

VERIFICATION AND VALIDATION GUIDELINES

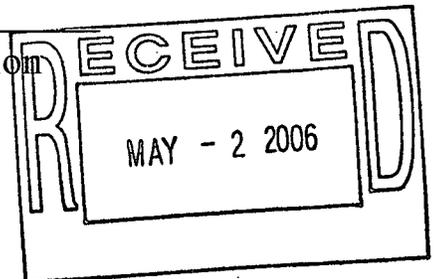
FOR

INORGANIC METALS

DA-SS05-v3

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Approved: E. A. Bronskey
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By: Roger S. Cichorz U/NU
ASD Project Lead - T130C
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V & V GUIDELINE CHANGE DESCRIPTION FORM

Instructions: Replace Version 2 with Version 3

Guideline: DA-SS01

Version: 3

Originator: Ed Brovsky

Description: Verification and Validation Guidelines for Inorganic Metals

Section No.	Change Description
N/A	New version and Effective date
Introduction	A new introduction was written to incorporate the BOA SOW rather than PSA Modules.
Entire Document	For clarity, change bars appearing on a Section Title indicate changes to the entire Section.
Entire Document	References to the BOA SOW and the RFETS BOA Implementation document GR03 & GR04, are incorporated throughout the document. References to PSA Modules were eliminated. References to Module Specific Verification and Validation (V & V) Guidelines were replaced with Analytical Specific V & V guidelines.
Data Review Checklist	All references to the Data Review Checklist and its examination were removed from the Guidelines.
Entire Document	All actions that involve Reason Codes 801, or 803 were revised to include an NCN be issued to request missing, incomplete data, or corrected data. The action requires the discontinuation of further assessment until corrected data is received and the action also requires a comment in the DQA Report identifying the request for missing or corrected data.
Section 2.3	The entire section for Sample Results was revised to include steps that meet BOA and GR03 requirements. The section was also reorganized to include data assessment steps for "Validation Only".
Sample Preparation Method	The section titled, "Sample Preparation Method," was eliminated from this document.
2.20	A section was added for TCLP assessment.

1. PURPOSE AND INTRODUCTION

This document presents those data assessment steps which are unique to Inorganic Metals Analyses. This Analytical Specific document is to be used in conjunction with DA-GR01, "General Guidelines for data Verification and Validation.

The purpose of this document is to provide guidance in the completion of Data Verification, and Data Validation activities as part of the Rocky Flats Environmental Technology Site (RFETS) Analytical Services Division Data Assessment Process as described in DA-GR01.

This version of DA-SS05 is applicable to Inorganic Metals Sample Data Packages generated under the National Basic Ordering Agreement (BOA) Statement of Work (SOW) and the Rocky Flats Environmental Technology Site (Site) BOA Implementation Requirements documents, GR03 & GR04.

2. VERIFICATION AND VALIDATION INSTRUCTIONS

The instructions contained in this section are specific to Inorganic Metals analyses. They are to be used in conjunction with the general instructions for Verification and Validation found in Analytical Services Division's General Guidelines for Verification and Validation, DA-GR01.

2.1. Chain of Custody, Holding Times, and Sample Preservation

Review Items: COC, Laboratory Sample Receiving Documentation, Cover Page Comments, Sample Case Narrative, raw data, data summary forms, and sample preparation/extraction log.

Objective: The objective is to ascertain the validity of results based on the method required holding times, sample preservation, and the continuity of sample custody.

Source: BOA Attachment 1, § 3.1.2, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Determine if the samples were properly preserved prior to laboratory sample receipt using the criteria provided in Table 1a and Table 1b.

Action 1: If samples were not acid-preserved and/or were not maintained at $4^{\circ} \pm 2^{\circ} \text{C}$ prior to receipt by the laboratory, do not qualify the sample results. However, comment and assign the reason code [703] to all applicable samples.

Item 2: Check for documentation that the sample pH was adjusted to ≤ 2 by the laboratory if an aqueous sample was not adjusted to the proper pH prior to receipt by the laboratory.

Action 2: If an aqueous sample was not adjusted to the proper pH by the laboratory, when required, issue a Non-Compliance Notification (NCN) and qualify all results as estimated [J 201].

- Item 3:** Determine if samples were properly preserved after sample receipt.
- Action 3:** If documentation specifically indicates sample preservation was not maintained after sample receipt, but prior to analysis, issue a NCN requesting a corrective action to prevent recurrence and qualify all results as estimated [J 201].
- Item 4:** Determine the actual analysis and preparation holding times by comparing the preparation and analysis dates on the raw data and the sample collection date on the COC. If the actual holding time is greater than the maximum allowable holding time per Table 1a or Table 1b, qualify all results according to the following guidelines:
- Action 4a:** Qualify all positive results when the actual holding time was greater than the maximum holding time as follows:
- If the hold time violation is attributed to the lab issue a NCN and estimate [J 101] all applicable data.
 - If the hold-time violation is not attributed to the laboratory, estimate [J 701] all applicable data.
- Action 4b:** Qualify all non-detects as rejected (R) and detects as estimated (J) when the actual holding time was greater than two times the maximum holding time as follows:
- If the hold time violation is attributed to the lab, issue a NCN and assign qualifier [R/J 102].
 - If the hold-time violation is not attributed to the laboratory, assign qualifier [R/J 702].
- Action 4c:** Qualify all non-detects when the actual holding time was greater than the maximum holding time but less than two times the maximum holding time as follows:
- If the hold time violation is attributed to the lab, issue a NCN and assign the qualifier [UJ 101].
 - If the hold-time violation is not attributed to the laboratory, assign qualifier [UJ 701].

Table 1a HOLDING TIME AND PRESERVATION CRITERIA

Analyte	Holding Time (maximum)	Preservation Non-Aqueous Matrix	Preservation Aqueous Matrix
Mercury	28 days	Storage at 4°C	pH <2 w/HNO ₃
Metals other than Hg	180 days	Storage at 4°C	pH <2 w/HNO ₃

Table 1b TCLP EXTRACT HOLDING TIME AND PRESERVATION

Holding Time (Days)		Preservation	
TCLP Extraction	Extract Analytical	Non-Aqueous Matrix	Aqueous Matrix
Mercury 28 Other Metals 180	Mercury 28 Other Metals 180	Storage at 4°C	HNO ₃ to < pH 2 Storage at 4°C

2.2. Sample Data Package Narrative Requirements

Review Item: Sample Case Narrative

Objective: Review the narrative for compliance to requirements and for information useful to data assessment.

Source: GR03 § 3.2, BOA Attachment 1, § 3.1.6.2

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that the SDP Narrative is present and includes the following as applicable:

- Procedures and/or Standard Method reference for preparation and analysis.
- Descriptions of significant technical difficulties encountered in preparing and analyzing the samples.
- Justification of all dilutions.
- Explanations of any QC deficiencies, missed holding times, or inability to achieve the required detection limits (RDLs).
- Reasons for reanalysis, reanalysis Analytical Batch Identifications Numbers, and a synopsis of the reanalysis Analytical Batch QC Assessment.
- Explanations and descriptions of all deviations from routine protocols, including deviations from approved standard operating procedures (SOPs), detection limit modifications, etc. If it was necessary to contact the CTR for instructions due to the nature of the deviation, the laboratory shall document those instructions in the narrative.

Action 1: If any of the above items are non-compliant, do not qualify the results, comment and include the reason codes [227] and/or [805] as appropriate. Use professional judgement to determine if the issuance of a NCN is warranted.

2.3. Sample Results

Review Items: Forms 1 or Equivalent, Form 5A or equivalent, Form 6 or equivalent, and Form 9 or equivalent.

Objective: To confirm that sample results and qualifiers are correctly entered on the Form 1.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 1A is present for each sample in the Report Identification Number (RIN).

Action 1: If forms are missing, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that all applicable Form 5A's, Form 6's, and Form 9's or their equivalents are present.

Action 2: If forms are missing, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Check that one and only one result is reported on Forms 1 for each requested analyte.

Action 3: If more than one result is reported and neither is identified as "Do Not Use data", issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 4 Verify the following are compliant, complete, and without errors:

- Check that results for samples are reported in the same units as those used for the Required Detection Limits (RDL) provided in Attachment K to BOA Attachment 1 or in GR03.
- Check that results are reported to the correct number of significant figures. (Note: The concentration result shall be reported to 2 significant figures if the result is < 10; to 3 significant figures if the value is ≥ 10 .)
- Check that Form 1 results for all samples associated with a sample flagged on Forms 5A, 6, and 9 are flagged with the relevant qualifiers by checking the QC Forms 5A, 6, and 9 for Q qualifiers to be assigned. Check that Q qualifiers are entered for all associated samples or samples processed in the same analytical batch as the QC samples receiving a qualifier.

Action 4a: Noncompliant items, omissions, or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and

assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 4b: For other noncompliant items, omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: *The following item applies to validation only:*

Item 5: Verify the following are compliant, complete, and without errors:

- Check that results for detected analytes are factored by all dilutions. Perform this check for at least three analytes per sample. At least one analyte checked must be determined by CVAA or GFAAS, if those techniques are utilized for the SDP.
- Check that results for non-detected analytes are reported at the IDL and factored for any dilutions.
- Check that C and M qualifiers are entered correctly for each analyte.
- Check that the detection limit of each diluted sample (IDL * dilution factor) for a non-detected analyte is \leq the specified RDL provided in Attachment K to BOA Attachment 1 or in GR03.

Action 5a: Noncompliant items, omissions, or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 5b: For other noncompliant items, omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 6: Recalculate from the raw data sample results for 3 elements and verify that the recalculated values agree with the values reported on each Form 1.

Action 6: If errors are identified, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.4. Calibration Verification (Summary Form 2A)

Review Items: Form 2 or equivalent, Form 8 or equivalent, preparation logs, standard logs, instrument logs, instrument printouts, and raw data.

Objective: To determine that all analytical results were obtained from instrumentation that was in calibration according to the analytical method. Initial calibration verification (ICV) is performed to provide assurance of the accuracy of the calibration standards. Sources of standards used for calibration and ICV must be independent. If these

sources are truly independent and ICV results meet the evaluation criteria, then the probability of gross calibration error is small. The analysis of continuing calibration verification standards (CCV) establishes that the upper concentration portion of the initial calibration is still valid by checking the performance of the instrument on a continual basis.

Sources: BOA Attachment 1, § 3.2.3; Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 2As are present.

Action 1: If forms are missing, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that ICV and CCV results are reported for all requested analytes.

Action 2: If ICV or CCV results are not reported for an element, issue a NCN and qualify all results for the element as rejected [R 104].

Item 3: Verify that all ICV percent recoveries (%R) are between 80 and 120% for mercury and 90-110% for all other metals.

Action 3: If the ICV %R for any element falls outside the acceptance windows, issue a NCN and qualify all data from the analytical run for that element according to the following guidelines:

- If the ICV %R falls outside the acceptance windows but within the ranges of 75-89% or 111-125% (Hg, 65-79% or 121-135%), qualify all results greater than the IDL as estimated [J 104].
- If the ICV %R falls outside the acceptance windows but within the ranges of 75-89% (Hg, 65-79%), qualify all results less than the IDL as estimated [J 104].
- If the ICV %R is <75% (Hg, <65%) qualify all results as rejected [R 104].
- If the ICV is >125% (Hg >135%) qualify all results greater than the IDL as rejected [R 104]. Do not qualify results less than the IDL. However, assign the reason code [104] to all applicable data.

Item 4: If the CCV %R for any element fell outside the acceptance windows of 90-110% (Hg 80-120%), determine if a reanalysis of the affected samples, bracketed by CCV analyses with compliant %R values, was performed.

Action 4: If a reanalysis meeting the above CCV %R acceptance criteria was not performed, issue a NCN and qualify data for the affected element(s) according to the following guidelines:

- If the CCV %R falls outside the acceptance windows but within the ranges of 75-89% or 111-125% (Hg, 65-79% or 121-135%), qualify all bracketed results greater than the IDL as estimated [J 104].
- If the CCV %R falls outside the acceptance windows but within the ranges of 111-125% (Hg, 121-135%), results less than the IDL are acceptable. However, assign reason code [104].
- If the CCV %R falls outside the acceptance windows but within the ranges of 75-89% (Hg, 65-79%), qualify all bracketed results less than the IDL as estimated [J 104].
- If the CCV %R is <75% (Hg, <65%) qualify all bracketed results as Rejected [R 104].
- If the CCV is >125% (Hg >135%) qualify all bracketed results greater than the IDL as Rejected [R 104]. Do not qualify results less than the IDL. However, assign the reason code [104]

Note: The ICV and CCV shall be analyzed in the same fashion as an actual sample. Operations such as the number of replicate analysis, the number and duration of the instrument rinses, etc. affect the measured ICV or CCV result and are not to be applied to the ICV or CCV in a greater extent than they are applied to the associated analytical samples.

Evaluation: *The following item applies to validation only:*

Item 5: Verify that an ICV was analyzed at the beginning of each analytical sequence or after the calibration standards and before the analysis of site samples and the Initial Calibration Blank (ICB).

Action 5: If an ICV was not analyzed or site samples were analyzed before the ICV, issue a NCN and qualify all samples analyzed before the first calibration verification sample as rejected [R 129].

Item 6: Verify that a CCV standard was analyzed after the last analytical sample.

Action 6: If a CCV was not analyzed after the last site samples were analyzed, issue a Non-Compliance-Notification and qualify all samples analyzed after the last acceptable CCV as rejected [R 129].

Item 7: Verify that no more than 10 solutions were analyzed between the analyses of the ICV and the first CCV (include all solutions analyzed except ICBs, and CCVs). Check that no more than 10 solutions were analyzed between any two consecutive analyses of the CCV (include all solutions analyzed except CCBs and CCVs).

Action 7: If more than 10 solutions were analyzed between any calibration verification standard, comment and assign the reason code [129] to all samples analyzed within this calibration verification bracket.

- Item 8:** Check the time between calibration verifications is not more than two hours
- Action 8:* If more than two hours elapsed between consecutive calibration verifications, comment and assign the following reason code [129].
- Item 9:** Check that the ICV and CCV results and subsequent %R values for 3 elements from the instrument raw data compare with those results and subsequent %R results on Form 2A. If one or more of the raw data results do not agree with results reported on Form 2A to two significant figures; and the %R values do not agree to within 0.1%, check all reported ICV and CCV results and %R values against the raw data.
- Action 9:* If the raw data and reported data do not agree, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Item 10:** Recalculate from the raw data the ICV and CCV %R values for 3 elements and verify that the recalculated values agree with the values reported on Form 2A.
- Action 10:* If the results do not agree, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Item 11:** Verify that the minimum number of standards were used to calibrate the instrument upon use according to the appropriate base method specified in the Line Item Code (LIC).
- Action 11:* If the minimum number of standards were not used or the instrument was not calibrated at the appropriate frequency, qualify all sample results as rejected [R 106].
- Item 12:** Verify that the correlation coefficient for all analyses other than ICP was greater than 0.995.
- Action 12:* If the correlation coefficient is less than 0.995, qualify all associated results as estimated [J 103].

2.5. CRDL Check Sample (Summary Form 2B)

The CRDL check sample is only evaluated for ICP analysis. However, the check sample is analyzed for all methods of analysis.

- Review Items:** Form 2B or equivalent
- Objective:** To determine the extent to which the laboratory could demonstrate the validity of the calibration at levels near the CRDL or RDL of the method and verify the laboratory's interelement correction factors
- Sources:** Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both validation and verification:*

Item 1: Check that Form 2Bs are present.

Action 1: If forms are missing, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify that the CRDL %R values were within the limits of 80 to 120% for ICP analyses.

Action 2: Qualify sample results associated with non-compliant CRDL %R values as follows:

- If the CRDL result is within 40-79% or **greater than** 120% , qualify detected results less than 3 times the RDL as estimated [J 105] and issue a Non-Compliance Notification for the low recoveries.
- If the CRDL result is 40-79%, qualify results **less than** the IDL as estimated [J 105] and issue a Non-Compliance Notification for the low recoveries.
- If the CRDL result is less than 40%, qualify detected results less than 3 times the RDL as estimated [J 105] and results less than the IDL as rejected [R 105]. Issue a Non-Compliance Notification for the unacceptable recoveries.

Evaluation: *The following items apply to validation only:*

Item 3: Verify that the CRDL Standard was analyzed at the proper concentration (2 times the CRDL or IDL, whichever is greater) for each analyte.

Action 3: If the proper concentrations were not used, qualify all affected sample results for the applicable elements as estimated [J 105].

Item 4: Verify that the CRDL standard was analyzed at the beginning and end of each ICP analytical sequence (or every 8 hours) and at the beginning of each GFAA and CVAA analytical sequence.

Action 4a: Qualify all ICP results as estimated [J 129] if the CRDL frequency requirement was not met.

Action 4b: If the CRDL standard was not analyzed at the beginning of the GFAA and CVAA analytical sequence, comment and assign [129] to all applicable data.

2.6. Blanks (Summary Form 3)

- Review Items:** Form 3 or equivalent, Form 10 or equivalent, preparation logs, standard logs, instrument logs, instrument printouts, and raw data.
- Objective:** To determine the existence and magnitude of contamination resulting from preparation and analysis activities. Blanks may be assessed to establish potential false positive results attributable to variances in instrument operating conditions or due to contamination introduced into the analytical system.
- Sources:** Attachment I to BOA Attachment 1, and Base Method
- Evaluation:** *The following items apply to both verification and validation:*
- Item 1:** Check that Form 3s are present.
- Action 1:** If forms are missing, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Item 2:** Check that ICB, CCB and PB results are reported for all requested analytes.
- If any ICB, CCB, and PB results are not reported issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Item 3:** Check that Form 3 results are reported to the IDLs listed on Form 10.
- Action 3:** If the Form 3 results are not reported to the IDLs listed on the Form 10, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Items 4:** Verify that the absolute value of any ICB, CCB, or PB result is not greater than the RDL for the element. Check for the reanalysis of all samples associated with unacceptable blanks, if applicable.
- Action 4a:** All sample results for an element which are greater than 10 times the absolute value of the blank result may be accepted without qualification.
- Action 4b:** For other results with non-compliant blanks (bracketing for calibration blanks) qualify data according to the following guidelines:
- If the blank result is greater than the RDL,
 - ◊ Qualify all sample results with non-detects as accepted without qualification. Do not change validation qualifier but Comment and assign reason code [159].
 - ◊ Qualify all positive results less than 5X the absolute value of the blank result as Rejected, with qualifier [R 159].

- ◇ Qualify all positive results less than $\leq 10X$ and $\geq 5X$ of the absolute value of the blank result as estimated, with qualifier **[J 159]**.
- If the blank result is below the negative RDL,
 - ◇ Qualify all sample results less than the IDL and all detected results less than $5X$ the absolute value of the blank result as Rejected with the reason code **[R 159]**.
- If the blank result is between the negative IDL and the negative RDL:
 - ◇ Qualify all sample results with non-detects as estimated **[J 107]**.
 - ◇ Qualify all positive results less than $5X$ the absolute value of the blank result as estimated **[J 107]**.
- If the blank result is greater than the IDL but less than the RDL:
 - ◇ All sample results less than the IDL are accepted without qualification.
 - ◇ Qualify all positive results less than $5X$ the blank result as estimated **[UJ 107]** to indicate estimated results with an elevated detection limit.

Evaluation: *The following items apply to validation only:*

Item 5: Obtain blank results from the instrument raw data for at least three elements with blank results less than the IDL, below the negative IDL, and greater than the IDL. Compare the results to those reported on Form 3.

Action 5: If one or more of the raw data results do not agree with results reported on Form 3 to an appropriate number of significant figures, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 6: Check that the initial calibration blank (ICB) is analyzed after the analytical standards but not before analysis of the ICV, during the initial calibration of the instrument.

Action 6: If the ICB is not analyzed in the proper order, comment and assign the reason code **[129]**.

Item 7: Check that the final CCB standard was analyzed after the last CCV was analyzed.

Action 7: If the final CCB was not analyzed after the final CCV, comment and assign the reason code **[129]**.

Item 8: Check that no more than 10 solutions were analyzed between any two consecutive analyses of CCB (include all solutions analyzed except ICVs/ICBs and CCVs/CCBs).

Action 8: If more than 10 solutions were analyzed between calibration verification samples, comment and assign the reason code **[129]** to all affected samples.

- Item 9:** Check that no more than two hours elapsed between consecutive analyses of calibration verification blanks (ICBs or CCBs).
- Action 9:** If more than 2 hours have elapsed between consecutive calibration blank analyses, comment and assign the reason code [129] to all affected samples.

Notes to the Data Assessor Regarding Blanks

- Note 1:** The analysis should be terminated when the absolute value of any blank result exceeds the RDL.
- Note 2:** Calibration blanks are to be analyzed in the same fashion as an actual sample. Operations such as the number of replicate analysis, the number and duration of the instrument rinses, affect the measured blank result and are not to be applied to the blank in a greater extent than they are applied to the associated analytical samples.
- Note 3:** Preparation Blank results are to be in the same units as the sample under all circumstances. However, results with a soil matrix will be corrected for the percent moisture in the sample. In addition, the ICB and CCB analyses are always reported in aqueous units. Therefore, when determining potential blank qualification for soil samples, the data assessor may find it easier to work from the raw data when applying the 5X criteria to soil sample data, or calibration blank data.

2.7. Interference Check Sample (Summary Forms 4A and 4B)

- Review Items:** Form 4A or equivalent, Form 4B or equivalent.
- Objective:** To assess the impact of the contract laboratory ICPES interelement interferences on sample results with high levels of interferent elements.
- Sources:** Attachment I to BOA Attachment 1, and Base Method
- Evaluation:** *The following items apply to both verification and validation:*
- Item 1:** Check that Form 4A and 4B are present for ICP analyses.
- Action 1:** If forms are missing, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Item 2:** Check that ICSA and ICSAB results are reported for every requested analyte determined by ICPES.
- Action 2:** For all elements in a sample without associated ICSA or ICSAB values and the sample results for aluminum, magnesium, iron, or calcium that exceed the ICSA found value, comment and qualify as estimated [J 229].
- Item 3:** Verify that an ICSA and ICSAB are analyzed at the beginning and end of each analytical sequence (or every 8 hours, whichever is more frequent).

- If the ICSA and ICSAB are not analyzed in the correct sequence and frequency, comment and assign reason code [250] to all results except aluminum, iron, calcium, and magnesium.

Evaluation: *The following items apply to validation only:*

Item 4: For samples that have aluminum, calcium, iron, or magnesium values that exceed the found value in the ICSA solution, verify the ICSAB %R for each element on Form 4A is within 80 to 120%.

Action 4: Qualify noncompliant data as follows:

- If the ICSAB recovery for an element is greater than 120% and the sample results for that element are less than the IDL, no qualification is required.
- If the ICSAB recovery for an element is greater than 120% and the sample results for that element are greater than the IDL, qualify the data as estimated [J 109].
- If the ICSAB recovery for an element falls between 50 and 79% and the sample results for that element are greater than the IDL, qualify the data as estimated [J 109].
- If sample results are less than the IDL and the ICSAB recovery for that analyte falls within the range of 50 - 79%, qualify the data for these samples as Estimated [J 109].
- If ICSAB recovery for an element is less than 50%, qualify all associated data as Rejected [R 109].

Item 5: Evaluate the ICSA results for all requested elements, **except** Al, Ca, Fe, and Mg, to verify that the absolute values are not greater than 2X IDL for those requested elements which are not present in the ICSA solution.

Action 5: Qualify all data for each element which are not bracketed by acceptable ICSA results as follows:

- If the ICSA results are $>(2*IDL)$ and the sample results are $< IDL$, no qualification is necessary.
- If the ICSA results are $>(2*IDL)$ and the sample results are $> IDL$ and $<(10)(\text{the ICSA reading})$, comment and qualify the results as estimated [J 109].
- If the ICSA results are $\leq(2*-IDL)$, comment and qualify all non-detected results and all detected sample results which are $\leq(10)(\text{the ICSA reading})$ as estimated [J 109].

Item 6: Check the raw data (ICP printouts, strip charts, bench sheets) to verify that the %R results on Form 4 were accurately transcribed.

Action 6: If the raw data and reported data do not agree, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 7: Recalculate from the raw data at least one ICSAB analyte and one interferent percent recovery (%R) and verify that the reported value on the Form 4 agrees within 0.1%.

Action 7: If the results do not agree to within 0.1%, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.8. Matrix Spike Analysis (Summary Forms 5A)

Review Items: Form 5 or equivalent and raw data.

Objective: To assess the impact of matrix effects on the sample analytical results. Analysis of spiked samples provides information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 5As are present.

Action 1: If forms are missing, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that at least one Form 5A is present for each method, matrix, waste type, and analytical batch.

Action 2a: If a matrix spike was not analyzed at the proper frequency, issue a NCN and qualify all samples associated with the missing matrix spike as estimated [J 168].

Action 2b: If no matrix spike was analyzed, regardless of frequency, issue a NCN for the missing QC and qualify all samples associated with the missing matrix spike as estimated [J 230].

Item 3: Check that control limits (75-125%) are correctly assigned.

Action 3: If the control limits are not assigned correctly, comment and assign the reason code [232]. Continue to evaluate spike results using the correct limits

Item 4: Check that the matrix spike %R is reported for all requested analytes.

Action 4: If spike results and subsequent %R values are not reported for an element, initiate a NCN and qualify all associated results as estimate [J 230].

Item 5: Check that an "N Flag" is present in the Q column for each element with the %R outside of the control limit listed on Form 5A or the correct limit, if the assigned control limit was in error. Spike recovery criteria do not apply for the following:

- Calcium, magnesium, potassium, sodium, iron and aluminum in soil samples.

- Calcium, magnesium, potassium, and sodium in water samples.
- When the sample result for an element is >4X (the spike level).

Action 5: If an "N Flag" is not present when required, comment and assign reason code [804].

Item 6: Evaluate sample spike recovery results.

Action 6: Qualify sample data associated with non-compliant spike recoveries as follows:

- If the spike recovery is >125% and the reported sample results are < IDL, no data qualification is necessary.
- If the spike recovery is >125% and the sample result is >(IDL), the data are estimated [J 112].
- If the spike recovery is within the range of 30-74%, all sample results are estimated [J 112].
- If the spike recovery is less than 30%, qualify all detected results as estimated [J 113] and all non-detected results as rejected [R 113].

Evaluation: *The following items apply to validation only:*

Item 7: Check the raw data (ICP printouts, strip charts, bench sheets) to verify that the results and subsequent %R results on Form 5A were accurately transcribed to within 2 significant figures for the results and 0.1% for the %R values.

Action 7: If the raw data and reported data do not agree, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 8: Calculate from the raw data one or more of the spiked sample percent recoveries (%R) using the following equation:

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

Where:

- SSR = Spiked Sample Result
- SR = Sample Result (see note)
- SA = Spike Added

Note: When the sample concentration is less than the instrument detection level (IDL), use SR=0 only for the purposes of calculating the %R.

Action 8: If the calculated value does not agree with the laboratory reported value on Form 5A to within 0.1%, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.9. Post Digestion Spike Analysis (Summary Form 5B)

Review Items: Form 5B or equivalent.

Objective: To evaluate the potential sources of poor matrix spike recoveries determined with a post digestion spike. Data are not qualified solely on the basis of post digestion spike recovery data. Samples spiked after preparation or digestion steps (post-digestion spikes) provide information about the effect of each sample matrix on the measurement methodology. Post-digestion spikes are performed when pre-digestion spikes do not meet expected recovery levels. If both pre- and post digestion spike recovery are similar, then matrix effects are suspected.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 5B results are present for each pre-digestion matrix spike element which did not meet the matrix spike recovery criteria.

Action 1: If Form 5B results are not available, comment and assign the reason code [802] to all results subject to post digestion spike analysis.

Item 2: Verify that a discussion of the Post Digestion Spike Results are given in the Narrative. This discussion should address the root cause of pre-digestion matrix spike outliers.

Action 2: If information is not available in the Narrative, comment and assign the reason code [805] to all results subject to post digestion spike analysis.

Note: Post Digestion Spike Results are not to be used to qualify sample results.

Evaluation: *The following items apply to validation only:*

Item 3: Check the raw data (ICP printouts, strip charts, bench sheets) to verify that the results and subsequent %R results on Form 5B were accurately transcribed to within 2 significant figures for the results and 0.1% for the %R values.

Action 3: If the raw data and reported data do not agree, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 4: Calculate from the raw data one or more of the spiked sample percent recoveries (%R) using the following equation:

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

Where:

SSR = Spiked Sample Result

SR = Sample Result (see note)

SA = Spike Added

Note: When the sample concentration is less than the instrument detection level (IDL), use SR=0 only for the purposes of calculating the %R.

Action 4: If the calculated value does not agree with the laboratory reported value on Form 5B to within 0.1, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.10. Laboratory Duplicate Analysis (Summary Forms 6)

Review Items: Form 6 or equivalent and raw data.

Objective: To verify acceptable precision of sample results for the sample matrix, laboratory preparation, and analysis procedure. Duplicate sample determinations are used to measure variability due to a combination of factors including laboratory precision, method precision, and sample homogeneity.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Replicate Spiked Sample Verses Duplicate Sample

If a replicate spiked sample is analyzed instead of a duplicate sample then the spike and spiked replicate are reported on Form 6. Use the same evaluation rules, substituting measured spiked sample values for the sample concentrations.

Item 1: Check that Form 6s are present.

Action 1: If the Form 6s are not available, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify that at least one duplicate analysis was performed for each matrix, waste type, and analytical batch.

Action 2a: If a duplicate analysis was not performed at the appropriate frequency, issue a NCN and qualify all associated samples as estimated [J 168].

Action 2b: If an element for a duplicate analysis was not analyzed, issue a NCN and qualify the element for all associated samples as estimated [J 230].

Item 3: Verify that the Relative Percent Difference (RPD) control limits for each analyte are assigned as follows:

- If the original and duplicate sample values are <(IDL), the control limit column was left blank. (**Note:** No RPD should have been

entered on the Form 6 or the RPD column should have been left blank for this analyte.)

- If the original and duplicate sample values are $\geq(5)(RDL)$, the "Control Limit" was left blank and the RPD was entered in the Form 6 RPD column
- Note: The aqueous control limit is 20% RPD and the soil control limit is 35% RPD.
- If either one or both of the original and duplicate values is $<(5)(RDL)$ and $\geq(IDL)$, the RDL was entered as the limit in the "Control Limit" column of Form 6.
- Note: The RPD column should have not been left blank for this analyte.

Action 3: If the control limits are not assigned correctly, comment and assign the reason code [232]. Continue to evaluate duplicate results using the correct limits.

Item 4: Check that the original and duplicate results, and subsequent RPD when required, are reported for each requested element.

Action 4a: If duplicate results and subsequent RPD are not reported for a required element, issue a NCN and qualify all associated results as estimated [J 230].

Action 4b: If duplicate results were reported but the subsequent RPD was not entered on the Form 6 when required, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 5: Verify that a "* Flag" is present in the Q column if the RPD or the difference between the original and duplicate results is outside of the appropriate control limit. Use the correct control limit, if the assigned control limit was in error.

Action 5a: If an "* Flag" is not present when required, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 5b: If the original and duplicate result are $>(5)(RDL)$ and the RPD is greater than 20% for water matrix or greater than 35% for the soil matrix, qualify all results for the element as estimated [J 111].

Action 5c: If the original or duplicate result is $<(5)(RDL)$ and the difference between the duplicate and original sample is greater than the RDL for water samples and greater than 2X the RDL for soil matrix, qualify all results for the element as estimated [J 111].

Evaluation: *The following items apply to validation only:*

Item 6: Check the raw data (ICP printouts, strip charts, bench sheets) to verify that the original and duplicate results and subsequent RPD results on Form 6 were accurately transcribed to within 2 significant figures for the results and 0.1% for the RPD value.

Action 6: If the raw data and reported data do not agree, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 7: Calculate from the raw data one or more of the RPD values using the following equation:

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

Where:

RPD = Relative Percent Difference

S = First Sample Value (original sample)

D = Second Sample Value (duplicate)

Action 7: If the calculated value does not agree with the laboratory reported value on Form 6 to within 0.1%, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.11. Laboratory Control Sample Analysis (Summary Forms 7)

Review Items: Form 7 or equivalent and raw data.

Objective: To determine the overall laboratory performance of each step from preparation through analysis.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 7s are present.

Action 1: If the Form 7s are not present, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that one Form 7 or LCS is included for each analytical batch and matrix.

Note: LCS material may not be present for some matrices or waste types. The Case Narrative must explain that an equivalent matrix was used with CTR approval.

Action 2a: If an LCS was not present or an LCS with the incorrect matrix was used, issue a NCN and qualify all results as estimated [J 230].

Action 2b: If an LCS was present but not at the appropriate frequency, comment and qualify all results as estimated [J 168].

Item 3: Check that LCS results are reported for all requested elements and percent recoveries are within the control limits.

Action 3: If an LCS was not reported for an element, comment and qualify all results for that element as estimated [J 230].

Aqueous LCS Recoveries

Item 4: Determine if aqueous LCS recoveries meet acceptance criteria (except antimony and silver).

Action 4: Non-compliant data shall be qualified as follows:

- If the LCS recovery for any analyte falls within the range of 50% - 79% or >120%, qualify results > IDL as estimated [J 110].
- If the results are < IDL and the LCS recovery is greater than 120%, the data are valid.
- If the results are < IDL and the LCS recovery falls within the range of 50-79%, qualify the data for the affected analytes as estimated [J 110].
- If LCS recovery results are <50%, qualify the data for these samples as Rejected [R 110].

Solid LCS Recoveries

Item 5: Determine if solid LCS recoveries meet acceptance criteria and qualify non-compliant results as follows:

Action 5: Non-compliant data shall be qualified as follows:

- If the solid LCS recovery for any analyte is greater than the certificate control limits, qualify all sample results > IDL as estimated [J 110].
- If the LCS results are higher than the control limits and the sample results are < IDL, no qualification of data is necessary.
- If the LCS recovery for any analyte is lower than the control limits, then qualify all sample results as estimated [J 110].

Evaluation: *The following items apply to validation only:*

Item 6: Check the raw data (ICP printouts, strip charts, bench sheets) to verify that the reported percent recoveries (%R) on Form 7 were accurately transcribed to within 0.1%.

Action 6: If the raw data and reported data do not agree, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 7: Calculate from the raw data one or more of the reported recoveries (%R) using the following equation:

$$\%R = (\text{Found LCS Value} / \text{Expected LCS Value})100$$

Where:

Found LCS Value = Actual LCS result from laboratory analysis

Expected LCS Value = Expected LCS result based on certificate of analysis or equivalent record

Action 7: If the calculated value does not agree with the laboratory reported value to within 0.1%, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.12. Furnace AA Quality Assurance (Summary Forms 8)

Review Items: Form 8 or equivalent, GFAA raw data.

Objective: To ensure that all AA analyses were performed using the appropriate quality control measures including duplicate injections, analytical spikes, and Method of Standard Additions (MSA) when required.

Sources Attachment I to BOA Attachment I, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Verify that a Form 8 is present for each sample with results marked S or + in the Form 1 Q column. (Note: GFAA quality control procedures apply only to elements that are analyzed using the GFAA instrumentation.)

Action 1: If a Form 8 is not present when required, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: *The following items apply to validation only:*

Item 2: Verify that 2 replicate injections were performed and the readings agree within 20% Relative Standard Deviation (RSD) for sample concentrations greater than the RDL.

Action 2a: If the replicate injection RSD is greater than 20% and the sample has not been reanalyzed once as required, comment and qualify results greater than the RDL as estimated [J 130].

Action 2b: If the RSD of the reanalyzed sample is greater than 20% qualify the sample results as estimated [J 130].

Item 3: Determine that the post-digestion spike was run immediately after the sample and the spike concentration was 2x the RDL.

Action 3: If the post-digestion spike was not run immediately after the sample or the

spike concentration was not 2x the RDL, issue a Non-Compliance Notification and assign the reason code [114].

Item 4: Determine if there are samples with an absorbance or concentration that is **less than** 50% of the analytical spike value.

Action 4a: Qualify samples with an absorbance or concentration that is less than 50% of the analytical spike value as follows:

- If the analytical spike recovery is not within 85-115% and the sample result is greater than the IDL, then qualify the sample as estimated [J 216].
- If the analytical spike recovery is between 10-84% and the sample result is a non-detect, then the result is estimated [UJ 216].
- If the analytical spike recovery is less than 10% and the result is a non-detect, then the result is rejected [R 217].
- If the analytical spike recovery is greater than 115% and the result is a non-detect, comment and assign reason code [216].

Item 5: Determine if there are samples with an absorbance or concentration that is greater than 50% of the spike concentration and the analytical spike recovery is less than 40%.

Action 5: Qualify samples with an absorbance or concentration that is **greater than** 50% of the spike concentration and the analytical spike recovery is less than 40% as follows:

- Qualify all detected results as estimated [J 216].
- If the analytical spike recovery is between 10-39% and the result is a non-detect, then the result is estimated [UJ 216].
- If the analytical spike recovery is less than 10% and the result is a non-detect, then the result is rejected [R 217].

Item 6: Determine that the Method of Standard Additions (MSA) was completed for the following condition: a sample with an absorbance or concentration that is greater than 50% of the spike concentration, and the analytical spike recovery is between 40-84% or greater than 115%.

Action 6: If a Method of MSA was not completed as required, or did not meet the indicated criteria, qualify the sample results as follows:

- If the MSA was required and not performed or not reported, the result for the sample is qualified as estimated [J 115].
- If the MSA was performed using the incorrect spike concentrations or the incorrect number of spikes (3), then the result for the sample is qualified as estimated [J 115].
- If the MSA correlation coefficient is less than 0.995 then the result for the sample is estimated [J 116].

2.13. ICP Serial Dilution (Summary Forms 9)

Review Items: Form 9 or equivalent

Objective: To determine the extent and impact of physical and chemical interferences on the sample results.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 9s (serial dilution results) are present if any reported results were determined by ICPEs.

Action 1: If the results are not present when required, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify that at least one serial dilution was reported for each analytical batch and matrix analyzed by ICPEs.

Action 2a: If a serial dilution was not analyzed with an ICP analytical batch, qualify all detected results as estimated [J 230].

Action 2b: If a serial dilution was analyzed but not analyzed at the required frequency with an ICP analytical batch, qualify all detected results as estimated [J 168].

Item 3: Check that the original and serial dilution results, and subsequent %D when required, are reported for each requested element. The %D is required whenever the original sample concentration is > 50(IDL).

Action 3a: If %D results are not reported for a required element, issue a Non-Compliance Notification and qualify all associated results as estimated [J 230].

Action 3b: If serial results were reported but the subsequent %D was not entered on the Form 9 when required, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 4: Check that an "E Flag" is present if the sample result is > 50(IDL) and the %D is > 10%.

Action 4a: If an "E Flag" is not present as required, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 4b: If the sample result is > 50(IDL) and the %D is > 10%, comment and qualify all results for the affected element as estimated [J 117].

Evaluation: *The following items apply to validation only:*

Item 5: Check the raw data (ICP printouts, strip charts, bench sheets) to verify that the %D results on Form 9 were accurately transcribed to within 0.1%.

Action 5: If the raw data and reported data do not agree, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 6: Calculate from the raw data one or more of the %D values using the following equation:

$$\%D = \frac{|I - SD|}{I} (100)$$

Where:

%D = Percent Difference
I = Initial Sample Value
SD = Factored Serial Dilution Result

Action 6: Verify that the results have been correctly reported on Form 9. If the calculated results do not agree to within 0.1% of the results reported on Form 9, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.14. Instrument Detection Limit (Summary Form 10)

Review Items: Form 10 or equivalent

Objective: To check that the QC Summary was prepared against the appropriate set of RDLs.

Sources: Attachment I to BOA Attachment 1, GR04 § 4, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Verify that a Form 10 (Instrument Detection Limits) is present for each type of instrumentation used and IDL values are present for each element of the requested Line Item Code.

Action 1: If an IDL is not provided, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify that the IDL for each element is equal to or less than the Line Item Code specified RDL.

Action 2: If the IDL is greater than the RDL, issue a NCN and qualify all non-detected results as rejected [R 213].

Evaluation: *The following item applies to validation only:*

Item 3: Verify that the effective date for IDLs is within 3 months of the run date.

Action 3: No qualification is taken if the IDL effective date has exceeded 3 months. However, comment and assign reason code [214] to all affected results.

2.15. Interelement Correction Factors and Linear Range Studies (Summary Forms 11 and 12A)

Review Items: Form 11 or equivalent, and Form 12 or equivalent

Objective: To determine that the laboratory has provided current Interelement Correction Factor studies and Linear Range Studies for ICP analyses in the Sample Data Package.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 11s are present for each ICPES instrument used to report data.

Action 1: If the Form 11 is not included, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that Form 12 is present for each ICPES instrument used to report data.

Action 2: If the Form 12 is not included, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: *The following item applies to validation only:*

Item 3: Verify that all uncorrected results (for dilutions) are within the linear range of the instrument.

Action 3: If a reported result is above the linear range of the instrument, qualify all applicable results as estimated [J 155].

2.16. Preparation Logs (Summary Forms 13)

Review Items: Form 13 or equivalent

Objective: Confirm completeness and accuracy of the preparation summary form.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 13 is present for each analytical batch reported, if preparation is applicable.

Action 1: If the Form 13 is required and not present, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: *The following items apply to validation only:*

Item 2: Check that each reported sample is listed on Form 13. If a sample is not listed on the Form 13, check the raw data for preparation in an acceptable preparation batch.

Action 2a: If a sample was not prepared in an acceptable preparation batch, issue a NCN and qualify all results for the sample as rejected [R 207].

Action 2b: If a sample was prepared in an acceptable preparation batch but not included on a Form 13, comment and assign reason code [804].

Item 3: Check that samples, duplicates, spikes, preparation blanks, and control samples are identified according to the following criteria:

- Samples are identified by either the Site identifier or the lab identifier.
- Lab duplicates are identified by the sample identifier with a "D" appended.
- Preparation blanks are identified by PB plus a designator that ties the PB to the digestion batch
- Lab control samples are identified by LCS plus a designator that ties the control sample to the digestion batch
- Matrix spikes are identified by the sample identifier with a "MS" appended.

Action 3: If any of the above criteria are not in compliance, comment and assign the reason code [804].

Item 4: Check that samples are clearly linked to an associated spiked sample, lab duplicate sample, lab control sample, and preparation blank by comparing all preparation dates and sample identifiers reported on Form 13 with raw data preparation bench sheets.

Action 4: If samples cannot be clearly linked to the identified QC samples, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 5: Determine if Batch QC samples meet the following requirements:

- At least one set of duplicates, spikes, PBs, and LCSs for each analytical batch.
- At least one sample from each matrix type in the batch was run in duplicate and spiked. (**Note:** A spiked sample with a replicate spiked sample may be substituted for sample run in duplicate and spiked.)
- No digestion batch exceeds 20 analytical samples. (**Note:** Do not count PBs, LCSs, lab duplicates, spikes, or spike replicates as analytical samples.)

Action 5: If any of the above criteria is not met, comment and assign the reason code [168] to all applicable data.

2.17. Instrument Run Logs (Summary Forms 14)

Review Items: Form 14 or equivalent

Objective : To determine that the Form 14 analysis run log summary adequately and accurately reflects the sample analysis in the Sample Data Package.

Sources: Attachment I to BOA Attachment I, and Base Method

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that Form 14s are present for each run performed and include:

- Analysis start and end date
- Instrument Identifier
- Sample Identifiers including spikes, duplicates, preparation blanks, and control samples
- Dilution factors
- Analysis times

Action 1a: If the Form 14's are not present, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 1b: If the Form 14's are present but are not complete, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: *The following items apply to validation only:*

Item 2: Evaluate analytical spikes, dilution factors, and run times from the raw data.

- Compare instrument IDs and analysis run dates reported on Form 14 to Instrument IDs and dates printed on raw instrument data for one instrument run.
- Compare raw data and Form 14 data sample identifiers, run times, and dilution factors for at least three samples per run.
- For each GFAAS run, check that at least three analytical spike recoveries are correctly entered.
- Check that reported data for each analyte are indicated by X's.

Action 2: If any of the following items are non-compliant, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.18. Sample Preparation Raw Data

Review Items: Raw Data

Objective: To check that sample preparation raw data deliverable requirements have been met and that raw data are present in a form suitable for data assessment.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to validation only:*

Item 1: Check that preparation raw data (benchsheets and/or preparation logs) are included for all analyses performed and include the following:

- Analytical Batch identifier
- Date of preparation
- Identifiers for all samples, sample duplicates, and spikes
- Identifiers for at least one preparation blank and lab control sample
- For aqueous samples initial and final volumes for all samples and QC samples
- For solids and non-aqueous liquids reported by weight, initial weights and final volumes for all samples and QC samples
- For samples reported by weight, balance identifiers with dates of use.
- Dated signatures for at least one analyst and one reviewer

Action 1a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 1b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that sufficient raw data are included in the SDP to allow manual calculations of the final reported sample results.

Action 2: If insufficient data are included, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.19. Instrument Raw Data

Review Items: Raw Data

Objective: To verify that the instrument raw data is provided for all reported data and that the data is consistent with the results reported on the summary forms.

Source : Attachment I to BOA Attachment 1, and Base Method

Evaluation: *The following items apply to validation only:*

Item 1: Check that instrument raw data are included for all analyses by performing the following checks:

- Check that all instrument raw data for the RIN are included and legible.
- Check at least one ICP and one AAS printout for identified spiked samples and laboratory duplicates
- Check that instrument identifiers are on raw data
- Check at least ten raw data sheets for legibility and proper error correction techniques.
- Check that preparation blank and LCS data are clearly linked to the digestion batch.
- Check instrument raw data printouts for area spectroscopist review, signature, and date on each instrument batch.
- Check that there are sufficient raw data to perform calculations for all previous items.

Action 1a: Noncompliant items, omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 1b: For other noncompliant items, omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.20. TCLP Sample and Extract Preparation (Summary Form 2)

Review Items: Form 2 or equivalent, and raw data.

Objectives: To determine if samples were evaluated and prepared by the proper TCLP preparation method according to LIC, analyte, sample matrix, and analytical method utilized.

Sources: Attachment I to BOA Attachment 1, GR03 § 5, and Method 1311 for TCLP extraction.

Evaluation: *The following items apply to both verification and validation:*

Item 1: Check that a Form 2 or equivalent is present and the following information is included:

- Lab name, Lab Code, Analytical Batch Identifier and the RIN.
- Form 2 data for each sample.
- Physical descriptions of the samples (e.g. *multiphase liquid*, or *solids with no free liquid*) and a statement about which samples are of the same matrix.
- Result for the preliminary determination of percent solids and a description of the method of determination.

- An indication of whether particle size reduction was completed and how the reduction was completed, if reduction was required.
- A *Yes* or *No* to indicate whether free liquid was present in the sample.
- A *Yes*, *No*, or *N/A* to indicate whether any free liquid present was miscible with the extraction fluid.
- A volume recorded if a non-miscible liquid is present.
- A check that the preliminary evaluation of the pH of solids is recorded.
- A check that the evaluation of the pH of solids after the addition of acid is recorded, if applicable.
- A *Net Sample Weight (g)* or total weight of sample taken for the extraction process is recorded.
- A *Net Weight of Solids Extracted (g)* or the net weight of solids remaining after liquid solid separation is recorded.
- The type and weight of the extraction fluid added to the extraction vessel is recorded.
- The *Date and Time* of the start and end of the extraction period were recorded.
- The pH for the leachate solution after extraction and filtration, but before preservation was recorded.
- The method of preservation of the leachate was recorded.
- At least one spike-sample was prepared per waste type and analytical batch.
- At least one extraction blank was prepared per extraction fluid type and analytical batch.
- At least one duplicate sample was prepared per waste type and analytical batch.

Action 1a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 1b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: *The following items apply to validation only:*

Item 2: Determine that the appropriate TCLP Extraction method was completed for each sample.

Action 2: If the incorrect method was used for sample preparation and a CTR approved deviation was not documented, estimate [J 207] all applicable data.

Item 3: Check for evidence that samples with solids less than 0.5% were filtered as a TCLP Extract.

Action 3: If the percent solids is less than 0.5% and the sample was not filtered, estimate [J 220] positive results that exceed the regulatory level.

- Item 4:** Check for evidence of particle size reduction when the sample particle size exceeds 9.5 mm or the surface area is less than 3.1cm^2 .
- Action 4:* If particle size reduction is required and reduction was not performed, estimate [J 222] all sample results less than the regulatory level.
- Item 5:** Verify that TCLP results for extracts of samples with free liquids, both miscible and non-miscible, were reported appropriately.
- Action 5a:* If a single combined TCLP result was not reported for a sample with both miscible and non-miscible liquids and this deviation was not addressed in the narrative, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Action 5b:* If a single combined TCLP result was not reported for a sample with both miscible and non-miscible liquids and this deviation was addressed in the narrative, comment and assign the reason code [248].
- Item 6:** Verify that the correct Extraction Fluid Type was used for the TCLP according to the following:
- If the pH before or after (as applicable) the acidification is less than 5, Extraction Fluid Type 1 is to be used for the TCLP of all analyses.
 - If the pH after acidification is greater than 5, Extraction Fluid Type 2 is to be used for the TCLP of all analyses.
 - Extraction Fluid Type 1 is to have a pH of 4.93 ± 0.05
 - Extraction Fluid Type 2 is to have a pH of 2.88 ± 0.05
- Action 6a:* If an incorrect or improperly prepared Extraction Fluid Type was used for the TCLP, comment and qualify using professional judgment, but qualify at a minimum as estimated [J 233].
- Action 6b:* If the extraction fluids are not numbered and cannot be identified from the data, comment and qualify using professional judgment, but qualify at a minimum as estimated [J 224].
- Item 7:** Verify that the correct amount of sample was processed for the TCLP.
- Action 7:* If the net sample weight processed for TCLP is less than 100 grams, use professional judgment to determine if the sample size is too small. Consider the physical state of the sample, the availability of sample, potential mixed waste issues (waste minimization priority), and whether particle size reduction was performed. At a minimum, comment and assign the reason code [123].
- Item 8:** Verify that the extraction period was within 16 to 20 hours.
- Action 8:* If the extraction start and end dates and times are not available or if the extraction time is not within 16-20 hours, use professional judgment to evaluate the data. Results near the regulatory limit may be biased low if the extraction time is less than 16 hours and results just above the regulatory limit may be biased high if the extraction time is greater than 20 hours. Results just below the regulatory limit that are suspected of low bias due to an insufficiently short extraction time are Rejected [R 225].

Item 9: Verify that TCLP Extracts were preserved appropriately, if analysis was not completed immediately.

Action 9: If the TCLP Extracts were not analyzed immediately after extraction and were not preserved at $4 \pm 2^\circ \text{C}$ after extraction, comment and qualify all results less than the regulatory limit as estimated [**J 201**].

Item 10: Verify that a minimum of one TCLP Spike, Blank, and Duplicate are processed per waste type, preparation batch and extraction fluid type.

Action 10: If evidence of a spiked sample, duplicate sample, or extraction blank are not provided, comment and qualify all results as rejected [**R 168**].

Item 11: Verify that the ambient temperature during the extraction was maintained at $23 \pm 2^\circ \text{C}$.

Action 11: If the ambient temperature during TCLP extraction was not maintained at $23 \pm 2^\circ \text{C}$, estimate [**J 201**] all results less than the regulatory limit.

3. DATA QUALITY ASSESSMENT REPORT PREPARATION

Prepare a Data Quality Assessment Report according to the General Data Assessment guidelines presented in DA-GR01. A Data Quality Assessment Report template for DV-SS05 is presented as Attachment 1.

4. REFERENCES

- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, February 1994.
- Reason Codes for Data Assessment, Analytical Services Document
- RFETS BOA Implementation Requirements, GR03 Version A.5
- RFETS BOA Implementation Requirements, GR04 Version A
- Basic Ordering Agreement (BOA) for Laboratory Analytical Services administered by Westinghouse Savannah River Company on behalf of the Department of Energy.

ATTACHMENT 1: DATA QUALITY ASSESSMENT REPORT TEMPLATE

MET

**Data Quality Assessment Report
Rocky Flats Environmental Technology Site**

RIN Number	Analytical Method/Analytical Specific Line Item Code		Review Level
Analytical Laboratory	Assessment Performed by	Data Assessment Guideline Identifiers	Number of Samples

Sample Numbers: _____

Quality Control Items	Reviewed (Y or N)	Non-Compliance Identified
General (Cover Page, Narrative)		
Chain of Custody, Preservation, and Holdings		
Sample Results		
Calibration Verification, CRDL Standard		
Verification and Preparation Blanks		
Interference Check Sample		
Matrix Spike		
Duplicates		
Laboratory Control Sample		
Standard Additions		
ICP Serial Dilution		
Instrument Detection Limits		
Other: Interelement Correction Factors, Linear Range Studies, Preparation Logs, Instrument Run Log		
Preparation and Instrument RAW Data		
Standards		
EDD		
Other:		

Y Item was reviewed or non-compliance was identified
 N Item was not reviewed or non-compliance was not identified
 N/A Item is not applicable to the Line Item

MET
Data Quality Assessment Report
Rocky Flats Environmental Technology Site

Data Assessment results are classified as either Action Items or Comments. Action Items are technical non-compliances that result in qualification of analytical results. Data may be qualified as valid (V), estimated (J), presumptively estimated (NJ), estimated at an elevated level of detection (UJ), or rejected (R). Multiple qualifiers may be associated with any given data point based on the number of problems identified, however, the assigned qualifier is based upon the following hierarchy: R, UJ, NJ, J, V. All data points that are not qualified based upon action items in this report are considered valid (V). Comments are technical non-compliances or contractual non-compliances that do not result in qualification of data.

Action Items:

Comments:

Verification/Validation Signature _____

Date: _____

Reviewer Signature _____
(Validation Only)

Date: _____

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