actions to all samples if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity. Use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.
2. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Hexavalent Chromium Table 6. Spike Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike analysis not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
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

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VII. Target Analyte Quantitation

A. Review Items

Laboratory Result Reports, sample preparation sheets, data package narrative, instrument printouts and raw data.

B. Objective

The objective is to ensure that the reported results and quantitation limits for target analytes reported by the laboratory are accurate and sufficient to meet requirements.

C. Criteria

Final target analyte results and quantitation limits should be calculated according to the correct equations, taking into account amount of sample prepared, final sample volume, dilution factor, and percent solids, as appropriate.

D. Evaluation

1. Verify that the results for all positively identified target analytes are calculated and reported by the laboratory according to the equations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Verify that the reported Quantitation Limits (QLs) for non-detected target analytes are calculated and reported by the laboratory according to the equations in the QAPP or in the SOW.
3. Verify that all reported results and QLs have been adjusted to reflect percent solids, original sample mass/volume, and any applicable dilutions.

E. Action

If sample results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).

F. Example Equations

1. **Aqueous/Water Sample Concentration**

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

Where,

C = Instrument value in µg/L from the calibration curve
DF = Dilution Factor

2. **Adjusted DL (or MDL)/Adjusted QL**

To calculate the adjusted Detection Limit (DL) or adjusted Quantitation Limit (QL) for aqueous/water samples, substitute the value of the DL or QL, in the appropriate units, into the "C" term in the equation.

$$\text{Cr(VI) DL or QL } (\mu g/L) = C \times DF$$

Where,

C = DL or QL for Instrument in µg/L
DF = Dilution Factor

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TOTAL ORGANIC CARBON (TOC) DATA REVIEW

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

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, raw data, and narrative in the data package, 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 shipping or storage conditions and the holding time of the sample.

C. Criteria

1. The technical holding time is determined from the date of sample collection to the date of analysis.
2. The technical holding time criteria for aqueous/water samples 28 days or as specified in the Quality Assurance Project Plan (QAPP), preserved (with sulfuric or phosphoric acid) to pH < 2.
3. The technical holding time criteria for soil/sediment samples is 28 days, or as specified in the QAPP.
4. Aqueous/water samples and soil/sediment samples should be maintained at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of collection until receipt at the laboratory and should be stored at ≤ 6ºC (but not frozen) or as specified in the QAPP from the time of sample receipt until analysis.

D. Evaluation

1. Review the data package narrative, sampling Record documentation, and sample receipt forms to determine if the samples were properly preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperature at receipt, pH). If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples were properly stored at the laboratory. Use professional judgment to evaluate the effect of the problem on the sample results.
2. Verify that the analysis dates on the Laboratory Results Reports and the raw data are identical.
3. Establish the technical holding times by comparing the sample collection dates on the sampling documentation with the dates of analysis on the Laboratory Results Reports and the raw data.

E. Action

Refer to TOC Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected TOC results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

If a discrepancy is found between the sample analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

TOC Table 1. Preservation and Holding Time Actions

Criteria	Action	
	Detect	**Non-detect**
Aqueous/water samples received with pH ≥ 2	J-	R
Aqueous/water and soil/sediment samples received at a temperature > 6°C but ≤ 10°C	J	UJ
Aqueous/water and soil/sediment samples received at a temperature > 10°C*	J-	R
Technical Holding Time: Aqueous/water samples > 28 days	J-	R
Technical Holding Time: Soil/sediment samples > 28 days	J-	R
Samples properly preserved and analyzed within specified holding time	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

* For samples received with shipping container temperatures > 10ºC, the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review may allow the use of higher temperature criteria before assessing any actions for the affected samples.

II. Calibration

A. Review Items

Laboratory initial calibration and calibration verification reports (if available), preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data in the data package.

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 should be successfully calibrated daily, or as specified in the Quality Assurance Project Plan (QAPP) or the Statement of Work (SOW), and each time the instrument is set up. The calibration date and time should be included in the raw data.

NOTE: A blank and the number of calibration standards specified in the QAPP or in the SOW should be used to establish the calibration curve. At least one of the calibration standards should be at or below the Quantitation Limit (QL) in the QAPP or in the SOW but above the Detection Limit or the Method Detection Limit (MDL). Calibration standards at and above the QL should be continuous with none excluded to satisfy Quality Control (QC) requirements. The calibration curve may be fitted using linear regression or weighted linear regression, or other fits as specified in the QAPP. The curve may be forced through zero. For linear fits, the calibration curve should have a correlation coefficient greater than the value specified in the QAPP or in the SOW. The calculated percent differences (%Ds) or other specified statistical test values for all non-zero standards should fall within the limits in the QAPP or in the SOW.

2. Initial and Continuing Calibration Verification

   a. Initial Calibration Verification (ICV)

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

      ii. Analyses of the ICV should be conducted using a certified solution of TOC from an independent standard source, at concentration levels other than that used for instrument calibration and near the middle of the calibrated range (within ±30%).

   b. Continuing Calibration Verification (CCV)

      i. To ensure accuracy during each analytical sequence, the CCV standard should be analyzed and reported.

      ii. The CCV standard should be analyzed at the frequency specified in the QAPP, or every 10 samples during an analytical sequence. The CCV standard should also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

      iii. The CCV standard should be prepared using the same source and in the same acid matrix as the calibration standards at a concentration at or near the mid-level (within ±30%) of the respective calibration curve.

      iv. The same CCV standard solution should be used throughout the analysis for a data package.

v. The CCV should 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 and documented in the data package narrative, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

vi. An instrument blank should not be analyzed before the CCV.

D. Evaluation

1. Verify that the instrument was calibrated as specified in the QAPP or in the SOW and each time the instrument was set up, utilizing a blank and at least the minimum number of standards specified by the QAPP or in the SOW. Confirm that at least one of the calibration standards was analyzed at or below the QL in the QAPP or in the SOW, but above the Detection Limit or MDL and that all subsequent calibration standards are consecutive with none removed to satisfy QC requirements. For linear fits, verify that the correlation coefficient of the calibration curve is greater than the value specified in the QAPP or in the SOW. Verify that the %Ds for all non-zero standards are within the SOW limits or that other statistical test values are within the limits specified in the QAPP.
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. Confirm that an instrument blank was not analyzed before the CCV.
4. Verify that the ICV and CCV %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of TOC measured in the analysis of the ICV or CCV solution
True (value) = Concentration of TOC in the ICV or CCV source

E. Action

Refer to TOC Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected TOC results in the samples associated with deficient initial calibrations or calibration verification standards.

1. For initial calibrations or ICV standard analyses that do not meet the technical criteria, apply the actions to the associated samples reported from the analytical sequence.
2. For CCV standard analyses that do not meet the technical criteria, apply the actions 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.
3. 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 the QL but above the MDL), qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

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

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

TOC Table 2. Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Calibration not performed or not performed at specified frequency	R	R
Calibration incomplete (insufficient number of standards or required concentrations missing)	J or R	UJ or R
For linear fits, the correlation coefficient < 0.995	J	UJ
%D outside ±30%, or other specified statistical test values outside limits	J	UJ
ICV/CCV not performed at specified frequency	J	UJ
ICV/CCV %R < 65%	J- or R	R
ICV/CCV %R 65-79%	J-	UJ
ICV/CCV %R 80-120%	No qualification	No qualification
ICV/CCV %R 120-135%	J+	No qualification
ICV/CCV %R > 135%	J+ or R	No qualification
Instrument blank analyzed prior to CCV	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

III. Blanks

A. Review Items

Laboratory blanks reports (if available), preparation logs, calibration standard logs, instrument logs, and raw data in the data package.

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) should be analyzed after the analytical standards, but not before analysis of the ICV standard during the initial calibration of the instrument. The ICB result (absolute value) should not be greater than or equal to the Quantitation Limit (QL).
3. A Continuing Calibration Blank (CCB) should be analyzed immediately after every Continuing Calibration Verification (CCV) standard. The CCB should be analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) during the analytical sequence. The CCB should 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) should not be greater than or equal to the QL.
4. At least one Preparation Blank should be prepared and analyzed for each matrix, with every data package, or with each batch of samples prepared, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
5. If the TOC concentration in the Preparation Blank is greater than or equal to the QL, the lowest concentration of TOC in the associated samples should be ≥ 10x the Preparation Blank concentration. Otherwise, all associated samples with a TOC concentration < 10x the Preparation Blank concentration and ≥ the QL should be reprepared and reanalyzed. The laboratory is not to correct the sample concentration for the blank value.
6. If the TOC concentration in the Preparation Blank is less than or equal to the limit in the QAPP multiplied by -1 or ≤ (-QL), all associated samples with a TOC concentration < 10x the limit specified in the QAPP or the QL should be reprepared 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 analysis; and Preparation Blanks are prepared and analyzed as appropriate for the data package (e.g., total number of samples, various types of matrices present, number of preparation batches, etc.).
2. For an ICB or a CCB, verify that if the absolute value of the TOC concentration was greater than or equal to the QL the analysis was terminated, the problem corrected and documented in the data package narrative, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.
3. For a Preparation Blank, verify that if the concentration of TOC was greater than or equal to the QL in a Preparation Blank, all associated samples with a TOC concentration ≥ the QL but < 10x the Preparation Blank concentration were reprepared and reanalyzed. Verify that if the TOC concentration was ≤ (-QL) in a Preparation Blank, all associated samples with a TOC concentration < 10x the QL were reprepared and reanalyzed.

4. Evaluation of field and equipment blanks should also be performed according to the QAPP or appropriate guidance.

E. Action

Refer to TOC Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected TOC results in the samples associated with deficient blanks.

1. For ICB analyses that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.
2. For CCB analyses that do not meet the technical criteria, apply the actions 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.
3. For Preparation Blank analyses that do not meet the technical criteria, apply the actions to all associated samples prepared in the same preparation batch.
4. 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.
5. If the absolute value of an ICB or a CCB result is ≥ QL, the analysis should have been terminated and the affected samples re-analyzed. If samples were not re-analyzed, qualify as described in Table 3 below.
6. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and ≥ QL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, qualify as described in Table 3 below.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute run, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. For blank results ≤ (-MDL) but > (-QL), the possibility of false negatives exists.

NOTE: 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 the Laboratory Results Reports will not be on the same basis (units, dilution) as the calibration blank data. 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.

When two separate qualifiers are listed as actions, use professional judgment to qualify detects and non-detects based on the extent to which the criteria is not met.

TOC Table 3. Blank Actions

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Not analyzed at the specified frequency	Non-detect	UJ
		Detect	J
ICB/CCB	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
ICB/CCB		Non-detect	UJ

Blank Type	Blank Result	Sample Result	Action
	≤ (-MDL) but > (-QL)	Detect	J- or no qualification
ICB/CCB	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < ICB/CCB Result	Report at ICB/CCB Result and qualify U
		≥ ICB/CCB Result	J+ or no qualification
ICB/CCB	≤ (-QL)	Non-detect	UJ or R
		Detect < QL	J-
		≥ QL	J-
Preparation Blank	Not analyzed at specified frequency	Non-detect	UJ
		Detect	J
Preparation Blank/Field Blank	Detect < QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL	J+ or no qualification
Preparation Blank/Field Blank	≤ (-MDL) but > (-QL)	Non-detect	UJ
		Detect	J- or no qualification
Preparation Blank/Field Blank	≥ QL	Non-detect	No qualification
		Detect < QL	Report at QL and qualify U
		≥ QL but < 10x the Preparation Blank Result	Report at Preparation Blank Result and qualify J+ or R
		≥ 10x the Preparation Blank Result	No qualification
Preparation Blank/Field Blank	≤ (-QL)	Non-detect	UJ
		Detect < QL	J-
		≥ QL but < 10x QL	J-
		≥ 10x QL	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Laboratory Control Sample

A. Review Items

Laboratory LCS reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Aqueous/water and soil/sediment LCSs should be analyzed for TOC utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples.
2. One LCS should be prepared and analyzed for every group of aqueous/water or soil/sediment samples in a data package or with each batch of samples prepared, whichever is more frequent. The LCS should be spiked such that it contains TOC at the levels specified in the Quality Assurance Project Plan (QAPP) or at 2x the Quantitation Limit (QL) for the associated matrix.
3. All LCS Percent Recoveries (%Rs) should fall within the control limits of in the QAPP or in the SOW. 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 documented in the data package narrative, and the samples prepared with that LCS reprepared and reanalyzed.

D. Evaluation

1. Verify, using the laboratory reports, preparation logs, and raw data, that the appropriate number of required LCSs were prepared and analyzed for the data package.
2. Verify that all %R values fall within the established control limits.
3. Verify that the %R values are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

Found (value) = Concentration of each analyte measured in the analysis of the LCS
True (value) = Concentration of each analyte in the LCS

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

E. Action

Refer to TOC Table 4 below for the evaluation criteria and corresponding actions for detected and non-detected TOC results in the samples associated with deficient LCSs. For an LCS analysis that does not meet the technical criteria, apply the actions to all samples in the same preparation batch.

Matrix spike data can be reviewed to determine batch quality if an LCS was not prepared and analyzed with the samples.

TOC Table 4. LCS Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not prepared with samples	J	UJ
LCS not prepared at specified concentration	J	UJ
Aqueous/water and soil/sediment %R < 45%	J-	R
Aqueous/water and soil/sediment %R 45-74%	J-	UJ
Aqueous/water and soil/sediment %R 75-125%	No qualification	No qualification
Aqueous/water and soil/sediment %R 126-145%	J+	No qualification
Aqueous/Water and soil/sediment %R > 145%	R	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

V. Duplicate Sample Analysis

A. Review Items

Data package Cover Page, laboratory duplicate reports (if available), preparation logs, instrument printouts, and raw data in the data package.

B. Objective

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

C. Criteria

1. Field samples should be used as source samples for duplicate analysis.
2. At least one duplicate sample should be prepared and analyzed from each group of samples of a similar matrix type (e.g., aqueous/water or soil/sediment) or for each data package. Duplicates cannot be averaged for reporting on the Laboratory Results Reports. Additional duplicate sample analyses may be required. Alternately, the data user may require that a specific sample be used for the duplicate sample analysis.
3. The Relative Percent Difference (RPD) control limit specified in the Quality Assurance Project Plan (QAPP) or of 20% should be used for original and duplicate sample values ≥ 5x the Quantitation Limit (QL).
4. For samples analyzed under the Statement of Work (SOW), a control limit of the QL should be used if either the sample or duplicate value is < 5x the QL.

D. Evaluation

1. Verify, from the data package Cover Page, laboratory reports, preparation log and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed.
2. Verify, using the raw data, that the duplicate results fall within the established control limits.
3. Verify that the duplicate analysis was performed on a field sample.
4. Verify that the RPD values are correct by recalculating one or more of the RPDs using the raw data and the following equation:

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

Where,

S = Sample Result (original)
D = Duplicate Result

NOTE: When the Sample or Duplicate Result is reported as a non-detect, use a value of zero (0) only for calculating the RPD. This will always yield an RPD of 200%.

E. Action

Refer to TOC Table 5 below for the evaluation criteria and corresponding actions for detected and non-detected TOC results in the samples associated with deficient duplicates.

1. For a duplicate sample analysis that does not meet the technical criteria, apply the actions 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), alkalinity or buffering capacity, reactive sulfide, anions]. Additionally, use the sample data (e.g., similar concentrations of TOC) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the duplicate sample should be qualified.
2. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.
3. For high RPDs (i.e., > 100%), use professional judgment to qualify the data as this may be indicative of a sampling problem.

TOC Table 5. Duplicate Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Duplicate analysis not performed at the specified frequency	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD > 20%*	J	UJ
Both original sample and duplicate sample results are ≥ 5x QL and RPD ≤ 20%	No qualification	No qualification
RPD > 100%	Use professional judgment	Use professional judgment
For samples analyzed under the SOW, original sample or duplicate sample results < 5x QL (including non-detects) and absolute difference between sample and duplicate > QL*	J	UJ
For samples analyzed under the SOW, original sample or duplicate sample result < 5x QL (including non-detects) and absolute difference between sample and duplicate ≤ QL	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* 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**, The QAPP or the project-specific Standard Operating Procedures (SOPs) for data review may allow the use of less restrictive criteria (e.g., 35% RPD, 2x QL) to be assessed against duplicate soil samples.

VI. Spike Sample Analysis

A. Review Items

Data package Cover Page, laboratory matrix spike reports (if available), preparation logs, instrument printouts, and raw data in the data package.

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. Field samples should be used as source samples for matrix spike analysis.
2. At least one spiked sample should be prepared and analyzed from each group of samples with a similar matrix type (e.g., aqueous/water or soil/sediment), or for each data package. Additional matrix spike sample analyses may be required. Alternately, the data user may require that a specific sample be used for the matrix spike sample analysis.
3. The spike Percent Recovery (%R) should be within the established acceptance limits. However, for samples analyzed under the Statement of Work (SOW), spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data should be reported unqualified, even if the %R does not meet the acceptance criteria.
4. If the spiked sample analysis was performed on the same sample that was selected for the duplicate sample analysis, spike calculations should be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for determining the %R.

NOTE: The final spike concentration required is presented in the method described in the Quality Assurance Project Plan (QAPP) or in the SOW.

D. Evaluation

1. Verify, using the data package Cover Page, laboratory reports, preparation log and raw data, that the appropriate number of required spiked samples was prepared and analyzed.
2. Verify that the matrix spike analysis was performed on a field sample.
3. Verify, using the raw data, that all Matrix Spike sample results fall within the established control limits.
4. Verify that the %R values for the matrix spike are correct by recalculating one or more of the %Rs using the raw data and the following equation:

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

Where,

SSR = Spiking analyte result in spiked sample
SR = Result of the same analyte in the original sample
SA = Spike added in the spike sample

NOTE: When the Sample Result is reported as a non-detect, use SR = 0 only for calculating the %R.

E. Action

Refer to TOC Table 6 below for the evaluation criteria and corresponding actions for detected and non-detected TOC results in the samples associated with deficient matrix spikes.

1. For a matrix spike sample analysis that does not meet the technical criteria, apply the actions 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), alkalinity or buffering capacity, reactive sulfide, anions]. Additionally, use the sample data (e.g., similar concentrations of TOC) in determining similarity between samples in the data package. Two possible determinations are: 1) only some of the samples in the data package 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 analysis, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.
2. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

TOC Table 6. Spike Sample Actions

Criteria	Action	
	Detect	**Non-detect**
Matrix Spike analysis not performed at the specified frequency	J	UJ
Matrix Spike not prepared from field sample	J	UJ
Matrix Spike %R < 25%	J-	R
Matrix Spike %R 25-69%	J-	UJ
Matrix Spike %R 70-130%	No qualification	No qualification
Matrix Spike %R > 130%	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria; however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

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**, the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review 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.

VII. Target Analyte Quantitation

A. Review Items

Laboratory Result Reports, sample preparation sheets, data package narrative, instrument printouts and raw data.

B. Objective

The objective is to ensure that the reported results and quantitation limits for target analytes reported by the laboratory are accurate and sufficient to meet requirements.

C. Criteria

Final target analyte results and quantitation limits should be calculated according to the correct equations, taking into account amount of sample prepared, final sample volume, dilution factor, and percent solids, as appropriate.

D. Evaluation

1. Verify that the results for all positively identified target analytes are calculated and reported by the laboratory according to the equations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Verify that the reported Quantitation Limits (QLs) for non-detected target analytes are calculated and reported by the laboratory according to the equations in the QAPP or in the SOW.
3. Verify that all reported results and QLs have been adjusted to reflect percent solids, original sample mass/volume, and any applicable dilutions.

E. Action

1. If sample results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).
2. If the sample percent solids is < 30%, check if the sample was prepared at greater mass to maintain the QLs. Use professional judgment when this was not completed.

F. Example Equations

1. **Aqueous/Water Sample Concentration**

$$\text{TOC Concentration } (\text{mg/L}) = C \times DF$$

Where,

C = Instrument value in mg/L from the calibration curve
DF = Dilution Factor

2. **Soil/Sediment Sample Concentration**

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

Where,

Concentration = Analyte/Result (mg/kg)
C = Analyte Result from analysis (mg/L)
V_f = Final digestion volume mL
W = Initial aliquot amount g
S = %Solids/100
DF = Dilution Factor

3. **Adjusted DL (or MDL)/Adjusted QL**

   To calculate the adjusted Detection Limit (DL) or adjusted Quantitation Limit (QL) for aqueous/water substitute the value of the DL or QL, in the appropriate units, into the "C" term in the equation above.

   Calculate the adjusted DL or adjusted QL for soil/sediment samples as follows:

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

   Where,

C	=	Detection Limit (DL) or Quantitation Limit (QL) (mg/kg)
W_m	=	Method required minimum sample weight (g)
W	=	Initial aliquot amount (g)
S	=	%Solids/100
DF	=	Dilution Factor

APPENDIX A: GLOSSARY

Action Limit – A result for a Performance Evaluation (PE) sample that is outside the 99% (±3σ) control limits. The laboratory may be required to apply and document corrective actions to bring the analytical results back into control.

Analyte – The element or ion an analysis seeks to determine; the element of interest.

Analytical Sample – Any prepared field sample or extract thereof that is introduced into an instrument for the purpose of measuring any target analyte. This definition excludes any instrument quality control samples (e.g., standards associated with initial calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), Continuing Calibration Blank (CCB), and tune verifications). The following are also defined as analytical samples: diluted samples; matrix spike and matrix spike duplicate samples; duplicate samples; serial dilution samples, post-digestion/post-distillation spike samples; Laboratory Control Samples (LCSs); Performance Evaluation (PE) samples; Preparation/Method Blanks; Field Blanks (FBs); and Leachate Extraction Blanks (LEBs).

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 should 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 reagents and in the same concentration as those used in the analytical sample preparation. This blank is digested/distilled 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 calibration 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.

Chain of Custody (COC) Record – A sample identification form completed by the sampler, which accompanies the sample during shipment to the laboratory and is used to document sample identity, sample chain of custody, sample condition, and sample receipt by the laboratory.

Contamination – A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may result from other samples, sampling equipment, or from introduction while in transit, from laboratory reagents, from the laboratory environment, or from analytical instruments.

Continuing Calibration Blank (CCB) – A reagent water sample that is designed to detect any carryover contamination.

Continuing Calibration Verification (CCV) – A single parameter or multi-parameter standard solution prepared from the same source as the initial calibration standards by the analyst and used to periodically verify the stability of the instrument calibration during analysis of samples. The CCV can be one of the calibration standards with the concentration near the middle of the calibration range.

Control Limits – A range within which specified measurement results should fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

Data Package Narrative – Portion of the data package which includes laboratory information, sample identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Data Quality Assessment (DQA) – The scientific and statistical evaluation of environmental data to determine if they meet the planning objectives of the project, and thus are of the right type, quality, and quantity to support their intended use; refer to EPA QA/G-9R.

Data Quality Objectives (DQO) - Qualitative and quantitative statements that clarify technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Detection Limit (DL) - A generic term for the minimum measured concentration of a substance that can be reported with a specified confidence that the measured concentration is distinguishable from blank results. Includes Method Detection Limit (MDL), Limit of Detection (LOD), and other means of establishing this limit.

Duplicate – A second aliquot of a sample that is treated the same as the original sample in order to evaluate the precision.

Field Blank (FB) –A blank used to provide information about contaminants that may be introduced during sample collection, shipment, storage, and/or preparation and analysis in the laboratory. Examples of field blanks include trip blanks, rinse blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

Field Duplicate – A duplicate sample generated in the field, not in the laboratory.

Field Quality Control (QC) – Any QC samples submitted from the field to the laboratory. Examples include, but are not limited to, field blanks, and field duplicates.

Field Sample – A portion of material received from the field to be analyzed for analytes of interest.

Initial Calibration – Analysis of analytical standards at 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) – The analysis of 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 solution(s) should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources.

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 – The place where the samples are processed and tested.

Laboratory Control Sample (LCS) – A reference matrix spiked with target analytes at a known concentration. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

Leachate Extraction Blank (LEB) – A blank carried through the entire Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction with the resulting leachate extracted, digested, or distilled by an appropriate aqueous method from the analytical method.

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 is not synonymous with phase (liquid or solid).

Matrix Spike – Aliquot of a sample (aqueous/water or soil/sediment) fortified (spiked) with known quantities of specific analytes and subjected to the entire analytical procedure to estimate recovery.

Method Detection Limit (MDL) – The minimum measured concentration of a substance that can be reported with 99% confidence such that the measured concentration is distinguishable from method blank results. . Additional information about the procedure is provided in Title 40 of the Code of Federal Regulations (CFR), Chapter 1, Subchapter D, part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit, Revision 2.

Percent Difference (%D) – The relative difference between two values (e.g., a measured and expected value) expressed as a percentage of one of the values (e.g., expected value).

Percent Solids (%Solids) – The proportion of solid in a soil/sediment sample determined by drying an aliquot of the sample.

Performance Evaluation (PE) Sample – A sample prepared by a third party at known concentrations that are unknown to the analytical laboratory and is provided to test whether the laboratory can produce analytical results within specified performance limits.

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. For ICP-AES analysis of wipes, when possible a preparation blank includes a clean wipe.

Preparation Log – A record of sample preparation (e.g., digestion, extraction, distillation) at the laboratory.

Quality Assurance Project Plan (QAPP) – A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization, or laboratory for ensuring quality in its products and utility to its users.

Quantitation Limit – The minimum level of acceptable quantitation that is supported by the analysis of standards.

Raw Data – The originally recorded and unprocessed measurements from any measuring device such as analytical instruments, balances, pipettes, thermometers, etc. Reported data are processed raw measurement values that may have been reformatted from the original measurement to meet specific reporting requirements such as significant figures and decimal precision.

Relative Percent Difference (RPD) – The absolute of the relative difference between two values normalized to the mean of the two values expressed as a percentage.

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 portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Sampling and Analysis Plan (SAP) – A document which specifies the procedural and analytical requirements for one-time, or time-limited, projects involving the collection of water, soil, sediment, or other samples taken to characterize areas of potential environmental contamination.

Sample Identifier – A unique identification number that appears on the Chain of Custody (COC) Records or sampling forms which documents information for a sample.

Serial Dilution – The dilution of a sample by a factor of five. When corrected by the Dilution Factor (DF), the diluted sample should agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents [Inductively Coupled Plasma (ICP) only].

Soil – Synonymous with soil/sediment and sediment as used herein.

Statement of Work (SOW) – A document which specifies how laboratories analyze samples under a contract, such as the 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 analyzed to serve as an initial demonstration of Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) accuracy, resolution, and precision prior to calibration. May also be called Instrument Performance Check sample (IPC).

Warning Limit - A result for a Performance Evaluation (PE) sample that is outside the 95% ($\pm 2\sigma$) control limits. The laboratory should apply and document corrective actions to bring the analytical results back into control.

APPENDIX B: INORGANIC DATA REVIEW SUMMARY

Event ID/Case No. (if applicable)_____ Site _____

Laboratory _____ No. of Samples/Matrix _____

Modified Analysis No. (if applicable)_____ Data Package ID (if applicable) _____

Reference Method (if applicable)_____ Project/EPA Region (if applicable) _____

Reviewer Name _____ Completion Date _____

Action _____ FYI _____

Validation Label _____

REVIEW CRITERIA	METHOD/ANALYTE			
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. Performance Evaluation Sample				
12. Quality Assurance and Quality Control				
13. Overall Assessment of Data				

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