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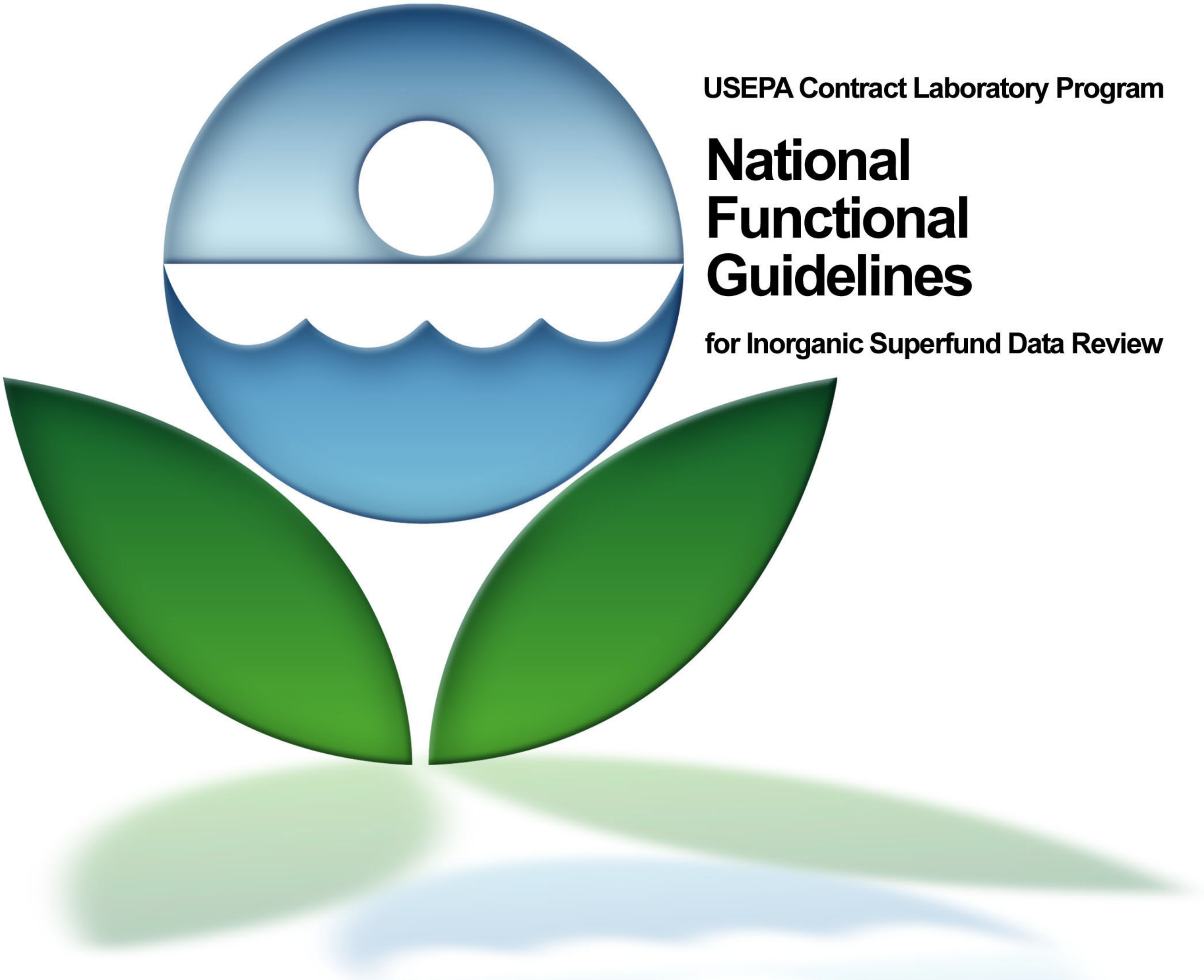

USEPA Contract Laboratory Program

National Functional Guidelines

for Inorganic Superfund Data Review

Final

NOTICE

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

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ACRONYMS

AA	Atomic Absorption
ASB	Analytical Services Branch
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CO	Contracting Officer
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
CVAA	Cold Vapor AA
DAT	Data Assessment Tool
DF	Dilution Factor
DQO	Data Quality Objective
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Sample
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
MDL	Method Detection Limit
NIST	National Institute of Standards and Technology
OSRTI	Office of Superfund Remediation and Technology Innovation
OSWER	Office of Solid Waste and Emergency Response
PE	Performance Evaluation
%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%RSD	Percent Relative Standard Deviation
%S	Percent Solids
PO	Project Officer
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center Coordinator
SDG	Sample Delivery Group
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TAL	Target Analyte List
TR/COC	Traffic Report/Chain of Custody Documentation
USEPA	United States Environmental Protection Agency

TARGET ANALYTE LIST

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

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INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the usability of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) ISM01.X Inorganic Superfund Methods (Multi-Media, Multi-Concentration), hereinafter referred to as the ISM01.2 SOW, and any future editorial revisions of ISM01.2. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process. It should not be used to establish specific contract compliance; use of this document to evaluate data generated under Inorganic SOWs other than ISM01.2 is cautioned. Definitive guidance is provided where performance should be fully under a laboratory's control [e.g., blanks, calibration verification standards, Interference Check Samples (ICSs), Quality Control (QC) audit samples, and instrument performance checks (tuning)], while general guidance is provided for evaluating subjective data that is affected by site conditions.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and QC criteria established in the SOW, and (b) the usability and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for re-sampling. To make judgments at this level requires the reviewer to have a complete understanding of the intended use of the data. The reviewer is strongly encouraged to establish a dialogue with the user prior to and after data review, to discuss usability issues and to answer questions regarding the review. It should also be understood that in all cases, data which do not meet specified criteria are never to be fully acceptable without qualification.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as professional judgment, should also be used to determine the ultimate usability of data, especially in those cases where all data does not meet specific technical criteria. The reviewer should also be aware that minor modifications to some of the analytical methods may be made through the "Request for Quote (RFQ) for Modified Analysis" to meet site-specific requirements, and that these modifications could affect certain validation criteria such as Contract Required Quantitation Limits (CRQLs) and Target Analyte Lists (TALs). A copy of any modification request made to the analytical method should be included in the data package by the laboratory.

Please visit the CLP Web site at http://www.epa.gov/superfund/programs/clp/index.htm for more information on how to obtain service through the CLP.

DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process. If the Regions choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

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

DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the Data Assessment Tool (DAT) report is a useful tool in the data review process. The DAT report incorporates Contract Compliance Screening (CCS) and National Functional evaluation results, and is transmitted electronically via email. For more information about DAT, please refer to the following CLP Web site:

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

The DAT report will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data.

If there are any concerns regarding the data package, contact the CLP Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP Web site:

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

PRELIMINARY REVIEW

This document is for the review of analytical data generated through the ISM01.2 SOW and any future editorial revisions of ISM01.2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) documentation, SDG Narratives, etc.].

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The reviewer should contact the appropriate Regional Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken, and
2. The complete list of samples with information on:
    a. Sample matrix;
    b. Field blanks*;
    c. Field duplicates*;
    d. Field spikes*;
    e. PE samples*;
    f. Shipping dates;
    g. Preservatives;
    h. Types of analysis; and
    i. Laboratories involved.

    * If applicable.

The TR/COC documentation includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

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

DATA REVIEW NARRATIVE

A Data Review Narrative, including the Inorganic Data Review Summary form (see Appendix B), must accompany the laboratory data forwarded to the intended data recipient (client) or user to promote communication. A copy of the Data Review Narrative should be submitted to the CLP PO assigned oversight responsibility for the laboratory producing the data.

The Data Review Narrative should include comments that clearly identify the problems associated with a Case or SDG and state the limitations of the data. Documentation should also include the CLP Sample Number, analytical method, extent of the problem, and assigned qualifiers.

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:

An Example Analytical Sequence for ICP-AES

S0
S
S
S
S
S
ICV
ICB
ICSA
ICSAB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

Preservation and Holding Times

A. Review Items:

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

B. Objective:

The objective is to ascertain the validity of the analytical results based on the sample condition, and the holding time of the sample from the date of collection to the date of analysis.

C. Criteria:

1. The technical holding time criteria for aqueous/water metal samples is 180 days, preserved (with nitric acid) to pH ≤ 2. The addition of nitric acid to adjust the pH is only required for aqueous/water samples.
2. The technical holding time criteria for soil/sediment metal samples is 180 days, based on the technical holding time criteria for aqueous/water samples.
3. The technical holding time criteria for wipe and air filter samples is 180 days, based on the technical holding time criteria for aqueous/water samples.
4. Aqueous/water and soil/sediment samples shall be maintained at 4°C (±2°C) until preparation. Wipe and air filter samples may be maintained at room temperature until preparation.

D. Evaluation:

Technical holding times are established by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form XIII-IN and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on the Form XIIIs and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication that there were problems with the samples, the integrity of the samples may be compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

E. Action:

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

1. If the pH of aqueous/water metal samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH of the sample to ≤ 2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-), and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data, based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-), and qualify non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment, wipe, and filter samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria to soil/sediment, wipe, or filter samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

Table 1. Technical Holding Time Actions for ICP-AES Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water metals samples received with pH > 2 and pH not adjusted	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Aqueous/water and soil/sediment metals samples not maintained at 4°C (±2°C)	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Aqueous/water Metals > 180 day	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Soil/sediment, wipe, filter Metals > 180 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

Calibration

A. Review Items:

Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

C. Criteria:

1. Initial Calibration

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

a. A blank and at least five calibration standards shall be used to establish each analytical curve. At least one of these standards shall be at or below the CRQL. All measurements shall be within the instrument working range where the interelement correction factors are valid. A minimum of three replicate exposures are required for standardization, all Quality Control (QC), and sample analyses. The average result of all the multiple exposures for the standardization, QC, and sample analyses shall be used. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient ≥ 0.995. The percent differences calculated for all of the non-zero standards must be within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

2. Initial and Continuing Calibration Verification (ICV and CCV)

The acceptance criteria for the ICVs and CCVs are presented in Table 2:

Table 2. Acceptance Criteria for ICVs and CCVs,

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

a. Initial Calibration Verification (ICV)

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

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

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

b. Continuing Calibration Verification (CCV)

1) To ensure accuracy during the course of each analytical run, the CCV shall be analyzed and reported for each wavelength used for the analysis of each analyte.
2) The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample.
3) The analyte concentration(s) in the CCV standard(s) shall be different than the concentration used for the ICV, and at a concentration equivalent to the mid level of their respective calibration curves.
4) The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG).
5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation:

1. Verify that the instrument was calibrated each time the instrument was set up, utilizing a blank and at least five calibration standards, one of which was at or below the CRQL.
2. Confirm that the measurements were within the working range, and were the average result of at least three replicate exposures.
3. Verify that the ICV and CCV standards were analyzed for each analyte at the proper frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
4. Recalculate one or more of the ICV and CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form IIA-IN.

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

Where,

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

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

E. Action:

NOTES: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

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

1. If the instrument was not calibrated each time the instrument was set up, qualify the data as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and at least one at or below CRQL), use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
2. If the correlation coefficient is <0.995, percent differences are outside the ±30% limit, or the y-intercept ≥CRQL, qualify sample results that are ≥MDL as estimated (J) and non-detects as estimated (UJ).

3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
    a. If the ICV or CCV %R is < 75%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as estimated low (J-) or unusable (R).
    b. If the ICV or CCV %R falls within the range of 75-89%, qualify sample results that are ≥ MDL as estimated low (J-), and qualify non-detects as estimated (UJ).
    c. If the ICV or CCV %R falls within the range of 111-125%, qualify sample results that are ≥ MDL as estimated high (J+).
    d. If the ICV or CCV %R is within the range of 111-125%, non-detects should not be qualified.
    e. If the ICV or CCV %R is > 125%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+) or unusable (R). Non-detects should not be qualified.
    f. If the %R is > 160%, qualify all results that are ≥ MDL as unusable (R).
4. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
5. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
6. If calibration criteria are grossly exceeded, note this for CLP Project Officer (CLP PO) action.

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

Table 3. Calibration Actions for ICP-AES Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) or unusable (R) Qualify non-detects as estimated (UJ) or unusable (R)
Correlation coefficient <0.995; %D outside ±30%; y-intercept ≥CRQL	Qualify results that are ≥MDL as estimated (J) Qualify non-detects as estimated (UJ)
ICV/CCV %R < 75%	Qualify results that are ≥ MDL as estimated low (J-) or unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 75-89%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 111-125%	Qualify results that are ≥ MDL as estimated high (J+)
ICV/CCV %R > 125%	Qualify results that are ≥ MDL as estimated high (J+) or unusable (R)
ICV/CCV %R > 160%	Qualify results that are ≥ MDL as unusable (R)

Blanks

A. Review Items:

Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective:

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, calibration blanks, field blanks, etc.). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blank(s).
2. The Initial Calibration Blank (ICB) shall be analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument (see Section II.C.1).
3. A Continuing Calibration Blank (CCB) shall be analyzed at each wavelength used for the analysis, immediately after every Continuing Calibration Verification (CCV). The CCB shall be analyzed at a frequency of every two hours during the run. The CCB shall be analyzed at the beginning of the run, and again after the last CCV that was analyzed after the last analytical sample of the run. The CCB result (absolute value) shall not exceed the Contract Required Quantitation Limit (CRQL) of each analyte for which analysis is performed.
4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water or a clean wipe or filter processed through the appropriate sample preparation and analysis procedure.
5. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10 times (10x) the Preparation Blank concentration. Otherwise, all samples associated with that Preparation Blank with the analyte's concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed for that analyte (except for an identified field blank). The laboratory is not to correct the sample concentration for the blank value.
6. If the concentration of the Preparation Blank for a certain analyte is < (-CRQL) all samples reported < 10x the CRQL (associated with that analyte in that blank), should be redigested and reanalyzed.

D. Evaluation:

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the proper frequency and location during the run, and Preparation Blanks were prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Review the results reported on the Blank Summary (Form III-IN), as well as the raw data for all blanks, and verify that the results were accurately reported.
3. Evaluate all of the associated blanks for the presence of target analytes. Verify that if target analytes were present in a Preparation Blank or if a concentration was < (-CRQL) the affected samples were redigested and reanalyzed. Verify that if target analytes were present in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

E. Action:

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

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

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

1. If the appropriate blanks were not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
    a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL) qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
    b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
    a. If the absolute value of an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples were not reanalyzed, report non-detects and results that are ≥ MDL, but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, use professional judgment to qualify the data as unusable (R) or to report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.
    b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to quality results that are > CRQL.
    c. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redigested and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redigestion (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redigest and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

Table 4. Blank Actions for ICP-AES Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
ICB/CCB	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < Blank Result	Report at level of Blank Result with a "U" or qualify data as unusable (R)
		> Blank Result	Use professional judgment
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	≥ MDL, or non-detect	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < 10x the Blank Result	Use professional judgment to qualify results as unusable (R) or estimated high (J+)
		≥ 10x the Blank Result	No action
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

Inductively Coupled Plasma - Interference Check Sample (ICP-ICS)

A. Review Items:

Form IVA-IN, Form XIII-IN, instrument printouts, and raw data.

B. Objective:

The Inductively Coupled Plasma (ICP) - Interference Check Sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples.

NOTE: The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements.

C. Criteria:

1. The 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 Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES).
2. An ICS must be run at the beginning of each sample analysis run. The ICS is <u>not</u> to be run prior to the Initial Calibration Verification (ICV), and is to be immediately followed by a Continuing Calibration Verification (CCV), which will be followed by a Continuing Calibration Blank (CCB).
3. Results for the analysis of ICS Solution A must fall within the control limits of ± Contract Required Quantitation Limit (CRQL), or ± 20% of the true value (whichever is greater) for the analytes and interferents.
4. Results for the analysis of ICS Solution AB must fall within the control limits of ± CRQL, or ± 20% of the true value (whichever is greater) for the analytes and interferents included in the solution.
5. If the value of an ICS result exceeds ± CRQL, or ± 20% of true value (whichever is greater) criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the new calibration then reverified, and the affected samples reanalyzed.
6. The ICS should be obtained from USEPA <u>if available</u>, and analyzed according to the instructions supplied with the solutions. The ICS may be prepared with the interferents at 2x the level specified in the Statement of Work (SOW) if high levels of interferents are found in the field samples. If the ICS is not available from USEPA, an independent ICS solution shall be prepared with the interferent and analyte concentrations at the levels specified in the method.

D. Evaluation:

1. Verify using the raw data (ICP instrument printout) that the ICS was analyzed at the proper frequency and location during the analytical run.
2. Evaluate the ICS raw data for results with an absolute value that is ≥ Method Detection Limit (MDL) for those analytes which are not present in the ICS solution.
3. Recalculate using the raw data and the following equation, one or more of the analyte Percent Recoveries (%R), and verify that the recalculated value agrees with the laboratory-reported values on Form IV-IN.

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

Where,

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

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

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

E. Action:

NOTE: For an ICS that does not meet the technical criteria, apply the action to all samples analyzed in the analytical run.

1. The raw data should, but may not, contain results for interferents. If not, the reviewer shall use professional judgment to qualify the data. If the data does contain results for interferents, the reviewer should apply the following actions to samples with concentrations of interferents that are comparable to, or greater than, their respective levels in the ICS:
   a. If the ICS %R for an analyte or interferent is > 120% (or greater than the true value + CRQL, as applicable) and the sample results are non-detects, the data should not be qualified.
   b. If the ICS %R for an analyte or interferent is > 120% (or greater than the true value + CRQL, as applicable) qualify sample results that are ≥ MDL as estimated high (J+). If the ICS %R (or true value) grossly exceeds the limits, use professional judgment to qualify the data.
   c. If the ICS %R for an analyte or interferent falls within the range of 50-79% (or less than the true value - CRQL, as applicable) qualify sample results that are ≥ MDL as estimated low (J-).
   d. If the ICS recovery for an analyte falls within the range of 50-79% (or less than the true value - CRQL, as applicable), the possibility of false negatives exists. Qualify sample non-detects as estimated (UJ).
   e. If the ICSAB %R for an analyte or interferent is < 50%, qualify all sample results that are ≥ MDL as estimated low (J-) and all sample non-detects as unusable (R).
2. If results that are ≥ MDL are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify sample results that are ≥ MDL as estimated high (J+). Non-detects should not be qualified.
3. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is ≥ MDL, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents, qualify non-detects for the affected analytes as estimated (UJ), and results that are ≥ MDL but < 10x the absolute value of the negative result as estimated low (J-).

4. In general, ICP-AES sample data can be accepted if the concentrations of Al, Ca, Fe, and 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, the reviewer should investigate the possibility of other interference effects as given in the ICP-AES method or as indicated by the laboratory's interelement correction factors reported on Forms XA-IN and XB-IN for that particular instrument. The analyte concentration equivalents presented in the method should be considered only as estimated values since the exact value of any analytical system is instrument-specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is > 2x the CRQL, and also > 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).
5. If the raw data does not contain results for the interferents, note it in the Data Review Narrative.
6. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be extremely complex. Use professional judgment to determine the need for the associated sample data to be qualified. The reviewer may need to obtain additional information from the laboratory. All interpretive situations should then be recorded in the Data Review Narrative.
7. If the ICS acceptance criteria are grossly exceeded, note the specifics for CLP PO action.

Table 5. Interference Check Actions for ICP-AES Analysis

Interference Check Sample Results	Action for Samples
ICS %R > 120% (or greater than true value + CRQL)	Qualify results that are ≥ MDL as estimated high (J+)
ICS %R 50-79% (or less than true value - CRQL)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICSAB %R < 50%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Potential false positives in field samples with interferents	Qualify results that are ≥ MDL as estimated high (J+)
Potential false negatives in field samples with interferents	Qualify results that are ≥ MDL but < 10x(|negative value|) as estimated low (J-) Qualify non-detects as estimated (UJ)

Laboratory Control Sample (LCS)

A. Review Items:

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

B. Objective:

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation.

C. Criteria:

1. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples.
   a. One LCS shall be prepared and analyzed for every group of aqueous/water or soil/sediment samples in a Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The LCS shall be spiked such that the final digestate contains each analyte at two times the CRQL for the associated matrix.
   b. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%, except for Sb and Ag which must fall within the control limits of 50-150%. If the %R for the aqueous/water and soil/sediment LCS falls outside of the control limits, the analysis should be terminated, the problem corrected, and the samples prepared with that LCS redigested and reanalyzed.
   c. One LCS shall be prepared and analyzed for each group of wipe or filter samples in an SDG, or with each batch of wipe or filter samples digested, whichever is more frequent. The wipe or filter LCS shall be spiked such that the final digestate contains each analyte at two times the CRQL for the associated matrix.
   d. All wipe or filter sample LCS %R shall fall within the control limits of 70-130%.

D. Evaluation:

1. Verify using Form VII-IN, Form XII-IN, and raw data that the appropriate number of required LCSs were prepared and analyzed for the SDG.
2. Evaluate Form VII-IN and verify that all results for each analyte fall within the established control limits.
   a. Check the raw data to verify that the %Rs on Form VII-IN were accurately transcribed. Recalculate one or more of the reported %Rs using the following equation:

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

Where,

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

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

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

E. Action:

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

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

1. **Aqueous/Water and Soil/Sediment LCS:**
   a. If the LCS %R falls within the range of 40-69% (20-49% for Ag and Sb), qualify sample results that are ≥ Method Detection Limit (MDL) as estimated low (J-). If the LCS %R is > 130% (150% for Ag and Sb), qualify sample results that are ≥ MDL as estimated high (J+).
   b. If the LCS recovery is > 130% (150% for Ag and Sb) and the sample results are non-detects, the data should not be qualified.
   c. If the LCS recovery falls within the range of 40-69% (20-49% for Ag and Sb), qualify non-detects as estimated (UJ).
   d. If LCS %R is < 40% (<20% for Ag and Sb), qualify all results that are ≥ MDL as estimated low (J-) and all non-detects as unusable (R).
   e. If the LCS %R is > 150% (170% for Ag and Sb), qualify all affected data (both detects and non-detects) as unusable (R).
2. **Wipe/Filter LCS:**
   a. If the LCS %R is in the range of 40-69%, qualify sample results that are ≥ MDL as estimated low (J-) and qualify non-detects as estimated (UJ).
   b. If the LCS %R is < 40%, qualify all results that are ≥ MDL as estimated low (J-) and all non-detects as unusable (R).
   c. If the LCS %R is > 130% and the sample results are non-detects, do not qualify the data.
   d. If the LCS %R is > 130%, qualify all results ≥ MDL as estimated high (J+).
3. If a laboratory fails to analyze an LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries, note this for CLP Project Officer (CLP PO) action.
4. Whenever possible, the potential effects on the data due to out-of-control LCS results should be noted in the Data Review Narrative.

Table 6. LCS Actions for ICP-AES Analysis

LCS Result	Action for Samples
Aqueous/Water and Soil/Sediment %R 40-69% (20-49% Ag, Sb)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
Aqueous/Water and Soil/Sediment %R > 130% (150% Ag, Sb)	Qualify results that are ≥ MDL as estimated high (J+)
Aqueous/Water and Soil/Sediment %R < 40% (<20% Ag, Sb)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Aqueous/Water and Soil/Sediment %R > 150% (>170% Ag, Sb)	Qualify all results as unusable (R)
Wipe/Filter %R 40-69%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
Wipe/Filter %R > 130%	Qualify results that are ≥ MDL as estimated high (J+)
Wipe/Filter %R < 40%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

Duplicate Sample Analysis

A. Review Items:

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

B. Objective:

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analyses are also performed to generate data that determines the long-term precision of the analytical method on various matrices. Non-homogenous samples can impact the apparent method precision. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG). Duplicates are not required for wipe or filter samples. Duplicates cannot be averaged for reporting on Form I-IN. Additional duplicate sample analyses may be required by USEPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).
4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form VI-IN. If both samples are non-detects, the RPD is not calculated for Form VI-IN.

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

D. Evaluation:

1. Verify from the Cover Page, Form XII-IN, and the raw data that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
2. Evaluate Form VI-IN and the raw data to verify that all duplicate results for each analyte and method fall within the established control limits.
3. Verify that a field blank or PE sample was not used for duplicate analysis.
4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form VI-IN:

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

Where,

RPD = Relative Percent Difference

S = Sample Result (original)

D = Duplicate Result

E. Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the reviewer considers the samples to be sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data when determining similarity, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus that only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for a particular analyte fall outside the appropriate control limits, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated (J) and non-detects as estimated (UJ).
3. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
4. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 7. Duplicate Sample Actions for ICP-AES Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD > 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

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

Spike Sample Analysis

A. Review Items:

Cover Page, Form V-IN (Part A & B), Form XII-IN, instrument printouts, and raw data.

B. Objective:

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. Non-homogenous samples can impact the apparent method recovery. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three. If the spike is added to the sample before the digestion (e.g., prior to the addition of other reagents), it is referred to as a spiked sample, pre-digestion spike, or Matrix Spike. If the spike is added to the sample after the completion of the digestion procedures, it is referred to as a post-digestion spike, or analytical spike.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for spiked sample analysis.
2. At least one spiked sample (pre-digestion) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each Sample Delivery Group (SDG). Matrix Spikes are not required for wipe or filter samples.
3. When the Matrix Spike recovery falls outside of the control limits and the sample result is < four times (4x) the spike added, a post-digestion spike shall be performed for those analytes that do not meet the specified criteria. An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the Contract Required Quantitation Limit (CRQL), whichever is greater.

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

4. The spike Percent Recovery (%R) shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the “original sample”. The average of the duplicate results cannot be used for the purpose of determining %R.

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

D. Evaluation:

1. Verify using the Cover Page, Form VA-IN, Form XII-IN, and raw data, that the appropriate number of required spiked samples were prepared and analyzed for the SDG.
2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
3. Evaluate Form VA-IN and the raw data to verify that all pre-digestion spiked sample results for each required analyte fall within the established control limits. If not, verify that a post-digestion/post-distillation spike was prepared and analyzed.
4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms V (A & B)-IN:

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

Where,

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

NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purpose of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

E. Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make 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, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the Matrix Spike, and thus that only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-digestion spike was not performed, note this for CLP PO action.
4. If the Matrix Spike %R is < 30%, verify that a post-digestion spike was analyzed (if required). If the post-digestion spike %R is < 75% or is not performed, qualify sample results that are ≥MDL as estimated low (J-) and non-detects as unusable (R). If the post-digestion spike %R is ≥ 75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
5. If the Matrix Spike %R is 30-74% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed (if required). If the %R for the post-digestion is also < 75% or is not performed, qualify the affected data as estimated low (J-). If the %R for the post-digestion spike is ≥ 75%, qualify the affected data as estimated (J).
6. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
7. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.

8. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed if required. If the %R for the post-digestion spike is also > 125% or is not performed, qualify the affected data as estimated high (J+). If the %R for the post-digestion spike is ≤ 125%, qualify the affected data as estimated (J).
9. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 8. Spike Sample Actions for ICP-AES Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% Post-digestion spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion Spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125% Post-digestion spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+)
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	Qualify affected results that are ≥ MDL as estimated (J)
Matrix Spike %R < 30% No post-digestion spike performed (e.g., not required for Ag)	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74% No post-digestion spike performed (e.g., not required for Ag)	Qualify affected results that are ≥ MDL as estimated low (J-) and non-detects as estimated (UJ)
Matrix Spike %R > 125% No post-digestion spike performed (e.g., not required for Ag)	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

ICP Serial Dilution

A. Review Items:

Form I-IN, Form VIII-IN, instrument printouts, and raw data.

B. Objective:

The serial dilution of samples quantitated by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) determines whether or not significant physical or chemical interferences exist due to sample matrix.

C. Criteria:

1. An ICP Serial Dilution analysis shall be performed on a sample from each group of samples with a similar matrix type (e.g., water, soil, wipe, or filter) or for each Sample Delivery Group (SDG), whichever is more frequent.
2. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for the ICP Serial Dilution analysis.
3. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution shall be less than 10.

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

D. Evaluation:

1. Verify that a field blank or PE sample was not used for the serial dilution analysis.
2. Check the raw data and recalculate the %D using the following equation. Verify that the serial dilution analysis results, and the calculated %D results agree with the values reported by the laboratory on Form VIII-IN:

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

Where,

I = Initial Sample Result
S = Serial Dilution Result

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

E. Action:

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to all samples of the same matrix if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make 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, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the serial dilution sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for serial dilution, and thus that only the field sample used to prepare the serial dilution sample should be qualified.

1. If the required %D criteria are not met, qualify all affected results that are ≥ MDL as estimated (J) and all affected non-detects as estimated (UJ).
2. If evidence of positive or negative interference is found, use professional judgment to qualify the associated sample data. Note the potential effects on the reported data in the Data Review Narrative.
3. It should be noted for Contract Laboratory Program Project Officer (CLP PO) action and in the Data Review Narrative if a field blank or PE sample was used for the serial dilution analysis.

Table 9. Serial Dilution Actions for ICP-AES Analysis

Serial Dilution Result	Action for Samples
Sample concentration > 50x MDL and %D > 10*	Qualify affected results that are ≥ MDL as estimated (J) Qualify affected non-detects as estimated (UJ)
Interferences present	Use professional judgment

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

Regional Quality Assurance (QA) and Quality Control (QC)

A. Review Items:

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

B. Objective:

Regional QA/QC samples refer to any QA and/or QC sample initiated by the Region, including field duplicate samples, Performance Evaluation (PE) samples, blind spikes and blind blanks. The use of these QA/QC samples is highly recommended (e.g., the use of field duplicates can provide information on sampling precision and homogeneity).

C. Criteria:

Criteria are determined by each Region.

D. Evaluation:

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

Calculate the Relative Percent Difference (RPD) between field duplicates and provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

Overall Assessment

A. Review Items:

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

B. Objective:

1. The objective is to ensure that the reported sample quantitation results are accurate. It is appropriate for the data reviewer to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case. This is particularly appropriate when there are several Quality Control (QC) criteria that are outside of the specification parameters. The additive nature of QC factors that fall outside of specification parameters is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user of data quality and data limitations to assist that user to avoid inappropriate use of the data, while not precluding any consideration of the data. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the acceptance or performance criteria were provided. The Inorganic Review Summary (see Appendix B) and supplementary documentation must be included with the review.

C. Criteria:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method.

D. Evaluation:

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

1. Evaluate any technical problems not previously addressed.
2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).
3. Verify that appropriate methods and amounts were used in preparing the samples for analysis. If reduced volumes were used, verify that the laboratory had received Contract Laboratory Program Project Officer (CLP PO) approval for the use of the reduced volume.
4. Verify that there are no transcription or reduction errors [e.g., dilutions, Percent Solids (%S), sample weights, etc.] on one or more samples.
5. Verify that results fall within the calibrated range(s) of the Inductively Coupled Plasma (ICP) instrument(s).
6. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the Standard Operating Procedure(s) (SOPs), and communication with the user concerning the intended use and desired quality of these data.

E. Action:

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

2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the Sample Delivery Group (SDG) Narrative for Contract Laboratory Program Project Officer (CLP PO) action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.
3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

Calculations for ICP-AES

Aqueous/Water Samples by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES):

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

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

Where,

C = Instrument value in µg/L (The average of all replicate exposures.)

V_f = Final digestion volume (mL)

V = Initial aliquot amount (mL)

DF = Dilution Factor

Soil/Sediment Samples by ICP-AES:

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

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

Where,

C = Instrument value in µg/L (The average of all replicate exposures).

V_f = Final digestion volume (mL)

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

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

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

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

Where,

C = MDL or CRQL (mg/kg)

W_M = Minimum method required aliquot amount (g) (1.00 g or 0.50 g)

W = Initial aliquot amount (g)

V_M = Method required final sample digestion volume (mL) (100 mL or 50 mL)

V_f = Final digestion volume (mL)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

Wipe/Filter 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

ICP-MS DATA REVIEW

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

An Example Analytical Sequence for ICP-MS

Tune
S0
S
S
S
S
S
ICV
ICB
ICSA
ICSAB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

I. Preservation and Holding Times

A. Review Items:

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

B. Objective:

The objective is to determine the validity of the analytical results based on the sample condition, and the holding time of the sample from the date of collection to the date of analysis.

C. Criteria:

1. The technical holding time criteria for aqueous/water metal samples is 180 days, preserved (with nitric acid) to pH ≤ 2. The addition of nitric acid to adjust the pH is only required for aqueous/water samples.
2. The technical holding time criteria for soil/sediment metal samples is 180 days, based on the technical holding time criteria for aqueous/water samples.
3. Samples shall be maintained at 4°C (±2°C) until preparation and analysis.

D. Evaluation:

Technical holding times are established by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form XIII-IN, and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on the Form XIIIs and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. Review the SDG Narrative to determine if the aqueous/water samples were received in glass containers. If there is an indication that there were problems with the samples, the integrity of the samples may be compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

E. Action:

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

1. If the pH of aqueous/water metals samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH to ≤2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-), and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-), and qualify non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply 180 day holding time criteria to soil/sediment samples. If it is applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note this for Contract Laboratory Program Project Officer (CLP PO) action.

Table 10. Technical Holding Time Actions for ICP-MS Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water metals samples received with pH > 2 and pH not adjusted	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Metals samples not maintained at 4°C (±2°C)	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Aqueous/water Metals > 180 day	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Soil/sediment Metals > 180 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

ICP-MS Tune Analysis

A. Review Items:

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

B. Objective:

The Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) tune serves as an initial demonstration of instrument stability and precision.

C. Criteria:

1. Prior to calibration, the laboratory shall analyze or scan the ICP-MS tuning solution at least five times (5x) consecutively. The tuning solution contains 100 µg/L of Be, Mg, Co, In, and Pb. The solution shall contain all required isotopes of the above elements. The laboratory shall make any adjustments necessary to bring peak width within the instrument manufacturer's specifications and adjust mass resolution 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 must be < 5%.

D. Evaluation:

1. Verify using the raw data and Form XIV-IN that the appropriate number of analyses or scans of the ICP-MS tuning solution were performed, and that the appropriate analytes were present in the solution.
2. Verify using the raw data and Form XIV-IN that the mass calibration falls within the limits for each isotope of each analyte.
3. Verify using the raw data and Form XIV-IN that the %RSD is < 5% for each isotope of each analyte.
4. Check the raw data to verify that the reported average mass and %RSD on Form XIV-IN was accurately calculated. Recalculate one or more of the average masses and %RSDs for an isotope using the following equations:

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

Where,

x = Mass from analysis

n = Number of analyses

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

Where,

$\bar{x}$ = Mean

σ_{n-1} = Standard Deviation

E. Action:

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

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

2. If the tuning solution was not analyzed or scanned at least 5x consecutively or the tuning solution does not contain the required analytes spanning the analytical range, the reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be recorded in the Data Review Narrative and noted for Contract Laboratory Program Project Officer (CLP PO) action.
3. If the resolution of the mass calibration is not within 0.1 u for any isotope in the tuning solution, qualify all analyte results that are ≥ Method Detection Limit (MDL) associated with that isotope as estimated (J), and all non-detects associated with that isotope as estimated (UJ). The situation should be recorded in the Data Review Narrative and noted for CLP PO action.
4. If the %RSD exceeds 5% for any isotope in the tuning solution, qualify all sample results that are ≥ MDL associated with that tune as estimated (J), and all non-detects associated with that tune as estimated (UJ). The situation should be recorded in the Data Review Narrative and noted for CLP PO action.

Table 11. ICP-MS Tune Actions for ICP-MS Analysis

ICP-MS Tune Results	Action for Samples
Tune not performed	Qualify all results as unusable (R)
Tune not performed properly	Use professional judgment
Resolution of mass calibration not within 0.1 u	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
%RSD > 5%	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)

Calibration

A. Review Items:

Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continual basis.

C. Criteria:

1. Initial Calibration

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

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

2. Initial and Continuing Calibration Verification (ICV and CCV)

The acceptance criteria for the ICVs and CCVs are presented in Table 12:

Table 12. Acceptance Criteria for ICV and CCV Standards

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

a. Initial Calibration Verification (ICV)

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

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

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

b. **Continuing Calibration Verification (CCV)**

   1) To ensure accuracy during the course of each analytical run, the CCV shall be analyzed and reported for each mass used for the analysis of each analyte.
   2) The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample.
   3) The analyte concentration(s) in the CCV standard(s) shall be different than the concentration used for the ICV, and at a concentration equivalent to the mid level of their respective calibration curves.
   4) The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG).
   5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant calibration verification reanalyzed.

D. Evaluation:

1. Verify that the instrument was calibrated each time the instrument was set up, utilizing a blank and at least five calibration standards, one of which was at or below the CRQL.
2. Confirm that the measurements were within the working range, and were the average result of at least three replicate exposures.
3. Verify that the ICV and CCV standards were analyzed for each analyte at the proper frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
4. Recalculate one or more of the ICV and CCV %Rs using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form IIA-IN.

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

Where,

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

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

E. Action:

NOTE: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

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

1. If the instrument was not calibrated each time the instrument was set up, qualify the data as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and at least one at or below CRQL), use professional judgment to qualify results that are ≥ MDL as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
2. If the correlation coefficient is <0.995, percent differences are outside the ±30% limit, or the y-intercept ≥CRQL, qualify sample results that are ≥MDL as estimated (J) and non-detects as estimated (UJ).

3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
    a. If the ICV or CCV %R is < 75%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as estimated low (J-) or unusable (R).
    b. If the ICV or CCV %R falls within the range of 75-89%, qualify sample results that are ≥MDL as estimated low (J-), and qualify non-detects as estimated (UJ).
    c. If the ICV or CCV %R falls within the range of 111-125%, qualify sample results that are ≥ MDL as estimated high (J+).
    d. If the ICV or CCV %R falls within the range of 111-125%, non-detects should not be qualified.
    e. If the ICV or CCV %R is > 125%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+) or unusable (R). Non-detects should not be qualified.
    f. If the %R is > 160%, qualify all results that are ≥ MDL as unusable (R).
4. If the laboratory failed to provide adequate calibration information, the USEPA Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
5. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
6. If calibration criteria are grossly exceeded, note this for Contract Laboratory Program Project Officer (CLP PO) action.

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

Table 13. Calibration Actions for ICP-MS Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) or unusable (R) Qualify non-detects as estimated (UJ) or unusable (R)
Correlation coefficient <0.995; %D outside ±30%; y-intercept ≥CRQL	Qualify results that are ≥MDL as estimated (J) Qualify non-detects as estimated (UJ)
ICV/CCV %R < 75%	Qualify results that are ≥ MDL as estimated low (J-) or unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 75-89%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 111-125%	Qualify results that are ≥ MDL as estimated (J)
ICV/CCV %R > 125%	Qualify results that are ≥ MDL as estimated high (J+) or unusable (R)
ICV/CCV %R > 160%	Qualify results that are ≥ MDL as unusable (R)

Blanks

A. Review Items:

Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective:

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, calibration blanks, field blanks, etc.). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blank(s).
2. The Initial Calibration Blank (ICB) shall be analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument (see Section III.C.1).
3. A Continuing Calibration Blank (CCB) shall be analyzed at each mass used for the analysis, immediately after every Continuing Calibration Verification (CCV). The CCB shall be analyzed at a frequency of every two hours during the run. The CCB shall be analyzed at the beginning of the run, and again after the last CCV that was analyzed after the last analytical sample of the run. The CCB result (absolute value) shall not exceed the Contract Required Quantitation Limit (CRQL) of each analyte for which analysis is performed.
4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
5. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10 times (10x) the Preparation Blank concentration. Otherwise, all samples associated with that Preparation Blank with the analyte's concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed for that analyte (except for an identified field blank). The laboratory is not to correct the sample concentration for the blank value.
6. If the concentration of the Preparation Blank for a certain analyte is < (-CRQL), all samples reported < 10x the CRQL (associated with that analyte in that blank), should be redigested and reanalyzed.

D. Evaluation:

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the proper frequency and location during the run, and Preparation Blanks were prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Review the results reported on the Blank Summary (Form III-IN), as well as the raw data for all blanks, and verify that the results were accurately reported.
3. Evaluate all of the associated blanks for the presence of target analytes. Verify that if target analytes were present in a Preparation Blank or if a concentration was < (-CRQL), the affected samples were redigested and reanalyzed. Verify that if target analytes were present in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

E. Action:

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

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

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

1. If the appropriate blanks were not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
    a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL), qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
    b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
    a. If the absolute value of an ICB or a CCB result > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples were not reanalyzed, report non-detect and results that are ≥ MDL but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, use professional judgment to qualify the data as unusable or to report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.
    b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to qualify results that are > CRQL.
    c. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redigested and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redigestion (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redigest and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

Table 14. Blank Actions for ICP-MS Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥MDL but ≤ CRQL	Non-detect	No action
		≥MDL but ≤CRQL	Report CRQL value with a “U”
		> CRQL	Use professional judgment
ICB/CCB	> CRQL	≥MDL but ≤CRQL	Report CRQL value with a “U”
		> CRQL but < Blank Result	Report at level of Blank Result with a “U” or qualify data as unusable (R)
		> Blank Result	Use professional judgment
ICB/CCB	≤(-MDL), but ≥(-CRQL)	≥MDL, or non-detect	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x CRQL	Qualify results that are ≥CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥MDL but ≤CRQL	Report CRQL value with a “U”
		> CRQL but < 10x the Blank Result	Qualify results as unusable (R) or estimated high (J+)
		≥10x the Blank Result	No action
Preparation Blank	≥MDL but ≤ CRQL	Non-detect	No action
		≥MDL but ≤CRQL	Report CRQL value with a “U”
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x CRQL	Qualify results that are ≥CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

Inductively Coupled Plasma - Interference Check Sample (ICP-ICS)

A. Review Items:

Form IVA-IN, Form IVB-IN, Form XIII-IN, instrument printouts, and raw data.

B. Objective:

The Inductively Coupled Plasma-Interference Check Sample (ICP-ICS) verifies the analytical instrument's ability to overcome isobaric interferences typical of those found in samples.

C. Criteria:

1. The 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 Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).
2. An ICS must be run at the beginning of each analysis run. The ICS is not to be run prior to the Initial Calibration Verification (ICV), and shall be immediately followed by a Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB).
3. Results for the ICP-MS analysis of the ICS Solution A shall fall within the control limits of ± 2x the CRQL, or ± 20% of the true value (whichever is greater) for the analytes included in the solution.
4. Results for the ICP-MS analysis of the ICS Solution AB must fall within the control limits of ± 2x the CRQL, or ± 20% of the true value (whichever is greater) for the analytes included in the solution.
5. If the value of an ICS result exceeds ± 2x the CRQL, or ± 20% of true value (whichever is greater) criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the new calibration then reverified, and all analytical samples analyzed since the last compliant ICS reanalyzed.
6. The ICS should be obtained from USEPA, if available, and analyzed according to the instructions supplied with the solutions. If the ICS is not available from USEPA, an independent ICS solution shall be prepared with the interferent and analyte concentrations at the levels specified in the method.

D. Evaluation:

1. Verify using the raw data (ICP instrumental printout) that the ICS was analyzed at the proper frequency and location during the analytical run.
2. Evaluate the ICS raw data for results with an absolute value that is ≥ Method Detection Limit (MDL) for those analytes that are not present in the ICS solution.
3. Recalculate using the raw data and the following equation, one or more of the analyte Percent Recoveries (%R), and verify that the recalculated value agrees with the laboratory-reported values on Form IV-IN.

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

Where,

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

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

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

E. Action:

NOTE: For an ICS for ICP-MS that does not meet the technical criteria, apply the action to all samples reported from the analytical run.

1. The raw data may not contain results for interferents. In this case, the reviewer shall use professional judgment to qualify the data. If the data does contain results for interferents, the reviewer should apply the following actions to samples with concentrations of interferents that are comparable to, or greater than, their respective levels in the ICS:
    a. the ICS %R for an analyte is > 120% (or greater than the true value + 2x the CRQL as applicable) and the sample results are non-detects, the data should not be qualified.
    b. If the ICS %R for an analyte is > 120% (or greater than the true value + 2x the CRQL as applicable) qualify sample results that are ≥ MDL as estimated high (J+). If the ICS %R (or true value) grossly exceeds the limits, use professional judgment to qualify the data.
    c. If the ICS %R for an analyte falls within the range of 50-79% (or less than the true value - 2x the CRQL as applicable) qualify sample results that are ≥ MDL as estimated low (J-).
    d. If the ICS recovery for an analyte falls within the range of 50-79% (or less than the true value - 2x the CRQL as applicable), the possibility of false negatives exists. Qualify sample non-detects as estimated (UJ).
    e. If the ICSAB %R for an analyte or interferent is < 50%, qualify all sample results that are ≥ MDL and all sample non-detects as unusable (R).
2. If results that are ≥ MDL are observed for analytes which are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify sample results that are ≥ MDL as estimated high (J+). Non-detects should not be qualified.
3. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is ≥ MDL, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents, qualify non-detects for the affected analytes as estimated (UJ), and results that are ≥ MDL but < 10x the absolute value of the negative result as estimated low (J-).
4. If the raw data does not contain results for the interferents, note this in the Data Review Narrative.
5. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be extremely complex. Use professional judgment to determine the need for the associated sample data to be qualified. The reviewer may need to obtain additional information from the laboratory. All interpretive situations should then be recorded in the Data Review Narrative.
6. If the ICS acceptance criteria are grossly exceeded, note the specifics for CLP PO action.

Table 15. Interference Check Actions for ICP-MS Analysis

Interference Check Sample Results	Action for Samples
ICS %R > 120% (or > true value + 2x the CRQL)	Qualify results that are ≥ MDL as estimated high (J+)
ICS %R 50-79% (or < true value - 2x the CRQL)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICSAB %R < 50%	Qualify all sample data as unusable (R)
Potential false positives in field samples with interferents	Qualify results that are ≥ MDL as estimated high (J+)
Potential false negatives in field samples with interferents	Qualify results that are ≥ MDL but < 10x(|negative value|) as estimated low (J-) Qualify non-detects as estimated (UJ)

Laboratory Control Sample (LCS)

A. Review Items:

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

B. Objective:

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation.

C. Criteria:

1. LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples.
   a. LCS shall be prepared and analyzed for every group of aqueous/water or soil/sediment samples in a Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The aqueous/water and soil/sediment LCS shall be spiked such that the final digestate contains each analyte at two times the CRQL for the associated matrix.
   b. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%. If the %R for the LCS falls outside of the control limits, the analysis should be terminated, the problem corrected, and the samples prepared with that LCS redigested and reanalyzed.

D. Evaluation:

1. Verify using Form VII-IN, Form XII-IN, and raw data that the appropriate number of required LCSs were prepared and analyzed for the SDG.
   a. Check the LCSs documentation from the supplier and verify that the LCSs identification and the control limits for it are listed on the Form VII-IN match those of the specific LCSs used for the analysis.
   b. Evaluate Form VII-IN and verify that all results for each analyte fall within the established control limits.
2. Check the raw data to verify that the %Rs on Form VII-IN were accurately transcribed. Recalculate one or more of the reported %Rs using the following equation:

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

Where,

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

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

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

E. Action:

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

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

1. **LCS:**
    a. If the LCS %R falls within the range of 40-69%, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated low (J-). If the LCS %R is > 130%, qualify sample results that are ≥ MDL as estimated high (J+).
    b. If the LCS recovery is > 130% and the sample results are non-detects, the data should not be qualified.
    c. If the LCS recovery falls within the range of 40-69%, qualify non-detects as estimated (UJ).
    d. If LCS %R is < 40%, qualify all results that are ≥ MDL as estimated low (J-) and all non-detects as unusable (R).
    e. If the LCS %R is > 150%, qualify all affected data (both detects and non-detects) as unusable (R).
2. If a laboratory fails to analyze an LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries, note this for Contract Laboratory Program Project Officer (CLP PO) action
3. Whenever possible, the potential effects on the data due to out-of-control LCS results should be noted in the Data Review Narrative.

Table 16. LCS Actions for ICP-MS Analysis

LCS Result	Action for Samples
Aqueous/Water and Soil/Sediment %R 40-69%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
Aqueous/Water and Soil/Sediment %R > 130%	Qualify results that are ≥ MDL as estimated high (J+)
Aqueous/Water and Soil/Sediment %R < 40%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Aqueous/Water and Soil/Sediment %R > 150%	Qualify all results as unusable (R)

Duplicate Sample Analysis

A. Review Items:

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

B. Objective:

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analyses are also performed to generate data that determines the long-term precision of the analytical method on various matrices. Non-homogenous samples can impact the apparent method precision. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG). Duplicates cannot be averaged for reporting on Form I-IN. Additional duplicate sample analyses may be required by USEPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).
4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form VI-IN. If both samples are non-detects, the RPD is not calculated for Form VI-IN.

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

D. Evaluation:

1. Verify from the Cover Page, Form XII-IN, and the raw data that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
2. Evaluate Form VI-IN and the raw data to verify that all duplicate results for each analyte and method fall within the established control limits.
3. Verify that a field blank or PE sample was not used for duplicate analysis.
4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form VI-IN:

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

Where,

RPD = Relative Percent Difference

S = Sample Result (original)

D = Duplicate Result

E. Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples to be sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, when determining similarity, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples were not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for a particular analyte fall outside the appropriate control limits, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
3. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
4. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 17. Duplicate Sample Actions for ICP-MS Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD> 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

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

Spike Sample Analysis

A. Review Items:

Cover Page, Form V-IN (Parts A & B), Form XII-IN, instrument printouts, and raw data.

B. Objective:

The spike sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. Non-homogenous samples can impact the apparent method recovery. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three. If the spike is added to the sample before the digestion (e.g., prior to the addition of other reagents), it is referred to as a spiked sample, pre-digestion spike, or Matrix Spike. If the spike is added to the sample after the completion of the digestion procedures, it is referred to as a post-digestion spike, or analytical spike.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for spiked sample analysis.
2. At least one spiked sample (pre-digestion) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each Sample Delivery Group (SDG).
3. When the matrix spike recovery falls outside of the control limits and the sample result is < four times (4x) the spike added, a post-digestion spike shall be performed for those analytes that do not meet the specified criteria. An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the Contract Required Quantitation Limit (CRQL), whichever is greater.
4. The spike Percent Recovery (%R) shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining %R.

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

D. Evaluation:

1. Verify using the Cover Page, Form VA-IN, Form XII-IN, and raw data that the appropriate number of required spiked samples were prepared and analyzed for the SDG.
2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
3. Evaluate Form VA-IN and the raw data to verify that all pre-digestion spiked sample results for each required analyte fall within the established control limits. If not, verify that a post-digestion spike was prepared and analyzed.
4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms V (A & B)-IN:

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

Where,

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

NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purposes of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

E. Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make 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, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the Matrix Spike, and thus that only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-digestion spike was not performed, note this for CLP PO action.
4. If the Matrix Spike %R is < 30%, verify that a post-digestion spike was analyzed if required. If the post-digestion spike %R is < 75% or is not performed, qualify sample results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R). If the post-digestion spike %R is ≥ 75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
5. If the Matrix Spike %R is 30-74% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed, if required. If the %R for the post-digestion spike is also < 75% or is not performed, qualify the affected data as estimated low (J-). If the %R for the post-digestion spike is ≥ 75%, qualify the affected data as estimated (J).
6. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
7. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.

8. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed, if required. If the %R for the post-digestion spike is also > 125% or is not performed, qualify the affected data as estimated high (J+). If the %R for the post-digestion spike is ≤ 125%, qualify the affected data as estimated (J).
9. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 18. Spike Sample Actions for ICP-MS Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% Post-digestion spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) affected non-detects as estimated (UJ)
Matrix Spike %R > 125% Post-digestion spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+)
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	Qualify affected results that are ≥ MDL as estimated (J)
Matrix Spike %R < 30% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

ICP Serial Dilution

A. Review Items:

Form I-IN, Form VIII-IN, instrument printouts, and raw data.

B. Objective:

The serial dilution of samples quantitated by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) determines whether or not significant physical or chemical interferences exist due to sample matrix.

C. Criteria:

1. An ICP Serial Dilution analysis shall be performed on a sample from each group of samples with a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG), whichever is more frequent.
2. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for the ICP serial dilution analysis.
3. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution analysis (a five-fold dilution) after correction shall be less than 10.

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

D. Evaluation:

1. Verify that a field blank or PE sample was not used for the serial dilution analysis.
2. Check the raw data and recalculate the %D using the following equation. Verify that the serial dilution analysis results, and the calculated %D results agree with the values reported by the laboratory on Form VIII-IN:

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

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

E. Action:

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to all samples of the same matrix if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make 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, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the serial dilution sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for serial dilution, and thus only the field sample used to prepare the serial dilution sample should be qualified.

1. If the required %D criteria are not met, qualify all affected results that are ≥ MDL as estimated (J) and all affected non-detects as estimated (UJ).
2. If evidence of positive or negative interference is found, use professional judgment to qualify the associated sample data. Note the potential effects on the reported data in the Data Review Narrative.
3. It should be noted for CLP Project Officer (CLP PO) action and in the Data Review Narrative if a field blank or PE sample was used for the serial dilution analysis.

Table 19. Serial Dilution Actions for ICP-MS Analysis

Serial Dilution Result	Action for Samples
Sample concentration > 50x MDL and %D > 10*	Qualify affected results that are ≥ MDL as estimated (J) Qualify affected non-detects as estimated (UJ)
Interferences present	Use professional judgment

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

ICP-MS Internal Standards

A. Review Items:

Form XIII-IN, Form XV-IN, Form XVII-IN, instrument printouts, and raw data.

B. Objective:

The analysis of Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) internal standards determines the existence and magnitude of instrument drift and physical interferences. The criteria for evaluation of internal standard results apply to all analytical and Quality Control (QC) samples analyzed during the run, beginning with the calibration.

C. Criteria:

1. All samples analyzed during a run, with the exception of the ICP-MS tune, shall contain internal standards. A minimum of five internal standards from the following list shall be added to each sample: Li (the Li^6 isotope); Sc; Y; Rh; Tb; Ho; Lu; and Bi. If the laboratory uses lithium as an internal standard, the laboratory shall use an Li^6-enriched standard. The laboratory shall monitor the same internal standards throughout the entire analytical run and shall assign each analyte to at least one internal standard.
2. The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in the sample shall fall within 60-125% of the response in the calibration blank.
3. If the %RI of the response in the sample falls outside of these limits, the laboratory shall reanalyze the original sample at a two-fold dilution with internal standard added.

D. Evaluation:

1. Verify using Forms XV-IN, XVII-IN, and the raw data that a minimum of five internal standards from the specified list were used for the analysis, that the same internal standards were monitored for the entire run, and that each analyte was associated to at least one internal standard.
2. Verify using Form XV-IN and the raw data that these internal standards were added to each sample in the run, including calibrations, samples, and QC samples (except tune).
3. Verify using Form XV-IN that the %RI between an internal standard in a sample and the internal standard in the calibration blank was reported for each sample.
4. Verify using Form XIII-IN, Form XV-IN, and the raw data that if the %RI for a sample was outside the limits (60-125%), the sample was reanalyzed of a 2x dilution with internal standard added.

E. Action:

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

1. If no internal standards were analyzed with the run, the sample data should be qualified as unusable (R). Record this in the Data Review Narrative and note for CLP Project Officer (CLP PO) action.
2. If less than five of the required internal standards were analyzed with the run, or a target analyte(s) is (are) not associated to an internal standard, the sample data, or analyte data not associated to an internal standard should be qualified as unusable (R). Record this in the Data Review Narrative and note for CLP PO action.
3. If the %RIs for all internal standards in a sample is within the 60-125% limit, the sample data should not be qualified.
4. If the %RI for an internal standard in a sample is not within the 60-125% limit, qualify the data for those analytes associated with the internal standard(s) outside the limit as follows:

a. If the sample was reanalyzed at a two-fold dilution with internal standard %RI within the limits, report the result of the diluted analysis without qualification. If the %RI of the diluted analysis was not within the 60-125% limit, report the results of the original undiluted analyses and qualify the data for all analytes that are ≥ Method Detection Limit (MDL) in the sample associated with the internal standard as estimated (J), and non-detected analytes associated with the internal standard as estimated (UJ).

b. If the sample was not reanalyzed at a two-fold dilution, the reviewer should use professional judgment to determine the reliability of the data. The reviewer may determine that the results are estimated (J) or unusable (R).

Table 20. Internal Standard Actions for ICP-MS Analysis

Internal Standard Results	Action for Samples
No internal standards	Qualify all results as unusable (R)
< 5 of the required internal standards	Qualify all analyte results not as unusable (R)
Target analyte not associated with internal standard	Qualify all analyte results not associated with internal standard as unusable (R)
%RI < 60% or > 125%, and original sample reanalyzed at 2-fold dilution	If %RI of diluted sample analysis 60-125%, do not qualify the data If the %RI of the diluted sample analysis is outside the 60-125% limit, qualify results that are ≥ MDL as estimated (J) and qualify non-detects as estimated (UJ)
Original sample not reanalyzed at 2-fold dilution	Use professional judgment Qualify sample results as estimated (J) or unusable (R)

Regional Quality Assurance (QA) and Quality Control (QC)

A. Review Items:

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

B. Objective:

Regional QA/QC samples refer to any QA and/or QC samples initiated by the Region, including field duplicate samples, Performance Evaluation (PE) samples, blind spikes, and blind blanks. The use of these QA/QC samples is highly recommended (e.g., the use of field duplicate samples can provide information on sampling precision and sample homogeneity).

C. Criteria:

Criteria are determined by each Region.

D. Evaluation:

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

Calculate the Relative Percent Difference (RPD) between field duplicates and provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

Overall Assessment

A. Review Items:

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

B. Objective:

The objective is to ensure that the reported sample quantitation results are accurate. It is appropriate for the data reviewer to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case. This is particularly appropriate when there are several Quality Control (QC) criteria that are outside of the specification parameters. The additive nature of QC factors that fall outside of specification parameters is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user of data quality and data limitations to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the acceptance or performance criteria were provided. The Inorganic Review Summary (see Appendix B) and supplementary documentation must be included with the review.

C. Criteria:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method.

D. Evaluation:

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

1. Evaluate any technical problems not previously addressed.
2. Examine the raw data for any anomalies (e.g., baseline shifts, negative response, mass dependent drift, omissions, illegibility, etc.).
3. Verify appropriate methods and volumes were used in preparing the samples for analysis. If reduced volumes were used, verify that the laboratory had received Contract Laboratory Program Project Officer (CLP PO) approval for the use of the reduced volume.
4. Verify that there are no transcription or reduction errors [e.g., dilutions, Percent Solids (%S), sample weights, etc.] on one or more samples.
5. Verify that results fall within the calibrated range(s) of the Inductively Coupled Plasma (ICP) instrument(s) (Form XI).
6. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the Standard Operating Procedure(s) (SOPs), and communication with the user concerning the intended use and desired quality of these data.

E. Action:

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

2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the SDG Narrative for CLP PO action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.
3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

Calculations for ICP-MS

Aqueous/Water Sample Concentration by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS):

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

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

Where,

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

Soil/Sediment Sample Concentration by ICP-MS:

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

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

Where,

C = Instrument value in µg/L (The average of all replicate integrations).
V_f = Final digestion volume (mL)
W = Initial aliquot amount (g)
S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
DF = Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

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

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

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

Where,

C = MDL or CRQL (mg/kg)
W_M = Minimum method required aliquot amount (g) (1.00g or 0.50g)
W = Initial aliquot amount (g)
V_M = Method required final sample digestion volume (mL) (100 mL)
V_f = Final digestion volume (mL)
S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
DF = Dilution Factor

MERCURY DATA REVIEW

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

An Example Analytical Sequence for Mercury

S0
S0.2
S1.0
S2.0
S5.0
S10.0
ICV
ICB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

I. Preservation and Holding Times

A. Review Items:

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

B. Objective:

The objective is to ascertain the validity of the analytical results based on the sample condition, and the holding time of the sample from the date of collection to the date of analysis.

C. Criteria:

1. The technical holding time criteria for aqueous/water mercury samples is 28 days; preserved (with nitric acid) to pH ≤ 2. The addition of nitric acid to adjust the pH is only required for aqueous/water samples.
2. The technical holding time criteria for soil/sediment mercury samples is 28 days, based on the technical holding time criteria for aqueous/water samples.
3. Mercury samples shall be maintained at 4°C (±2°C) until preparation and analysis to allow for re-preparation.
4. Samples and standards shall be analyzed with 48 hours of preparation.

D. Evaluation:

Technical holding times are established by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form XIII-IN, and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on the Form XIIIs and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication that there were problems with the samples, the integrity of the samples may be compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

E. Action:

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

1. If the pH of aqueous/water metals samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH to ≤2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-), and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-), and qualify non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria to soil/sediment samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

6. When shipping or storage temperatures grossly exceed the requirements, the loss of volatile mercury compounds or metallic mercury is possible. The expected bias would be low. Use professional judgment to qualify the samples and note for CLP PO action.

Table 21. Technical Holding Time Actions for Mercury Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water metals samples received with pH > 2 and pH not adjusted	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Samples not maintained at 4°C (±2°C)	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding time exceeded: Aqueous/water Samples > 28 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Soil/sediment Metals > 28 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

Calibration

A. Review Items:

Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for mercury. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

C. Criteria:

1. Initial Calibration

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

a. Cold Vapor Mercury Analysis

1) A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curves for mercury shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must fall within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL. All sample results shall be reported from an analysis within the calibrated range.

2. Initial and Continuing Calibration Verification (ICV and CCV)

The acceptance criteria for the ICVs and CCVs are presented in Table 22. These standards shall be prepared by the same method used to prepare the samples for analysis.

Table 22. Acceptance Criteria for ICVs and CCVs

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

a. Initial Calibration Verification (ICV)

1) Immediately after each Atomic Absorption (AA) system has been calibrated, the accuracy of the initial calibration must be verified and documented for mercury by the analysis of an ICV solution(s). If the ICV %R falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.
2) If the ICV is not available from USEPA, or where a certified solution of the analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration level other than that used for instrument calibration, but within the calibrated range.

b. Continuing Calibration Verification (CCV)

1) To ensure accuracy during the course of each analytical run, the CCV shall be analyzed and reported.
2) The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample.
3) The analyte concentration in the CCV standard shall be different than the concentration used for the ICV, and shall be at the mid level of the calibration curve.
4) The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG).
5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation:

1. Verify that the instrument was calibrated daily (once every 24 hours) and each time the instrument was set up, utilizing a blank and at least five calibration standards. Confirm that at least one of the calibration standards was analyzed at or below the CRQL. Confirm that calibration standards and samples were prepared at the same time.
2. Confirm that the measurements were within the documented working range of the calibration standards.
3. Verify that the ICV and CCV standards were analyzed for mercury at the proper frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
4. Recalculate one or more of the ICV or CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form IIA-IN.

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

Where,

Found (value) = Concentration (in µg/L) of mercury <u>measured</u> in the analysis of the ICV or CCV solution

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

E. Action:

NOTES: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

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

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

2. If the correlation coefficient is < 0.995, percent differences are outside the ±30% limit, or the y-intercept is >CRQL, qualify sample results that are ≥ MDL as estimated (J), and non-detects as estimated (UJ). Depending on the degree of the deviation from linearity, further qualification of the data may be required depending on the professional judgment of the reviewer [e.g., unusable data (R)].
3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
   a. If the ICV or CCV %R is < 70%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as estimated low (J-) or unusable (R).
   b. If the ICV or CCV %R falls within the range of 70-84%, qualify sample results that are ≥ MDL as estimated low (J-) and qualify non-detects as estimated (UJ).
   c. If the ICV or CCV %R falls within the range of 116-130%, qualify sample results that are ≥ MDL as estimated high (J+).
   d. If the ICV or CCV %R falls within the range of 116-130%, non-detects should not be qualified.
   e. If the ICV or CCV %R is > 130%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+) or unusable (R). Non-detects should not be qualified.
   f. If the %R is > 165%, qualify all results that are ≥ MDL as unusable (R).
4. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
5. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
6. If calibration criteria are grossly exceeded, note this for CLP Project Officer (CLP PO) action.

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

Table 23. Calibration Actions for Mercury Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) or unusable (R) Qualify non-detects as estimated (UJ) or unusable (R)
Correlation coefficient <0.995, %D outside ±30%, y-intercept ≥CRQL	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
ICV/CCV %R < 70%	Qualify results that are ≥ MDL as estimated low (J-) or unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 70-84%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 116-130%	Qualify results that are ≥ MDL as estimated (J)
ICV/CCV %R > 130%	Qualify results that are ≥ MDL as estimated high (J+) or unusable (R)
ICV/CCV %R > 165%	Qualify results that are ≥ MDL as unusable (R)

Blanks

A. Review Items:

Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective:

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, calibration blanks, field blanks, etc.). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blank(s).
2. The Initial Calibration Blank (ICB) shall be analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument (see Section II.C.1). The ICB shall be prepared by the same method used to prepare the samples for analysis.
3. A Continuing Calibration Blank (CCB) shall be analyzed immediately after every Continuing Calibration Verification (CCV). The CCB shall be prepared by the same method used to prepare the samples for analysis. The CCB shall be analyzed at a frequency of every hour during the run. The CCB shall be analyzed at the beginning of the run, and again after the last CCV that was analyzed after the last analytical sample of the run. The CCB result (absolute value) shall not exceed the Contract Required Quantitation Limit (CRQL) for mercury.
4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
5. If the mercury concentration in the Preparation Blank is > CRQL, the lowest concentration of mercury in the associated samples must be ≥ 10 times (10x) the Preparation Blank concentration. Otherwise, all samples associated with that Preparation Blank with a mercury concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed (except for an identified field blank). The laboratory is not to correct the sample concentration for the blank value.
6. If the concentration of the Preparation Blank for mercury is < (-CRQL), all samples reported < 10x the CRQL (associated with that analyte in that blank), should be redigested and reanalyzed.

D. Evaluation:

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the proper frequency and location during the run, and Preparation Blanks are prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Review the results reported on the Blank Summary (Form III-IN), as well as the raw data for all blanks, and verify that the results are accurately reported.
3. Evaluate all of the associated blanks for the presence of mercury. Verify that if mercury was present in a Preparation Blank or if a concentration was < (-CRQL), the affected samples were redigested and reanalyzed. Verify that if mercury was present in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

E. Action:

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

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

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

1. If the appropriate blanks are not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
   a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL), qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
   b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
   a. If the absolute value of an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples are not reanalyzed, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, use professional judgment to qualify the data as unusable (R), or to report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.
   b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, report non-detect and results that are ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to qualify results that are > CRQL.
   c. If the mercury concentration in the Preparation Blank is > CRQL, the lowest concentration of mercury in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redigested and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redigestion (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redigest and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

Table 24. Blank Actions for Mercury Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	Absolute value is ≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
ICB/CCB	Absolute value is > CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < Blank Result	Report at level of Blank Result with a "U" or qualify data as unusable (R)
		> Blank Result	Use professional judgment
ICB/CCB	≤ (-MDL), but ≥ (-CRQL)	≥ MDL, or non-detect	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < 10x the Blank Result	Qualify results as unusable (R) or estimated high (J+)
		≥ 10x the Blank Result	No action
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL with a "U"
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

Duplicate Sample Analysis

A. Review Items:

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

B. Objective:

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analyses are also performed to generate data that determines the long-term precision of the analytical method on various matrices. Non-homogenous samples can impact the apparent method precision. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG). Duplicates cannot be averaged for reporting on Form I-IN. Additional duplicate sample analyses may be required by USEPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).
4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the “Control Limit” column on Form VI-IN. If both samples are non-detects, the RPD is not calculated for Form VI-IN.

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

D. Evaluation:

1. Verify from the Cover Page, Form XII-IN, and the raw data that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
2. Evaluate Form VI-IN and the raw data to verify that all mercury duplicate results for each method fall within the established control limits.
3. Verify that a field blank or PE sample was not used for duplicate analysis.
4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form VI-IN:

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

Where,

RPD = Relative Percent Difference

S = Sample Result (original)

D = Duplicate Result

E. Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make 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, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus that only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for mercury fall outside the appropriate control limits, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated (J) and non-detects as estimated (UJ).
3. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
4. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 25. Duplicate Sample Actions for Mercury Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD> 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

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

Spike Sample Analysis

A. Review Items:

Cover Page, Form V-IN (Parts A & B), Form XII-IN, instrument printouts, and raw data.

B. Objective:

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. Non-homogenous samples can impact the apparent method recovery. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three. If the spike is added to the sample before the digestion (e.g., prior to the addition of other reagents), it is referred to as a spiked sample, pre-digestion spike, or Matrix Spike.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for spiked sample analysis.
2. At least one spiked sample (pre-digestion) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each Sample Delivery Group (SDG).
3. The spike Percent Recovery (%R) shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ four times (4x) the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
4. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining %R.

NOTE: The final spike concentrations required for mercury are presented in the method described in the Statement of Work (SOW).

D. Evaluation:

1. Verify using the Cover Page, Form VA-IN, Form XII-IN, and raw data that the appropriate number of required spiked samples were prepared and analyzed for the SDG.
2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
3. Evaluate Form VA-IN and the raw data to verify that all Matrix Spike sample results for mercury fall within the established control limits.
4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms V (A & B)-IN:

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

Where,

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

NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purposes of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

E. Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make 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, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the Matrix Spike, and thus that only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike %R is < 30%, qualify affected results that are ≥ MDL as estimated low (J-). Qualify affected non-detects as unusable (R).
4. If the Matrix Spike %R falls within the range of 30-74% and the sample results are ≥ MDL, qualify the affected data as estimated low (J-).
5. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
6. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.
7. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, qualify the affected data as estimated high (J+).
8. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 26. Spike Sample Actions for Mercury Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

Regional Quality Assurance (QA) and Quality Control (QC)

A. Review Items:

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

B. Objective:

Regional QA/QC samples refer to any QA and/or QC sample initiated by the Region, including field duplicates, Performance Evaluation (PE) samples, blind spikes, and blind blanks. The use of these QA/QC samples is highly recommended (e.g., the use of field duplicates can provide information on sampling precision and homogeneity).

C. Criteria:

Criteria are determined by the Region.

D. Evaluation:

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

Calculate the Relative Percent Difference (RPD) between field duplicates and provide his information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

Overall Assessment

A. Review Items:

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

B. Objective:

The objective is to ensure that the reported sample quantitation results are accurate. It is appropriate for the data reviewer to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case. This is particularly appropriate when there are several Quality Control (QC) criteria that are outside of the specification parameters. The additive nature of QC factors that fall outside of specification parameters is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the acceptance or performance criteria are provided. The Inorganic Review Summary (see Appendix B) and supplementary documentation must be included with the review.

C. Criteria:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method.

D. Evaluation:

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

1. Evaluate any technical problems not previously addressed.
2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).
3. Verify that the appropriate methods and amounts were used to prepare samples and standards for analysis. If reduced volumes are used, verify that the laboratory had received Contract Laboratory Program Project Officer (CLP PO) approval for the use of the reduced volume.
4. Verify that there are no transcription or reduction errors [e.g., dilutions, Percent Solids (%S), sample weights, etc.] on one or more samples.
5. Verify that results fall within the calibrated range for mercury.
6. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the Standard Operating Procedure(s) (SOPs), and communication with the user concerning the intended use and desired quality of these data.

E. Action:

1. Use professional judgment to determine if there is any need to qualify data which are not qualified based on the QC criteria previously discussed.
2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the SDG Narrative for CLP PO action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.

3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

Calculations for Mercury

Aqueous/Water Samples:

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

Where,

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

DF = Dilution Factor of the original sample

Soil/Sediment Samples:

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

Where,

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

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, multiply the value of the MDL (µg/L) or CRQL (µg/L) by the Dilution Factor (DF). Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

$$\text{Adjusted Concentration (mg/kg)} = \text{C} \times \frac{\text{W}_\text{m}}{\text{W x S}} \times \text{DF}$$

Where,

C = MDL or CRQL (mg/kg)

W_m = Method required minimum sample weight (g) (0.50 g)

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

CYANIDE DATA REVIEW

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

An Example Analytical Sequence for Cyanide

S0
S10
S50
S100
S200
S400
ICV
ICB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

I. Preservation and Holding Times

A. Review Items:

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

B. Objective:

The objective is to ascertain the validity of the analytical results based on the sample condition, and the holding time of the sample from the date of collection to the date of analysis.

C. Criteria:

1. The technical holding time criteria for aqueous/water cyanide samples is 14 days; oxidizing agents removed, then preserved (with sodium hydroxide) to pH ≥ 12. The addition of sodium hydroxide to adjust the pH is only required for aqueous/water samples.
2. The technical holding time criteria for soil/sediment cyanide samples is 14 days, based on the technical holding time criteria for aqueous/water samples.
3. Samples shall be maintained at 4°C (±2°C) until preparation and analysis.

D. Evaluation:

Technical holding times are established by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form XIII-IN, and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on the Form XIIIs and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication that there are problems with the samples, the integrity of the samples may be compromised and professional judgment should be used to evaluate the effect of the problem on the sample results. For aqueous/water cyanide samples, the reviewer should look for evidence that the samples were tested for the presence of sulfides or oxidizing agents, and whether the appropriate preservation steps were taken.

E. Action:

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

1. If oxidizing agents are detected in aqueous/water cyanide samples at the time of sample preparation, qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-) and non-detects as unusable (R). If sulfides are detected in aqueous/water cyanide samples at the time of sample preparation and there is no evidence that the laboratory removed the sulfides (using precipitation and filtration), qualify results that are ≥ MDL as estimated (J) and non-detects as unusable (R). If the pH of aqueous/water cyanide samples is < 12 at the time of sample receipt, use professional judgment to qualify the samples based on the pH of the sample. Qualify results that are ≥ MDL as estimated low (J-) and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples are properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria to soil/sediment samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.

5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

Table 27. Technical Holding Time Actions for Cyanide Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water cyanide samples received with oxidizing agents present.	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Aqueous/water cyanide samples received with sulfides present, and sulfides are not removed	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as unusable (R)
Aqueous/water cyanide samples received with pH < 12	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical holding time exceeded: Cyanide > 14 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

Calibration

A. Review Items:

Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for cyanide. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

C. Criteria:

1. Initial Calibration

   The instruments shall be successfully calibrated daily (or once every 24 hours), and each time the instrument is set up. The calibration date and time shall be included in the raw data.

   a. A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curve for cyanide shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must be within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

   b. All sample results shall be reported from an analysis within the calibrated range.

   c. All standards shall be distilled.

2. Initial and Continuing Calibration Verification (ICV and CCV)

   The acceptance criteria for the ICVs and CCVs are presented in Table 28:

Table 28. Acceptance Criteria for ICVs and CCVs

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

   a. Initial Calibration Verification (ICV)

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

      2) If the ICV is not available from USEPA, or where a certified solution of the analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration level other than that used for instrument calibration, but within the calibrated range.

      3) For cyanide analysis, the ICV standard solution shall be distilled.

b. Continuing Calibration Verification (CCV)
   1) To ensure accuracy during the course of each analytical run, the CCV shall be analyzed and reported.
   2) The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample.
   3) The analyte concentration in the CCV standard shall be different from the concentration used for the ICV, and shall be at the mid level of the calibration curve.
   4) The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG). The CCV shall be distilled.
   5) The CCV shall be processed and analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation:

1. Verify that the instrument was calibrated daily (once every 24 hours) and each time the instrument was set up, utilizing a blank and at least five calibration standards. Confirm that at least one of the calibration standards was analyzed at or below the CRQL.
2. Check the distillation log and verify that the calibration standards, the ICV and the CCVs were distilled and analyzed.
3. Verify that the ICV and CCV standards were analyzed for cyanide at the proper frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
4. Recalculate one or more of the ICV or CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form IIA -IN.

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

Where,

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

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

E. Action:

NOTES: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

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

1. If the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and a standard at or below the CRQL), use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
2. If the correlation coefficient is < 0.995, percent differences outside the ±30% limit, or y-intercept ≥CRQL, qualify sample results that are ≥ MDL as estimated (J), and non-detects as estimated (UJ).

3. If the standards, the ICV, or the CCVs are not distilled for cyanide, qualify sample results that are ≥ MDL as estimated (J).
4. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
   a. If the ICV or CCV %R is < 70%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as estimated low (J-) or unusable (R).
   b. If the ICV or CCV %R falls within the range of 70-84%, qualify sample results that are ≥ MDL as estimated low (J-), qualify non-detects as estimated (UJ).
   c. If the ICV or CCV %R falls within the range of 116-130%, qualify sample results that are ≥ MDL as estimated high (J+).
   d. If the ICV or CCV %R is within the range of 116-130%, non-detects should not be qualified.
   e. If the ICV or CCV %R is > 130%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+) or unusable (R). Non-detects should not be qualified.
   f. If the %R is > 165%, qualify all results that are ≥ MDL as unusable (R).
5. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
6. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
7. If calibration criteria are grossly exceeded, note this for Contract Laboratory Program Project Officer (CLP PO) action.

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

Table 29. Calibration Actions for Cyanide Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) or unusable (R) Qualify non-detects as estimated (UJ) or unusable (R)
Correlation coefficient < 0.995; %D outside ±30%, y-intercept ≥CRQL	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
Standards and QC not distilled	Qualify results that are ≥ MDL as estimated (J)
ICV/CCV %R < 70%	Qualify results that are ≥ MDL as estimated low (J-) or unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 70-84%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 116-130%	Qualify results that are ≥ MDL as estimated (J)
ICV/CCV %R > 130%	Qualify results that are ≥ MDL as estimated high (J+) or unusable (R)
ICV/CCV %R > 165%	Qualify results that are ≥ MDL as unusable (R)

Blanks

A. Review Items:

Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective:

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, calibration blanks, field blanks, etc.). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blank(s).
2. The Initial Calibration Blank (ICB) shall be analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument (see Section II.C.1).
3. A Continuing Calibration Blank (CCB) shall be analyzed immediately after every Continuing Calibration Verification (CCV). The CCB shall be analyzed at a frequency of every hour during the run. The CCB shall be analyzed at the beginning of the run, and again after the last CCV that was analyzed after the last analytical sample of the run. The CCB result (absolute value) shall not exceed the Contract Required Quantitation Limit (CRQL) of cyanide.
4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every Sample Delivery Group (SDG), or with each batch of samples distilled, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
5. If the cyanide concentration in the Preparation Blank is > CRQL, the lowest concentration of cyanide in the associated samples must be ≥ 10 times (10x) the Preparation Blank concentration. Otherwise, all samples associated with that Preparation Blank with a cyanide concentration < 10x the Preparation Blank concentration, and > CRQL, should be redistilled and reanalyzed cyanide (except for an identified field blank). The laboratory is not to correct the sample concentration for the blank value.
6. If the concentration of the Preparation Blank for cyanide is < (-CRQL), all samples reported < 10x the CRQL (associated with that blank), should be redistilled and reanalyzed.

D. Evaluation:

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the proper frequency and location during the run, and Preparation Blanks are prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Review the results reported on the Blank Summary (Form III-IN), as well as the raw data for all blanks, and verify that the results were accurately reported.
3. Evaluate all of the associated blanks for the presence of cyanide. Verify that if cyanide was present in a Preparation Blank or if a concentration was < (-CRQL), the affected samples were redistilled and reanalyzed. Verify that if cyanide was present in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

E. Action:

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

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

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

1. If the appropriate blanks are not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
   a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL) qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
   b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the <u>calibration</u> blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
   a. If the absolute value of an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples are not reanalyzed, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, use professional judgment to qualify the data as unusable (R), or report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.
   b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, no correction of the sample results should be performed, report non-detects and results ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to qualify results that are > CRQL.
   c. If the cyanide concentration in the Preparation Blank is > CRQL, the lowest concentration of cyanide in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redistilled and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redistillation (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redistill and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

Table 30. Blank Actions for Cyanide Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	Absolute value is ≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a ”U”
		> CRQL	Use professional judgment
ICB/CCB	Absolute value is > CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < Blank Result	Report at level of Blank Result with a "U" or qualify data as unusable (R)
		> Blank Result	Use professional judgment
ICB/CCB	≤ (-MDL), but ≥ (-CRQL)	≥ MDL, or non-detects	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < 10x the Blank Result	Qualify results as unusable (R) or estimated high (J+)
		≥ 10x the Blank Result	No action
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

Duplicate Sample Analysis

A. Review Items:

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

B. Objective:

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analyses are also performed to generate data that determines the long-term precision of the analytical method on various matrices. Non-homogenous samples can impact the apparent method precision. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG). Duplicates cannot be averaged for reporting on Form I-IN. Additional duplicate sample analyses may be required by USEPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).
4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the “Control Limit” column on Form VI-IN. If both samples are non-detects, the RPD is not calculated for Form VI-IN.

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

D. Evaluation:

1. Verify from the Cover Page, Form XII-IN, and the raw data that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
2. Evaluate Form VI-IN and the raw data to verify that all cyanide duplicate results for each method fall within the established control limits.
3. Verify that a field blank or PE sample was not used for duplicate analysis.
4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form VI-IN:

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

Where,

RPD = Relative Percent Difference

S = Sample result (original)

D = Duplicate result

E. Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make 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, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus that only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for cyanide fall outside the appropriate control limits, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated (J) and non-detects as estimated (UJ).
3. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
4. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 31. Duplicate Sample Actions for Cyanide Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD> 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

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

Spike Sample Analysis

A. Review Items:

Cover Page, Form V-IN (Parts A & B), Form XII-IN, instrument printouts, and raw data.

B. Objective:

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. Non-homogenous samples can impact the apparent method recovery. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three. If the spike is added to the sample prior to any distillation steps (e.g., cyanide), it is referred to as a spiked sample, pre-distillation spike, or Matrix Spike. If the spike is added to the sample after the completion of the distillation procedures, it is referred to as a post-distillation spike, or analytical spike.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for spiked sample analysis.
2. At least one spiked sample (pre-distillation) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each Sample Delivery Group (SDG).
3. When the pre-distillation spike recovery falls outside of the control limits and the sample result is < four times (4x) the spike added, a post-distillation spike shall be performed An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the Contract Required Quantitation Limit (CRQL), whichever is greater.
4. The spike Percent Recovery (%R) shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining %R.

NOTE: The final spike concentrations required for cyanide are presented in the method described in the Statement of Work (SOW).

D. Evaluation:

1. Verify using the Cover Page, Form VA-IN, Form XII-IN, and raw data that the appropriate number of required spiked samples were prepared and analyzed for the SDG.
2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
3. Evaluate Form VA-IN and the raw data to verify that all pre-distillation spiked sample results fall within the established control limits. If not, verify that a post-distillation spike was prepared and analyzed.
4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms V (A & B)-IN:

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

Where,

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

NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purposes of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Form V (A & B)-IN.

E. Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make 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, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the Matrix Spike, and thus that only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-distillation spike was not performed, note this for CLP PO action.
4. If the Matrix Spike %R is < 30%, verify that a post-distillation spike was analyzed if required. If the post-distillation spike %R is < 75% or is not performed, qualify sample results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R). If the post-distillation spike %R is ≥ 75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
5. If the Matrix Spike %R falls within the range of 30-74% and the sample results are ≥ MDL, verify that a post-distillation spike was analyzed if required. If the %R for the post-distillation spike is also < 75% or not performed, qualify the affected data as estimated low (J-). If the %R for the post-distillation spike is ≥ 75%, qualify the affected data as estimated (J).
6. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
7. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.
8. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, verify that a post-distillation spike was analyzed if required. If the %R for the post-distillation spike is also > 125% or is not performed, qualify the affected data as estimated high (J+). If the %R for the post-distillation spike is ≤ 125%, qualify the affected data as estimated (J).
9. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 32. Spike Sample Actions for Cyanide Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% Post-distillation spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) Qualify affected non-detects as unusable (R)
Matrix Spike %R < 30% Post-distillation spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) Qualify affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-distillation spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) Qualify affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-distillation spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) Qualify affected non-detects as estimated (UJ)
Matrix Spike %R > 125% Post-distillation spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+)
Matrix Spike %R > 125% Post-distillation spike %R ≤ 125%	Qualify affected results that are ≥ MDL as estimated (J)
Matrix Spike %R < 30% No post-distillation spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74% No post-distillation spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125% No post-distillation spike performed	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

Regional Quality Assurance (QA) and Quality Control (QC)

A. Review Items:

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

B. Objective:

Regional QA/QC samples refer to any QA and/or QC sample initiated by the Region, including field duplicate samples, Performance Evaluation (PE) sample, blind spikes, and blind blanks. The use of these QA/QC sample sis highly recommended (e.g., the use of field duplicates can provide information on sampling precision and homogeneity).

C. Criteria:

Criteria are determined by each Region.

D. Evaluation:

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

Calculate the Relative Percent Difference (RPD) between field duplicates and provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

Overall Assessment

A. Review Items:

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

B. Objective:

The objective is to ensure that the reported sample quantitation results are accurate. It is appropriate for the data reviewer to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case. This is particularly appropriate when there are several Quality Control (QC) criteria that are outside of the specification parameters. The additive nature of QC factors that fall outside of specification parameters is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the acceptance or performance criteria were provided. The Inorganic Review Summary (see Appendix B) and supplementary documentation must be included with the review.

C. Criteria:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method.

D. Evaluation:

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

1. Evaluate any technical problems not previously addressed.
2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).
3. Verify that the appropriate methods and amounts were used to prepare samples for analysis. If reduced volumes were used, verify that the laboratory had received Contract Laboratory Program Project Officer (CLP PO) approval for the use of the reduced volume.
4. Verify that there were no transcription or reduction errors [e.g., dilutions, Percent Solids (%S), sample weights, etc.] on one or more samples.
5. Verify that results fall within the calibrated range for cyanide.
6. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the Standard Operating Procedure(s) (SOPs), and communication with user concerning the intended use and desired quality of these data.

E. Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.
2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the SDG Narrative for CLP PO action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.

3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

Calculations for Cyanide

Aqueous/Water Sample Concentration:

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

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

Soil/Sediment Sample Concentration:

$$\text{CN Concentration (mg/kg)} = C \times \frac{V_f}{W \times S} \times (1/1000) \times \text{DF}$$

Where,

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 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
DF = Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

To calculate the adjusted aqueous/water MDL or adjusted aqueous/water CRQL, follow the instructions in Exhibit D - Data Analysis and Calculations, Section 11.1.1.

The adjusted soil/sediment MDL or adjusted soil/sediment CRQL for all methods shall be calculated as follows:

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

Where,

C = MDL or CRQL (mg/kg)
W_M = Minimum method required aliquot amount (1.00 g for Midi or 0.50 g for Micro)
W = Initial aliquot amount (g)
S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).
DF = Dilution Factor

APPENDIX A: GLOSSARY

Analyte -- The element of interest, ion, or parameter an analysis seeks to determine.

Analytical Services Branch (ASB) -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).

Analytical Sample -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICSs); Laboratory Control Samples (LCSs); and Preparation Blanks.

Associated Samples -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.

Blank -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

Calibration -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

Calibration Blank -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.

Calibration Curve -- A plot of instrument response versus concentration of standards.

Calibration Standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

Case -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

Continuing Calibration Blank (CCB) -- A reagent water sample that is run 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN) and designed to detect any carryover contamination.

Contract Compliance Screening (CCS) -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.

Continuing Calibration Verification (CCV) -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples. The CCV should have a concentration in the middle of the calibration range and shall be run every 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN).

Contract Laboratory Program (CLP) -- Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.

Contract Laboratory Program Project Officer (CLP PO) -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.

Contract Required Quantitation Limit (CRQL) -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

Duplicate -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

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

Holding Time -- The maximum amount of time samples may be held before they are processed.

Contractual -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

Technical -- The maximum amount of time that samples may be held from the collection date until analysis.

Initial Calibration -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Initial Calibration Blank (ICB) -- The first blank standard run to confirm the calibration curve.

Initial Calibration Verification (ICV) -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.

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.

Interference Check Sample (ICS) -- Verifies the contract laboratory's ability to overcome interferences typical of those found in samples.

Laboratory Control Sample (LCS) -- A control sample spiked at known level(s). LCSs are processed using the same sample preparation, reagents, and analytical methods employed for the USEPA samples received.

Matrix -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.

Matrix Spike -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified as a pre-distillation/digestion spike).

Method Detection Limit (MDL) -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

Narrative (SDG Narrative) -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Office of Solid Waste and Emergency Response (OSWER) – The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.

Percent Difference (%D) -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.

Performance Evaluation (PE) Sample -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.

Post Digestion 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.

Relative Percent Difference (RPD) -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

Regional Sample Control Center Coordinator (RSCC) -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.

Relative Standard Deviation (RSD) -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.

Sample -- A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.

Sample Delivery Group (SDG) -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
b. Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG. Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.

Sample Management Office (SMO) -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

Serial Dilution -- The dilution of a sample by a factor of five. When corrected by the Dilution Factor (DF), the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents [Inductively Coupled Plasma (ICP) only].

Statement of Work (SOW) -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

Tune -- Analysis of a solution containing a range of isotope masses to establish Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) mass-scale accuracy, mass resolution, and precision prior to calibration.

APPENDIX B:
INORGANIC DATA REVIEW SUMMARY

CASE NO. ______________________________ SITE ____

LABORATORY__________________________ NO. OF SAMPLES/MATRIX ___________________

MOD. REF. NO. _________________________ SDG NO. ____________________________________

SOW NO. _______________________________ REGION ____________________________________

REVIEWER NAME _____________________ COMPLETION DATE ________________________

CLP PO: ACTION _______________________ FYI ____

REVIEW CRITERIA **METHOD/ANALYTE**

	ICP-AES	ICP-MS	Mercury	Cyanide
1. Preservation/Holding Time	________	________	________	________
2. Calibration	________	________	________	________
3. Blanks	________	________	________	________
4. Interference Check Sample	________	________		
5. Laboratory Control Sample	________	________		
6. Duplicate Sample Analysis	________	________	_________	________
7. Spike Sample Analysis	________	________	_________	________
8. ICP Serial Dilution	________	________		
9. ICP-MS Tune Analysis	________			
10. ICP-MS Internal Standards	________			
11. Field Duplicates	________	________	_________	________
12. Overall Assessment	________	________	_________	________