USEPA REGION 4

Science and Ecosystem Support Division
Management & Technical Support Branch
Quality Assurance Section
980 College Station Road
Athens, Georgia 30605-2720

Data Validation Standard Operating Procedures

For

CONTRACT LABORATORY PROGRAM INORGANIC DATA BY

INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION SPECTROSCOPY

AND

INDUCTIVELY COUPLED PLASMA – MASS SPECTROSCOPY

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Periodic Review

Reviewer:				
Date:				

HISTORY OF REVISIONS

Revision Number	Issue Date	Action	Description
2.0	08/24/2011	Original	Original SOP for the Quality Assurance Section applicable to Data Review / Validation for Externally Generated Analytical Data Generated for Region 4

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1.0 Purpose

The United States Environmental Protection Agency (USEPA), Contract Laboratory Program (CLP) is a key provider of analytical services to the Superfund Program. The Quality Assurance Section of the Science and Ecosystems Support Division (SESD), in conjunction with the Environmental Services Assistance Team (ESAT) contractor, is responsible for providing data review and validation services in support of Superfund data collection activities performed within Region 4.

2.0 Applicability

This Standard Operating Procedure is applicable to the review of water, soils and sediment metals data by ICP-AES or ICP-MS analysis at trace and low to medium concentrations. It is further based on the quality assurance/quality control (QA/QC) and technical requirements specified in Exhibit D of SOW ISM01.2, and revisions.

This document provides the criteria for performing technical and quality assurance reviews of data generated by contract laboratories under the CLP Statement of Work (SOW) - ISM01.2, Inorganic Superfund Methods, Multi-Media, Multi-Concentration, January 2010, and revisions. This SOP incorporates the content of the National Functional Guidelines for Inorganic Superfund Data Review (NFG) January 2010, and revisions. However, this SOP provides additional guidance to limit the use of professional judgment by taking into account region-specific data review and validation requirements and reporting formats, etc. This SOP does not contain the procedures for entering qualified data into the Region 4 LIMS system – this information is contained in a separate SOP. Contract compliance or data usability issues pertinent to risk assessment activities, are not addressed in this document.

This SOP shall be followed without deviation to ensure that a consistent data review product is provided to the Region 4 - CLP Inorganic Task Order/Project Officer (TO/PO). If the data reviewer(s), using professional judgment, decide to take exception to any of the criteria or actions specified in this SOP, he/she must consult the TO/PO prior to making any changes. No deviations from the specified criteria or actions stipulated in this SOP will be undertaken by the data reviewer(s) unless those changes are authorized, in writing, by the TO/PO.

Authorized deviations will be documented in the data review memorandum.

3.0 Personnel Qualifications

For EPA personnel a minimum of a four year degree from an accredited college or university in a scientific field is required. Experience in analyzing environmental samples, and in performing data review / validation is also recommended.

4.0 Procedural Steps: Data Processing

Samples are collected by EPA, contractor, or state personnel and then are submitted to an assigned contract laboratory for analysis. The laboratory analyzes the samples according to specified analytical protocols, assembles a data package and an electronic data file in accordance with specifications in the contract. The original data package is submitted to the Science and Ecosystem Support Division (SESD), Athens, Georgia, and a copy, along with the electronic data deliverable (EDD), are delivered to the Sample Management Office (SMO) / Data Assessment Support Services (DASS) contractor.

4.1 Contact Compliance Screening

At SMO/DASS, the data package and the EDD are checked for compliance with the contract. A Contract Compliance Screening (CCS) report is issued to the region and is posted on the WebDat web site. The EDD is then processed electronically to evaluate QC performance against the NFG and Region 4 data quality guidelines by the Electronic data eXchange and Evaluation System (EXES). Currently, for the routine inorganic contracts, a SEDD Stage 2a EDD is submitted by the laboratories.

4.1.1 Electronic Data Review - National Functional Guideline Report

A report of this electronic review (the NFG report) is submitted to the region, along with a text file containing the results, qualified in accordance with the Region 4 data qualifier hierarchy. The data package delivered to SESD is audited for evidentiary completeness. The report(s) of the electronic review (if available for all samples in the case) is examined to identify any issues that warrant further investigation. The results of Performance Evaluation Samples (PES) are scored and the data are appropriately qualified.

4.1.2 Manual Data Review

In the event that no electronic review was performed or the report(s) is not available, the data are manually reviewed for technical quality and for compliance with Region 4 data quality requirements, beginning with the case or SDG (Sample Delivery Group) narrative, the original unprocessed or raw data, the QC summary forms, and the sample tracking and processing information included in the package. Region 4 data qualifiers, intended to provide the customer with a more complete understanding of the factors affecting data quality, are added to the results. A report of this review is prepared to complete the documentation of data quality, and the data are electronically entered into the Region 4 laboratory information management system, *Element*. Review reports and project documents are maintained by the SESD Quality Assurance Section (QAS), and the data package is archived. Completed data validation reports should contain the following statement: A Stage 4 validation consisting of electronic and manual review was performed on the inorganic samples submitted as part of this case.

4.2 Procedural Steps: Review / Validation of Metals Data by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) or Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)

4.2.1. Holding Times/Preservation

Holding times are evaluated from the perspective of technical or actual holding times. These are determined as the age of the sample from date and time of sample collection to the data and time of sample preparation/digestion, and analysis. The contractual holding times are determined from the Validated Time of Sample Receipt (VTSR) and are used for contract compliance but will not be addressed in this SOP.

The following guidance is based on past practice in Region 4 and on the best available information on matrix holding times from 40CFR Part 136 requirements, as well as other USEPA guidance: The technical holding time is calculated from the time and date of sample collection to the date of analysis. The time and date of collection is located on the Traffic Report/Chain-of-Custody (TR/COC) form included in the analytical data package. The dates of sample preparation and analysis are located on the Form XII-IN and the raw data. If holding times are exceeded or proper preservation has not occurred, describe this in the data review summary case narrative and take the appropriate actions.

Criteria:

- The technical holding time criteria for aqueous/water metal samples is 180 days, preserved with nitric acid to a pH < 2. The addition of nitric acid to the environmental sample applies only to water samples.

- The technical holding time criteria for soil/sediment metal samples is 180 days and is based on the technical holding time criteria for aqueous/water samples.

- The technical holding time criteria for wipe and air filter samples is 180 days and is based on the technical holding time criteria for aqueous/water samples.

- Aqueous/water and soil/sediment wipe samples shall be maintained at 4 ºC (± 2ºC) until sample preparation. Air filter and wipe samples may be maintained at room temperature until preparation.

NOTE: Concentrations of metals detected between the MDL and the CRQL, shall be qualified as J, Q-2.

Table 1 Holding Time & Preservation for Metals

Holding Time	Preservation	Action for Samples
Samples prep/analyzed within180 days of collection	Aqueous/water samples preserved in field with acid to pH < 2.	No action required.
Samples prep/analyzed within180 days of collection	Aqueous/water samples **were not** preserved in the field with acid to pH < 2. Lab **did not preserve** sample(s) upon receipt.	Non-Detects are unusable: R, P-4 Detects (> MDL but < CRQL): R, P-4, Q-2 Detects (≥ CRQL): J, P-4
Samples prep/analyzed within180 days of collection	Aqueous/water samples **were not** preserved in the field with acid to pH < 2. But Lab staff **preserved** sample(s) with nitric upon receipt.	***Lab Allowed sample to sit for at least 24 hour prior to prep/analysis***. Non-Detects: No Qualification Detects: No Qualification
Samples prep/analyzed within180 days of collection	Aqueous/water samples **were not preserved** in the field with acid to pH < 2. But Lab staff **preserved** sample with nitric upon receipt.	***Lab Did Not allow the sample to sit for at least 24 hours prior to prep/analysis.*** Non-Detects: J, P-6 Detects (>MDL but < CRQL): J, Q-2, P-6 Detects (≥ CRQL): J, P-6
Samples prep/analyzed after180 days of collection	Aqueous/water samples preserved in field with acid to pH < 2.	Non-Detects: R, H-1 Detects (> MDL but < CRQL): J, Q-2. H-1 Detects (≥ CRQL): J, H-1
Samples prep/analyzed after180 days of collection	Aqueous/water samples **were not preserved** in field or laboratory with acid to pH < 2.	All Results: R, H-1, P-2
Samples prep/analyzed within180 days of collection	Samples not received or stored at 2 ºC - 6 ºC for aqueous/water, soil / sediment samples	**Do Not Qualify Data. Document in Case Narrative.**
Samples prep/analyzed after180 days from collection	Aqueous, soil / sediment, air filter and wipe samples.	Non-Detects: R, H-1 Detects (> MDL but < CRQL): J, Q-2. H-1 Detects (≥ CRQL): J, H-1

4.2.2 ICP-MS Tune Analysis (Form XIV-IN)

The Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) tune serves as an initial demonstration of instrument stability, identification and precision. No analytical results should be reported if the tune analysis did not meet, in full, the SOW-ISM01.2 requirements.

For ICP-MS metals analysis, the laboratory must perform any necessary mass calibration and resolution routines to bring peak width within the manufacturer's specifications and adjust mass calibration to within 0.1 atomic mass unit (amu) over the range of 6 to 210 amu. Demonstrate instrument stability and precision by analyzing the tuning solution as a single analysis with at least five consecutive integrations. The percent relative standard deviation of the absolute signals for all the multiple integrations in the tuning solution, as calculated by the instrument, must be less than 5.0% for each analyte.

Criteria:

- 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 calibration to within 0.1 amu over the range of 6-210 amu.

- The Percent Relative Standard Deviation (%RSD) of the absolute signals for all analytes (and for each isotope) in the tuning solution must be < 5%.

Table 2 Tune Check for ICP-MS

ICP-MS Tune Results	Action for Samples
Tune not Performed prior to Initial Calibration	All Results: R, CLP37
Tune not Performed Properly	Non-Detects: R, CLP38 Detects (> MDL but < CRQL): R, CLP38 Detects (≥ CRQL): J, CLP38
Resolution of Mass Calibration not within 0.1 amu.	Non-Detects: J, CLP38 Detects (> MDL but < CRQL): J, Q-2, CLP38 Detects (≥ CRQL): J, CLP38
The Relative Percent Difference is > 5%.	Non-Detects: J, CLP38 Detects (> MDL but < CRQL): J, Q-2, CLP38 Detects (≥ CRQL): J, CLP38

4.2.3 Initial Calibration and Continuing Calibration (Form IIA-IN)

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.

For ICP-AES and ICP-MS analysis, 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 residuals for all of the non-zero standards must be within 70-130% of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

4.2.3.1 Initial and Continuing Calibration Verification (ICV and CCV) - Acceptance Criteria

The acceptance criteria for the ICP-AES and ICP-MS, ICVs and CCVs are:

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

4.2.3.2 Initial Calibration Verification (ICV) - The following ICV requirements apply to the ICP-AES and ICP-MS.

Criteria:

- Immediately after each ICP-AES & 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.

- 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.

- The ICV solution shall be run at each analytical wavelength (ICP-AES) and mass (ICP-MS) used for analysis.

4.2.3.3 Continuing Calibration Verification (CCV) - The following CCV requirements applies to the ICP-AES and ICP-MS.

Criteria:

- 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.

- 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.

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

- The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG).

- 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.

4.2.3.4 Preliminaries: ICV & CCV for ICP-AES & ICP-MS

Criteria:

- 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.

- 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). Refer to Table 3 below.

- 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).

- The Acceptance Criteria for ICV and CCVs are 90 – 110 of the true value.

Table 3 ICV & CCV for ICP-AES & ICP-MS

Calibration Result	Action for Samples
Calibration not performed	All Results: R, Custom Flag
Calibration incomplete	Non-Detects: R, Custom Flag Detects (> MDL but < CRQL): R, Custom Flag Detects (≥ CRQL): J, Custom Flag
Correlation coefficient <0.995; residuals outside 70-130%; y intercept ≥ CRQL	Non-Detects: R, CLP16 Detects (> MDL): J, CLP16
ICV/CCV %R < 75% (30% to 50%)	Non-Detects: R, QC-5 Detects (> MDL but < CRQL): J, QC-5, Q-2 Detects (≥ CRQL): J, QC-5
ICV/CCV %R < 90% (but > 75%)	Non-Detects: J, QC-5 Detects (> MDL but (< CRQL): J, QC-5, Q-2 Detects (≥ CRQL): J, QC-5
ICV/CCV %R < 125% (but > 110 %)	Non-Detects: No Qualification Required Detects (> MDL but < CRQL): J, Q-2, QC-6 Detects (≥ CRQL): J, QC-6
ICV/CCV %R > 125%	Non-Detects: No Qualification Required Detects (> MDL but < CRQL): R, Q-2, QC-6 Detects (≥ CRQL): J, QC-6
ICV/CCV %R > 160%	Non-Detects: No Qualification Required Detects (> MDL but < CRQL): R, Q-2, QC-6 Detects (≥ CRQL): R, QC-6

4.2.4. Blanks (Form III-IN)

The objective of evaluating blank analyses 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., preparation blanks and calibration blanks). However, it has been Region 4 data validation policy to evaluate the field blanks (i.e., equipment blanks, rinsates, and CLP metals blind blank, etc.) as part of the validation process but not to qualify the data based on these samples. The case narrative will address field blank contamination if data quality is compromised. Contact the Region 4 CLP Inorganic TO/PO for further direction regarding the treatment of blank contamination.

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. Contact the Region 4 CLP Inorganic TO/PO for further direction regarding the treatment of blank contamination.

Note: For non-CLP analyses, consult the CLP Inorganic TO/PO prior to data review to discuss how blank contamination will be treated.

Criteria:

- No contaminants should be found in the blank(s).
- 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.
- A Continuing Calibration Blank (CCB) shall be analyzed at each mass or wavelength used for the analysis, immediately after every ICV and 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.
- 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.
- 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 analyte concentrations < 10x the Preparation Blank concentration, and > CRQL, should be re-digested and reanalyzed for those analyte(s), with the exception being analytes detected in field blanks and CLP metals blind blanks (which are not used to qualify sample data.. The laboratory is not to correct the sample concentration for the blank value.

- 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 re-digested and reanalyzed.

  Note: To avoid confusion, use the blank containing the highest concentration of analyte(s) as a basis for qualifying all the sample(s) in the analytical sequence.

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

- For CCBs that do not meet the technical criteria, apply the action to all samples in the analytical sequence.

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

- The frequency and sequence of analysis for all of the required blanks should be consistent with requirements specified in ISM01.2. Exhibit D, Part A, Section 12.3 (for ICP-AES) and Exhibit D, Part B, Section 12.4 (for ICP-MS).

Table 4 Blanks for ICP-AES and ICP-MS

Blank Type	Blank Result[1]	Sample Result	Action for Samples
Preparation Blank ICB/CCB (No Field Blanks)[1]	Detects	Non-Detects	No Qualification Required
	< CRQL	< CRQL	Report CRQL value with a U.
	Detects	≥ CRQL and ≤ 10 x Blank[2]	Report result with a U, B-4
	Detects	≥ CRQL and > 10 x Blank	No Qualification
Preparation Blank ICB/CCB (No Field Blanks)	≤ - MDL but ≥ - CRQL	Non-Detect: absolute value	Report CRQL value with a U.
		Detects: absolute value at ≥ CRQL and < 10 x Blank	Non-Detects: Raise to CRQL and qualify U, B-4 Detects **(> MDL but < CRQL & < 10x blank**): Raise to CRQL and qualify U, B-4 Detects **(≥ CRQL but < 10 x blank**): Qualify result U, B-4 Detects (**≤ CRQL and ≥ 10x blank result**) : No qualification Detects (≥ CRQL and ≥ 10x blank): No qualification
		Detects: absolute value at ≥ CRQL and > 10 x Blank	No Qualification

NOTE: **From the various blanks analyzed for a given SDG, select the blank containing the highest concentration of a detected analyte(s) and use this blank to evaluate / qualify the associated samples/data.**

[1] If significant contamination of field blanks, CLP metals blind blank, and/or equipment/rinsate blanks occurs, the data user will be informed of this via the data validation memorandum. Do not qualify the data based on these blanks.

[2] The 10x rule applies to all metals detected in the relevant blank samples.

4.2.5 Interference Check Samples for ICP/AES and ICP/MS

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.

The laboratory shall analyze and report the results for the ICS for all elements on the Target Analyte List (TAL) and for all interferents (target and non-target) immediately after the initial calibration sequence, but not before the ICV/ICB.

The analysis of the ICS shall be immediately followed by the analysis of a CCV/CCB pair. The ICS solutions shall be obtained from USEPA, if available, and analyzed according to the instructions supplied with the ICS. The Contractor shall always initially run the ICS undiluted. Dilution of the ICS (for the highest concentration elements) may be necessary to meet the calibrated range values of the instrument.

Criteria: ICP/AES - Forms IVA-IN, IVB-IN, XIII-IN, instrument printouts, and raw data.

- 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).

- An ICS must be run at the beginning of each sample analysis run. The ICS is not 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).

- 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 target analytes and interfering analytes.

- 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 target analytes and interfering analytes included in the solution.

- 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 re-verified, and the affected samples reanalyzed.

- The ICS should be obtained from USEPA if available, and analyzed according to the instructions supplied with the solutions. The ICS may be prepared with the interfering analytes at 2x the level specified in the Statement of Work (SOW) if high levels of interfering analytes are found in the field samples. If the ICS is not available from USEPA, an independent ICS solution shall be prepared with the interfering and target analyte concentrations at the levels specified in the method.

Table 5 Interference Check Samples for ICP/AES

Interference Check Sample Results	Action for Samples[3]
ICS %R > 120% (or > true value + CRQL)	Non-Detects: No Qualification Detects > MDL: J
ICS %R 50-79% (or < true value - CRQL)	Non-Detects: J Detects > MDL: J
ICSAB %R < 50%	Non-Detects & Detects: R
Potential false positives in field samples with interfering analytes	Non-Detects: No Qualification Detects > MDL: J
Potential false negatives in field samples with interfering analytes	Non-Detects: J Detects > MDL but < 10x (|negative value|): J

Criteria: ICP-MS Forms IVA-IN, IVB-IN, XIII-IN, instrument printouts, and raw data

- The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interfering analytes, and Solution AB consists of the target analytes mixed with interfering analytes. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A, for all masses used for each target or interfering analyte reported by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).

[3] Depending on the analyte, you will need to determine whether interferences or over corrections have occurred based on the results of the ICSA or the ICSAB solutions. For isobaric interferences, the CLP22 definition flag should be assigned to the affected data point(s). If overcorrection is encountered, the CLP23 definition flag should be assigned to the affected data point(s)

- 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).

- 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.

- 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.

- 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 re-verified, and all analytical samples analyzed since the last compliant ICS reanalyzed.

- 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 interfering and target analyte concentrations at the levels specified in the method.

Table 6 Interference Check Samples for ICP/MS

Interference Check Sample Results	Action for Samples[4]
ICS %R > 120% (or > true value + 2x the CRQL)	Non-Detects: No Qualification Detects > MDL: J
ICS %R 50-79% (or < true value - 2x the CRQL)	Non-Detects: J Detects > MDL: J
ICSAB %R < 50%	Non-Detects & Detects: R
Potential false positives in field samples with interfering analytes	Non-Detects: No Qualification Detects > MDL: J
Potential false negatives in field samples with interfering analytes	Non-Detects: J Detects > MDL but < 10x (|negative value|): J

If overcorrection is suspected / encountered, the CLP23 definition flag should be assigned to the affected data point(s).

NOTE: All positive results for arsenic, selenium and thallium by ICP-AES (when ICP-MS analysis for these analytes was not used), shall be qualified with the CLP36 qualifier which states that the identification and quantitation of these results **have not been** confirmed by ICP-MS. This qualifier will be attached in addition to any other qualifiers that may be assigned to these data.

4.2.6 Internal Standards for ICP-MS (Form XI-IN)

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 within an analytical sequence that begins with the calibration.

[4] Depending on the analyte, you'll need to determine whether interferences or over corrections have occurred based on the results of the ICSA solution. For isobaric interferences, the CLP22 definition flag should be assigned to the affected data point(s).

This information may be used to correct potential problems caused by mass dependent drift, errors incurred in adding the internal standards, or increases in the concentrations of individual internal standards caused by background contributions from the sample.

Criteria:

- All samples analyzed within an analytical sequence, 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.

- 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.

- 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.

Table 7 Internal Standards for ICP-MS

Internal Standard Results	Action for Samples
No internal standards used.	Qualify all results as unusable: R, Custom Flag
< 5 of the required internal standards	Qualify **all associated analytical results** as estimated: J, Custom Flag
Target analyte not associated with internal standard	Non-Detects & Detects: R, Custom Flag
%RI < 30% for an undiluted analysis and no reanalysis was performed.	Reject all results (analytes) associated with the particular internal standard. Qualifiers: R, QI-1.
%RI < 60% but above 30% for an undiluted analysis and no reanalysis was performed.	Non-Detects: R, QI-1 Detects (> MDL but < CRQL): J, Q-2, QI-1 Detects (≥ CRQL): J, QI-1
%RI > 125%, and original sample reanalyzed at 2-fold dilution	If %RI of diluted sample analysis is 60-125%, do not qualify the data and use this result If the %RI of the diluted sample analysis is outside the 60- 125% limit, qualify undiluted results that are ≥ MDL: J, QI-1 Undiluted Non-Detects: J, QI-1
Original sample not reanalyzed at 2-fold dilution*	Undiluted results for Detects: R, QI-1 Undiluted results for Non-Detects: J, QI-1

* Assuming IS recoveries are suppressed.

4.2.7 Laboratory Control Samples (Form VII-IN)

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

Criteria for ICP-AES:

- 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.

- 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.

- 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 re-digested and reanalyzed.

- 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.

- All wipe or filter sample LCS %R shall fall within the control limits of 70-130%.

Criteria for ICP-MS:

- 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.

- 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.

- 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 re-digested and reanalyzed.

Table 8 Water, Soil/Sediment & Wipe Laboratory Control Samples for ICP-AES

Laboratory Control Samples	Action for Samples
If Water/Soil/Sediment LCS % Rec. are within 40 – 50% (20 – 49% for Ag & Sb)	Non-Detects: J, QL-1 Detects (> MDL but < CRQL): J, Q-2, QL-1 Detects (≥ CRQL): J, QL-1
If Water/Soil/Sediment LCS %Rec. are < 40% (<20% for Ag & Sb)	Non-Detects: R, QL-1 Detects (> MDL but < CRQL): R, Q-2, QL-1 Detects (≥ CRQL): J, QL-1
If Water/Soil/Sediment LCS % Rec. are %R > 130% (150% Ag, Sb)	Non-Detects: No Qualification Required. Detects (> MDL but < CRQL): J, Q-2, QL-2 Detects (≥ CRQL): J, QL-2
If Aqueous/Soil/Sediment % Rec. are > 150% (>170% Ag, Sb)	Non-Detects: No Qualification Required Detects (> MDL but < CRQL): R, Q-2, QL-2 Detects (≥ CRQL): J, QL-2
If Wipe/Filter LCS % Rec. are 40-50%	Non-Detects: J, QL-1 Detects (> MDL but < CRQL): J, Q-2, QL-1 Detects (≥ CRQL): J, QL-1
If Wipe/Filter LCS % Rec. are < 40%	Non-Detects: R, QL-1 Detects (> MDL but < CRQL): R, Q-2, QL-1 Detects (≥ CRQL): J, QL-1
If Wipe/Filter LCS % Rec. are > 130% but < 150%	Non-Detects: No Qualification Required Detects (> MDL but < CRQL): J, Q-2, QL-2 Detects (≥ CRQL): J, QL-2
If Wipe/Filter LCS % Rec. are > 150 %	Non-Detects: No Qualification Required Detects (> MDL but < CRQL): R, Q-2, QL-2 Detects (≥ CRQL): J, QL-2

Table 9 Aqueous & Soil/Sediment Laboratory Control Samples for ICP-MS

Laboratory Control Samples	Action for Samples
If Aqueous/Soil/Sediment LCS % Rec. are within 40 – 50% (20 – 49% for Ag & Sb)	Non-Detects: J, QL-1 Detects (> MDL but < CRQL): J, Q-2, QL-1 Detects (≥ CRQL): J, QL-1
If Aqueous/Soil/Sediment LCS %Rec. are < 40% (<20% for Ag & Sb)	Non-Detects: R, QL-1 Detects (> MDL but < CRQL): R, Q-2, QL-1 Detects (≥ CRQL): J, QL-1
If Aqueous/Soil/Sediment LCS % Rec. are %R > 130% (150% Ag, Sb)	Non-Detects: No Qualification Required. Detects (> MDL but < CRQL): J, Q-2, QL-2 Detects (≥ CRQL): J, QL-2
If Aqueous/Soil/Sediment % Rec. are > 150% (>170% Ag, Sb)	Non-Detects: No Qualification Required Detects (> MDL but < CRQL): R, Q-2, QL-2 Detects (≥ CRQL): J, QL-2

4.2.8. Duplicates, Matrix Spike/Matrix Spike Duplicates & Post Digestion Spikes (Forms VI-IN & VA-IN, VB-IN – Post Digestion Spike)

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.

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.

Criteria: Duplicate Samples - ICP-AES & ICP-MS

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

- 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 IIN. 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.

- A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original water sample and water duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of 35% for the Relative Percent Difference (RPD) shall be used for original soil/sediment and soil/sediment duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).

- 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.

Table 10 Duplicate Samples

Duplicate Sample Results	Action for Samples
Both original water sample and water duplicate sample > 5x the CRQL and RPD > 20%.	Non-Detects: No qualification required Detects (> MDL but < CRQL): J, Q-2, QM-4 Detects (≥ CRQL): J, QM-4
Both original soil/sediment sample and soil/sediment duplicate sample > 5x the CRQL and RPD > 35%.	Non-Detects: No qualification required Detects (> MDL but < CRQL): J, Q-2, QM-4 Detects (≥ CRQL): J, QM-4
Original samples or duplicate samples ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL.	Non-Detects: J, QM-4 Detects (> MDL but < CRQL): J, Q-2, QM-4 Detects (≥ CRQL): J, QM-4

Criteria: Matrix Spike Samples – ICP-AES & ICP-MS

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

- 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.

- 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 un-spiked sample shall be spiked at 2x the indigenous level or 2x the Contract Required Quantitation Limit (CRQL), whichever is greater.

- 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 un-qualified, even if the %R does not meet the acceptance criteria.

- 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.

- When the duplicate is not within the required acceptance limits, only qualify the sample associated with the duplicate sample.

- The required spike recoveries for metals are 75-125% for each analyte.

NOTES: Only the field sample associated with the non-performing MS/MSD should be qualified unless instructed otherwise by the EPA Inorganic TO/PO.

Table 11 Pre and Post Digestion Spike Samples for ICP-AES & ICP-MS

Spike Sample Results	Action for Samples*
Matrix Spike %R ≤ 10% Post-digestion spike %R < 75%	**Reject non-detects** for that analyte(s) in the field sample associated with the MS/MSD: R, QM-6, CLP34 Detect (> MDL but < CRQL): R, Q-2, QM-6, CLP34 Detect (≥ CRQL): J, CLP34, CLP34
Matrix Spike %R 10% - 75% Post-digestion spike %R < 75%	Non-Detects: R, QM-1, CLP-34 Detects (> MDL but < CRQL): J, Q-2, QM-1, CLP34 Detects (≥ CRQL): J, QM-1, CLP34
Matrix Spike %R 10% - 75% Post Digestion Spike % R 75% - 125%	Non-Detects: J, QM-1 Detects (> MDL but < CRQL): J, Q-2, QM-1 Detects (≥ CRQL): J, QM-1
Matrix Spike %R > 125% Post-digestion spike %R < 75%	Non-detects: J, QM-1, CLP34 Detects (> MDL but < CRQL): J, Q-2, QM-1, CLP-34 Detects (≥ CRQL): J, QM-1, CLP34
Matrix Spike %R > 125% Post-digestion spike %R 75 - 125%	Non-Detects: No qualification required. Detects: (> MDL but ≤ CRQL): J, Q-2, QM-2 Detects (≥ CRQL): J, QM-2
Matrix Spike %R > 125% Post-digestion spike %R >125% but <150%	Non-Detects: No qualification required. Detects (> MDL but ≤ CRQL): J, Q-2, QM-2, CLP-35 Detects (≥ CRQL): J, QM-2, CLP35
Matrix Spike %R > 125% & Post Digestion Spike ≥ 150%	Non-Detects: No Qualification **Reject detects** for that analyte(s) in the field sample associated with the MS/MSD: R, QM-2, CLP35
Matrix Spike %R < 10% No post digestion spike performed (e.g. not required for Ag)	**Reject all data** in the field sample associated with the MS/MSD: R, QM-1
Matrix Spike %R < 50% No post-digestion spike performed (e.g., not required for Ag)	Non-Detects: R, QM-1 Detects (> MDL but < CRQL): J, Q-2, QM-1 Detects (≥ CRQL): J, QM-1
Matrix Spike %R 50% - 74% No post-digestion spike performed (e.g., not required for Ag)	Non-Detects: J, QM-1 Detects (> MDL but < CRQL): J, Q-2, QM-1 Detects (≥ CRQL): J, QM-1
Matrix Spike %R > 125% No post-digestion spike performed (e.g., not required for Ag)	Non-Detects: No qualification required. **Reject detects** for that analyte(s) in the field sample associated with the MS/MSD: R, QM-1

4.2.9. Serial Dilutions for ICP-AES & ICP-MS (Form VIII-IN)

The serial dilution of samples quantitated by ICP-AES or ICP-MS determines whether or not significant physical or chemical interferences exist due to sample matrix.

Criteria:

- 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.
- Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for the ICP Serial Dilution analysis.
- If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the serial dilution analysis (a five-fold dilution) shall then agree within a 10 Percent Difference (%D) of the original determination after correction for dilution.

Table 12 Serial Dilutions

Serial Dilution Result	Action for Samples
Sample concentration > 50x MDL and the %D > 10	Non-Detects: J, Q-5. Detects > MDL but < CRQL J, Q-2, Q-5 Detects ≥ CRQL: J, Q-5
Interferences present	**Consult with the R4 CLP TO/PO**

4.2.10 Performance Evaluation Samples

A Performance Evaluation Sample (PES) or a set of PE samples are included as part of each project sample delivery group (SDG) submitted to the CLP Program for analysis. For larger projects, including sampling efforts extending for more than one week, multiple sets of PES may be used. The laboratories are required to prepare and analyze the PES with the field samples of the associated case/SDG. If the PES is not prepared, digested and/or analyzed concurrently with field samples for a particular project/SDG/case, the data reviewer shall contact the EPA Inorganic TO/PO for further instructions. The TO/PO may decide that it is not appropriate to use the PES for data qualification.

The table below summarizes data qualification required based on the PES scoring results. Under certain circumstances, the spiked analyte(s) are not evaluated by scoring software. This may occur when either lower limits do not exist for a particular analyte or the analyte was not evaluated.

The reviewer may describe instances in the narrative when the laboratory failed to identify a spiked analyte for which lower limits did not exist but PES database statistics suggest that the analyte should still have been identified by the laboratory. Additionally, all analytes which are scored as PES contaminants, either less than or greater than the CRQL, are treated as method blank contaminants, applying standard blank rules described in Section 5 above.

If only one set of PES is included in a case, all samples will be qualified based on the PES scoring. If multiple sets of PES are included, all data for the associated sampling week will be qualified based on the PES scoring.

Table 13 Performance Evaluation Samples

PES Score	Actions for Samples
Within Limits for all analytes	Non-Detects & Detects: No qualification required
Warning Low for any analyte	Non-Detects: J, CLP25 Detects (> MDL but < CRQL): J, Q-2, CLP25 Detects (≥ CRQL): J, CLP25
Action Low for any analyte	**Reject all Non-Detects**: R, CLP27 Detect (> MDL but < CRQL): J, Q-2, CLP27 Detect (≥ CRQL): J, CLP27
Warning High for any analyte	Non-Detects: No qualification required Detect (> MDL but < CRQL)): J, Q-2, CLP26 Detect (≥ CRQL): J, CLP26
Action High for any analyte	Non-Detects: No qualification required **Reject all detects** for that analyte: R, CLP28

4.2.11 Data Qualifier Definitions

Region 4 applies qualifiers to the inorganic data as defined in the SOWs referenced above, and in the National Functional Guidelines with the exception of the qualifiers, B, E, and P, which are not used in Region 4 data reporting.

The following definitions provide brief explanations of the qualifiers assigned to results during the electronic data validation process. An additional set of data qualifiers is applied as needed to provide further information to the data user about data quality.

Data Qualifier	Data Qualifier Definition[5]
D-6	The sample results are confirmed by ICP-MS or other analytical technique including analysis of a reference standard (the reference standard is not a PES – a reference standard is usually a certified reference material independent of standards available through the CLP program).
J	The analyte was positively identified in the sample, but the associated numerical value is an estimated concentration based on the associated quality control data/technical criteria.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	The analyte was included in the analysis, but was not detected above the method detection limit as defined in ISM01.2.

5.0 Data Review Documentation – Computer Aided Review

Use of the Electronic EXchange and Evaluation System (EXES) allows electronic validation of the CLP inorganic data.

The results of electronic data review are utilized to assist the data review process. If examination of the electronic review results and/or PES scoring results reveals discrepancies and/or serious data quality issues, the reviewer may investigate by going back to the hard copy data package.

[5] Current list of qualifier / definition flags are located at: http://www.epa.gov/region4/sesd/oqa/rassop.html

Each EXES - NFG report is downloaded as a self expanding executable file and distributed to the data review team. The EXES - NFG report is organized by SDG.

Two (2) copies of the EXES reports should be printed for each SDG for the data reviewer. A copy should be included in the data validation documentation to submit to the Quality Assurance Section (QAS) to be retained in the project file. The second copy should be archived with the actual data package.

A Data Review Document shall be prepared to document the inorganic data package validation. The document includes the Review Summary Narrative, Time Tracker, Performance Evaluation Sample (PES) Scores from the secure SPS-Web site, a copy of the spreadsheet used for data import into the *Element* data system, and the EXES - NFG. These reporting elements are described in greater detail below, and examples are included as attachments to this SOP.

5.1 Document Contents:

5.1.1 Inorganic Data Review Summary Narrative - This narrative is in a letter format to summarize the information pertinent to the samples, analytical methods, highlights of findings, and a brief assessment of the overall data quality. Descriptions of major data quality issues and their impact on overall data quality should be presented.

5.1.2 Time tracker - This document is for recording the time line and efforts at different stages of the data review process. This form must be utilized and included in the data review documents for CLP data. Any unusual issues or factors affecting the level of effort required to complete the review in a timely manner, are discussed here and in the corresponding data validation memorandum. The time tracker should include the peer review information as part of the validation package requirements.

5.1.3 PE Score (SPS-Web) - This form is generated by the SPS-Web program to report the evaluation of the results of the performance evaluation samples (PES) associated with the data package. The “EPA” versions of this form should be included as attachments to the data validation memorandum whereas the "laboratory" version should be emailed to the EPA inorganic TO/PO.

5.1.4 Excel® Spreadsheet - The reviewed data with final assigned qualifiers attached, (if any) as they appear in Element, are included in the data review report as an Excel® spreadsheet.

5.2 Recording and Reporting of Data

Please refer to SOP "Data Processing and Final Production for Contract Laboratory Data in Element®.

5.3. Data Package Archives

The CLP data packages must be properly archived for future reference. For each data package, the form "Record Transfer Inventory" must be utilized to record the proper information pertinent to the content. All of the raw data, EXES reports, and any communication records must be included. Multiple data packages from different projects may be stored in one single box if sufficient space is available.

Data packages for one Case that are stored in multiple boxes must be clearly identified on the Record Transfer Inventory forms. An appropriate numbering system must be maintained to ensure that each box containing the data review supporting documentation, have a unique archive number.

A copy of the inventory form should be kept within the box and an additional copy filed in a centralized system. The data package boxes shall be maintained under the custody of SESD as described in the Data Package Audit and Data Entry/Validation SOP. The Data Package Inventory Form is provided in Attachment E.

6.0 References

U.S. Environmental Protection Agency, Statement of Work for Inorganic Superfund Methods, Multi-Media, Multi-Concentration, ISM01.2, January 2010.

U.S. Environment Protection Agency, Contract Laboratory Program, National Functional Guidelines for Inorganic Superfund Data Review, January 2010.