Hazardous Waste Support Section
SOP N0. HW-3b Revision 1
ISM02.2
ICP-MS Data Validation

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NOTICE

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

This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

http://www.epa.gov/region2/qa/documents.htm

TABLE OF CONTENTS

LIST OF TABLES

ACRONYMS

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
DF	Dilution Factor
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
ESAT	Environmental Services Assistance Team
EXES	Electronic Data eXchange and Evaluation System
HWSS	Hazardous Waste Support Section
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
%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
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
Fe Iron
Pb Lead
Mg Magnesium
Mn Manganese
Ni Nickel
K Potassium
Se Selenium
Ag Silver
Na Sodium
Tl Thallium
V Vanadium
Zn Zinc

INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) ISM02.X Inorganic Superfund Methods (Multi-Media, Multi-Concentration), hereinafter referred to as the ISM02.2 SOW, and any future editorial revisions of ISM02.2. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process.

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 validity 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.

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 validity of data, especially in those cases where all data does not meet specific technical criteria.

DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process.

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 EXES Data Manager (EDM) is a useful tool in the data review process. For more information about EDM, please refer to the following Sample Management Office (SMO) website:

https://epasmoweb.fedcsc.com/help/guides/Submit%20and%20Inspect%20Data%20Quick%20Guide%20%28EXES%29.pdf

EDM 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 Contracting Officer Representative (CLP COR) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP website:

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

HWSS DATA VALIDATION PROCESS

After downloading the data package from EDM, the data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

A. case number i.e., 12345
B. SDG name i.e., MBXY12
C. level of validation performed i.e., S2BVE

Examples: **12345_MBXY12_S2BVE.xls**

12345_MBXY12_S2BVEM.xls

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website.

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for a list of the column headers included in this document).

PRELIMINARY REVIEW

This document is for the review of analytical data generated through the ISM02.2 SOW and any future editorial revisions of ISM02.2. 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 CLP COR 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.

An Example Analytical Sequence for ICP-MS

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

Preservation and Holding Times

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 samples, it is left to the discretion of the data reviewer whether to apply 180 day 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 1. 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)
Technical Holding Time exceeded: Aqueous/water Metals > 180 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 > 180 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

ICP-MS Tune Analysis

Action:

NOTES: 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 2. 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 0.1u	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

Table 3. 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

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.

NOTE: The data validator shall verify the correlation coefficient by calculating it using the standard concentrations and the corresponding instrument response.

1. If the instrument was not calibrated each time the instrument was set up and an ICV standard was not analyzed before field and QC samples, qualify the data as unusable (R). If the instrument was not calibrated with a blank and at least 5 calibration standards, use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J), and non-detects as estimated (UJ). 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), and non-detects as estimated (UJ). If there was not at least one calibration standard at or below the CRQL for each analyte, qualify results that are ≥ MDL but < 2x the CRQL as estimated (J), and non-detects as estimated (UJ) and write it in the Contract Problem/Non-Compliance Section of the Data Review Narrative.
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). If the correlation coefficient is < 0.990, qualify results that are ≥ MDL as estimated (J) and non-detects as unusable (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 < 75%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as 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+). 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 critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 4. Calibration Actions for ICP-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) Qualify non-detects as estimated (UJ)
Not at least one calibration standard at or below the CRQL for each analyte	Qualify results that are ≥ MDL but < 2x the CRQL as estimated (J) Qualify non-detects as estimated (UJ)
Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
Correlation coefficient < 0.990	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as unusable (R)
ICV/CCV %R < 75%	Qualify results that are ≥ MDL as 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+)
ICV/CCV %R > 160%	Qualify results that are ≥ MDL as unusable (R)

Calibration/Preparation Blanks

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.

NOTES: Convert soil sample result to mg/kg on wet weight basis to compare with the soil preparation result on Form III.
Associated samples are all samples digested with the preparation blank.

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, 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 5. Calibration/Preparation 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"
		> 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 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)

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.

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.

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. If the ICS %R for an analyte or interferent 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 or interferent 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 or interferent 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 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. If the raw data does not contain results for the interferents, note it 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 6. Interference Check Actions for ICP-MS Analysis

Interference Check Sample Results	Action for Samples
ICS not analyzed	Qualify detects and non-detects as unusable (R)
ICS not analyzed in proper sequence	Use professional judgment.
ICS %R>150%	Use professional judgment
ICS %R > 120% (or greater than true value + 2x the CRQL)	Qualify results that are ≥ MDL as estimated high (J+)
ICS %R 80-120%	No qualification
ICS %R 50-79% (or less than true value – 2x the CRQL)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICSAB %R < 50%	Qualify detects as estimated low (J-) and 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 the (|negative value|) as estimated low (J-) Qualify non-detects as estimated (UJ)

Laboratory Control Sample (LCS)

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 CLP 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 7. LCS Actions for ICP-MS Analysis

LCS Result	Action for Samples
%R 40-69%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
%R > 130%	Qualify results that are ≥ MDL as estimated high (J+)
%R 70-130%	No qualification
%R < 40%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
%R > 150%	Qualify detects as unusable (R) ; non-detects no qualification

Laboratory Duplicate Sample Analysis

Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to only the field sample used to prepare the duplicate sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

NOTE: Delete "*" from Form IAs.
If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL and use this difference to qualify sample results.
If more than one lab duplicate sample was analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

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 control limits for > 5x the CRQL, qualify aqueous sample results that are ≥ CRQL as estimated (J) if the RPD is between 20% - 100% and as unusable (R) if the RPD is > 100%. Qualify soil/sediment sample results that are ≥ CRQL as estimated (J) if the RPD is between 35% - 120% and as unusable (R) if the RPD is > 120%.
3. If the results from a duplicate analysis for a particular analyte fall outside the control limits for ≤ 5x the CRQL, qualify those results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
4. 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.
5. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 8. Duplicate Sample Actions for ICP-MS Analysis

Duplicate Sample Results	Action for Samples
Aqueous: Both original sample and duplicate sample > 5x the CRQL and 20% < RPD < 100%	Qualify those results that are ≥ CRQL as estimated (J)
Aqueous: Both original sample and duplicate sample > 5x the CRQL and RPD ≥ 100%	Qualify those results that are ≥ CRQL as unusable (R)
Soil/Sediment: Both original sample and duplicate sample > 5x the CRQL and 35% < RPD < 120%	Qualify those results that are ≥ CRQL as estimated (J)
Soil/Sediment: Both original sample and duplicate sample > 5x the CRQL and RPD ≥ 120%	Qualify those results that are ≥ CRQL as unusable (R)
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 as estimated (J) and non-detects as estimated (UJ)

Spike Sample Analysis

Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

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

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.

NOTES: Not required for Ca, Mg, K, and Na (both matrices); Al and Fe (soil only).
If more than one spiked sample was analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.
Disregard the out of control spike recoveries for analytes whose unspiked concentrations are ≥ 4x the spike added.
Delete "N" from Form IAs.

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 9. 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) 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	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 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

Action:

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to only the field sample used to prepare the serial dilution sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

NOTE: Serial dilution analysis is required only when the initial concentration is greater than 50x the MDL.
Delete "E" from Form IAs.

1. If the required %D criteria are not met, qualify affected aqueous results whose raw data are > MDL as estimated (J) if %D is between 10%-100% and qualify as unusable (R) if %D is ≥ 100%. Qualify affected soil/sediment results whose raw data are > MDL as estimated (J) if %D is between 15%-120% and qualify as unusable (R) if %D is ≥ 120%.
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 10. Serial Dilution Actions for ICP-MS Analysis

Serial Dilution Result	Action for Samples
Aqueous: Sample concentration > 50x MDL and 10% < %D < 100%	Qualify affected results whose raw data are > MDL as estimated (J)
Aqueous: Sample concentration > 50x MDL and %D ≥ 100%	Qualify affected results whose raw data are > MDL as unusable (R)
Soil/Sediment: Sample concentration > 50x MDL and 15% < %D < 120%	Qualify affected results whose raw data are > MDL as estimated (J)
Soil/Sediment: Sample concentration > 50x MDL and %D ≥ 120%	Qualify affected results whose raw data are > MDL as unusable (R)
Interferences present	Use professional judgment

ICP-MS Internal Standards

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 % Relative Intensities for all internal standards in a sample is within 60-125% of the response in the calibration blank, 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 11. 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 results as unusable (R)
Target analyte not associated with internal standard	Qualify all analyte results not associated with an internal standard as unusable (R)
60% < %RI< 125%, original sample reanalyzed at 2-fold dilution, and % RI of diluted sample analysis is between 60% and 125%	Do not qualify the data
% RI < 60% or > 125%, original sample reanalyzed at 2-fold dilution, and % RI of diluted sample analysis is outside the 60% to 125% limit	Qualify analytes associated with the failed internal standard that are ≥ MDL as estimated (J) and qualify associated 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)

Field Duplicates

Action:

NOTES: For field duplicates that do not meet the technical criteria, apply the action to only the field sample and its duplicate. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.
Check the Sampling Trip Report for the field duplicate pair.
Substitute MDL for CRQL when MDL > CRQL.
Do not calculate RPD when both values are non-detects.
If one value is > the CRQL and the other value is non-detect, calculate the absolute difference between the value > the CRQL and the MDL, and use this criteria to qualify the results.

1. If a field duplicate pair was collected and analyzed, calculate and report the RPD when the sample and its field duplicate values are both ≥ 5x the CRQL. Calculate and report the absolute difference when at least one value (sample or duplicate) is < 5x the CRQL.
2. When aqueous sample and duplicate values are both ≥ 5x the CRQL, and the RPD is > 20%, qualify the sample and its duplicate as estimated (J).
3. When aqueous sample and/or the duplicate value is < 5x the CRQL, and the absolute difference is > the CRQL, qualify results > the MDL as estimated (J) and non-detects as estimated (UJ).
4. When soil/sediment sample and duplicate values are both ≥ 5x the CRQL, and the RPD is > 50%, qualify the sample and its duplicate as estimated (J).
5. When soil/sediment sample and/or the duplicate value is < 5x the CRQL, and the absolute difference is > 2x the CRQL, qualify results > the MDL as estimated (J) and non-detects as estimated (UJ).

Table 12. Field Duplicate Actions for ICP-MS Analysis

Sample Type	Field Duplicate Result	Action for Samples
Aqueous	Sample and its field duplicate ≥ 5x the CRQL and RPD > 20%	Qualify sample and its duplicate as estimated (J)
	Sample and/or its field duplicate < 5x the CRQL and absolute difference > the CRQL	Qualify results > the MDL as estimated (J) Qualify non-detects as estimated (UJ)
Soil/Sediment	Sample and its field duplicate ≥ 5x the CRQL and RPD > 50%	Qualify sample and its duplicate as estimated (J)
	Sample and/or its field duplicate < 5x the CRQL and absolute difference > 2x the CRQL	Qualify results > the MDL as estimated (J) Qualify non-detects as estimated (UJ)

Field/Rinsate/Trip Blanks

Action:

NOTE: Designate "Field Blank" as such on Form IA.
Field Blank results previously rejected due to other criteria cannot be used to qualify field samples.
Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.
If the MDL is > the CRQL, substitute CRQL with 2x the MDL.

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 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 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. If the absolute value of any analyte in a Field/Rinsate/Trip Blank is > the CRQL, then the CRQL shall be raised to the level in the Field/Rinsate/Trip Blank and the associated sample data below this level shall be reported as CRQL-U.
5. Sample results > the Field/Rinsate/Trip Blank value but < 10x the Field/Rinsate/Trip Blank value shall be qualified as estimated (J).
6. Sample results ≥ the MDL but ≤ the CRQL shall be reported at the CRQL value with a "U".

Table 13. Field/Rinsate/Trip Blank Actions for ICP-MS Analysis

Blank Result	Sample Result	Action for Samples
> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
	> CRQL but < Blank Result	Report at level of Blank Result with a "U"
	> Blank Result but < 10x the Blank Result	Use professional judgment to qualify results as estimated (J)
	≥ 10x the Blank Results	No Qualification
≥ MDL but ≤ CRQL	See page 19 table 5 for criteria.	Qualify samples the same as ICB/CCB/PB method blank criteria (page 19, Table 5).

Linear Ranges

Action:

1. If any sample result was higher than the high linear range for ICP-MS and the sample was not diluted to obtain the result reported on Form I, qualify the affected results ≥ MDL as estimated (J).

Percent Solids of Sediments

Action:

1. If the percent solids in sediment for a sample are < 50%, qualify the affected results ≥ MDL as estimated (J) and the non-detects as estimated (UJ).

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

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

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-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:

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

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:

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

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 of ISM02.2 - 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:

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

Where,

C	=	MDL or CRQL (mg/kg)
W_M	=	Minimum method required aliquot amount (g) (1.00 g or 0.50 g)
W	=	Initial aliquot amount (g)
V_M	=	Method required final sample digestion volume (mL) (100 mL)
V_f	=	Final digestion volume (mL)
S	=	% Solids/100 (see Exhibit D of ISM02.2 - 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 EXECUTIVE NARRATIVE TEMPLATE

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
DESA/HWSB/HWSS
2890, Woodbridge Avenue, Edison, NJ 08837

EXECUTIVE NARRATIVE

Case No. :
Site:
SDG No.:
Laboratory:

QAPP
HWSS #:
Contractor #:
Number of Samples:
Sampling date:

SUMMARY:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data have been qualified "J" estimated.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings: None

Major Findings: None

Minor Findings: None

COMMENTS:

Validator's Signature: **Date:**
Name:
Affiliation:

Approver's Signature: **Date:**
Name:
Affiliation:

1

APPENDIX C: SAMPLE INORGANIC DATA SAMPLE SUMMARY

Case No: 00001	Contract: XYZ1234	SDG No: XY123	Lab Code: ABCD
Sample Number: XY123	Method: ICP_AES	Matrix: FLUFF	MA Number: DEFAULT
Sample Location: SOMEWHERE OUT THERE	pH: 15	Sample Date: 13322059	Sample Time: 24:03:00
% Moisture :		% Solids :	

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable	Validation Level
Aluminum	400	ug/L	1			Yes	S2BVEM
Antimony	40	ug/L	1	U	U	Yes	S2BVEM
Arsenic	40	ug/L	1	U	U	Yes	S2BVEM
Barium	40	ug/L	1	U	U	Yes	S2BVEM
Beryllium	40	ug/L	1	U	U	Yes	S2BVEM
Cadmium	40	ug/L	1	U	U	Yes	S2BVEM
Calcium	400	ug/L	1			Yes	S2BVEM
Chromium	40	ug/L	1	U	U	Yes	S2BVEM
Cobalt	40	ug/L	1	U	U	Yes	S2BVEM
Copper	40	ug/L	1	U	U	Yes	S2BVEM
Iron	40	ug/L	1	J	U	Yes	S2BVEM
Lead	40	ug/L	1	U	U	Yes	S2BVEM
Magnesium	400	ug/L	1			Yes	S2BVEM
Manganese	400	ug/L	1			Yes	S2BVEM
Nickel	40	ug/L	1	U	U	Yes	S2BVEM
Potassium	400	ug/L	1			Yes	S2BVEM
Selenium	40	ug/L	1	U	U	Yes	S2BVEM
Silver	40	ug/L	1	U	U	Yes	S2BVEM
Sodium	400	ug/L	1			Yes	S2BVEM
Thallium	40	ug/L	1	U	U	Yes	S2BVEM
Vanadium	40	ug/L	1	U	U	Yes	S2BVEM
Zinc	40	ug/L	1	U	U	Yes	S2BVEM

APPENDIX D: ELECTRONIC DATA DELIVERABLE TEMPLATE

DATA_PROVIDER	LAB_MATRIX_CODE	RESULT_UNIT
SYS_SAMPLE_CODE	ANAL_LOCATION	DETECTION_LIMIT_UNIT
SAMPLE_NAME	BASIS	TIC_RETENTION_TIME
SAMPLE_MATRIX_CODE	CONTAINER_ID	RESULT_COMMENT
SAMPLE_TYPE_CODE	DILUTION_FACTOR	QC_ORIGINAL_CONC
SAMPLE_SOURCE	PREP_METHOD	QC_SPIKE_ADDED
PARENT_SAMPLE_CODE	PREP_DATE	QC_SPIKE_MEASURED
SAMPLE_DEL_GROUP	LEACHATE_METHOD	QC_SPIKE_RECOVERY
SAMPLE_DATE	LEACHATE_DATE	QC_DUP_ORIGINAL_CONC
SYS_LOC_CODE	LAB_NAME_CODE	QC_DUP_SPIKE_ADDED
START_DEPTH	QC_LEVEL	QC_DUP_SPIKE_MEASURED
END_DEPTH	LAB_SAMPLE_ID	QC_DUP_SPIKE_RECOVERY
DEPTH_UNIT	PERCENT_MOISTURE	QC_RPD
CHAIN_OF_CUSTODY	SUBSAMPLE_AMOUNT	QC_SPIKE_LCL
SENT_TO_LAB_DATE	SUBSAMPLE_AMOUNT_UNIT	QC_SPIKE_UCL
SAMPLE_RECEIPT_DATE	ANALYST_NAME	QC_RPD_CL
SAMPLER	INSTRUMENT_ID	QC_SPIKE_STATUS
SAMPLING_COMPANY_CODE	COMMENT	QC_DUP_SPIKE_STATUS
SAMPLING_REASON	PRESERVATIVE	QC_RPD_STATUS
SAMPLING_TECHNIQUE	FINAL_VOLUME	BREAK_2
TASK_CODE	FINAL_VOLUME_UNIT	SYS_SAMPLE_CODE
COLLECTION_QUARTER	CAS_RN	LAB_ANL_METHOD_NAME
COMPOSITE_YN	CHEMICAL_NAME	ANALYSIS_DATE
COMPOSITE_DESC	RESULT_VALUE	TOTAL_OR_DISSOLVED
SAMPLE_CLASS	RESULT_ERROR_DELTA	COLUMN_NUMBER
CUSTOM_FIELD_1	RESULT_TYPE_CODE	TEST_TYPE
CUSTOM_FIELD_2	REPORTABLE_RESULT	TEST_BATCH_TYPE
CUSTOM_FIELD_3	DETECT_FLAG	TEST_BATCH_ID
COMMENT	LAB_QUALIFIERS	CASE
BREAK_1	VALIDATOR_QUALIFIERS	CONTRACT_NUM
SYS_SAMPLE_CODE	INTERPRETED_QUALIFIERS	SCRIBE_SAMPLE_ID
LAB_ANL_METHOD_NAME	ORGANIC_YN	SAMPLE_TIME
ANALYSIS_DATE	METHOD_DETECTION_LIMIT	FRACTION
TOTAL_OR_DISSOLVED	REPORTING_DETECTION_LIMIT	PH
COLUMN_NUMBER	QUANTITATION_LIMIT	DATA_VAL_LABEL
TEST_TYPE		