



VERIFICATION AND VALIDATION GUIDELINES

FOR

INORGANIC METALS

DA-SS05-v1

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1. PURPOSE AND INTRODUCTION

This procedure presents those data assessment steps which are unique to PSA Module SS05, Inorganic Metals. This procedure is to be used in conjunction with the general guidelines for data verification and validation, DA-GR01.

The purpose of this procedure is to provide guidance in the completion of Data Review Checklist (DRC) Examination, Data Verification, and Data Validation activities as part of the Rocky Flats Environmental Technology Site (RFETS) Analytical Services Division (ASD) Data Assessment Program. The Data Assessment Program is described in the Kaiser-Hill Analytical Services Division Procedure ASD-001, Performance Assurance Data Assessment Program..

This version of DA-SS05, until replaced by a more recent version, is applicable to all versions of the PSA Module SS05.

This procedure for the data quality assessment of SS05 Sample Data Packages is organized into the following Sections:

- ◇ DRC Examination Instructions
- ◇ Verification and Validation Instructions
- ◇ Data Quality Assessment Report Preparation
- ◇ References
- ◇ Revision History
- ◇ Attachments

2. DATA REVIEW CHECKLIST (DRC) EXAMINATION INSTRUCTIONS

The instructions contained in this section are specific to PSA Module SS05 for inorganic analyses. The instructions in this section are to be used in conjunction with the general instructions for DRC Examination found in ASD Procedure DA-GR01.

2.1. Examination of *NA* Replies

Several items in the DRC Checklist may be marked as "NA," indicating that the item was not applicable to the analysis performed or to the data package. For the following listed items in Table 2-1, enter √ in the √ column of the DRC for the following items to indicate that the "NA" response is accepted but not verified:

TABLE 2-1 NON APPLICABLE DRC ITEMS

Section 1	Section 3	Section 4	Section 5	Section 6	Section 8
1-d	3-b	4-b	5-c-1	6-b-5	8-a
		4-d	5-c-2	6-d-5	
		4-e	5-c-4	6-d-8	
		4-f	5-d	6-f-4	
		4-g	5-e	6-g	
			5-f	6-h-4	
				6-k-4	

2.1.1. Several DRC checklist items are not applicable to AA analysis and therefore should be marked as NA in the Reply column if ICP or ICP-MS was not used for any analysis in the Sample Data Package. Enter \checkmark in the \checkmark column for the following items if ICP or ICP-MS was not used for any analysis to indicate that the response is accepted:

- ◇ 6-e (all items)
- ◇ 6-k (all items)
- ◇ 6-m
- ◇ 6-n
- ◇ 6-o

2.1.2. For item 7-a, when marked *NA* in the Reply column, enter a " \checkmark " in the \checkmark column only if all samples in the RIN were analyzed for Line Item Codes designating *dissolved* analysis types. (Line Item Codes SS05*07 through SS05*12; SS05*17 through SS05*20; SS05*25, 26, 29, or 31, 37, 45, 46, 51, 54, 56, 58, and 59 where * denotes the latest revision of the PSA SS05 module.):

2.1.3. For all other items with "NA" marked in the Reply column, enter an "X" in the \checkmark column to indicate that verification is required for this item.

2.2. Examination of the Sample Narrative

Read the sample narrative for information which indicates additional items that need to be verified. Items to check include statements about data qualifiers, blank or reagent contamination, or sample handling problems.

- If the narrative states that "N Flags" are present, enter "X" in the \checkmark column of item 6-f-4 to indicate that verification is required for this item due to information provided in the Narrative.
- If the narrative states that "* Flags" are present, enter "X" in the \checkmark column of item 6-h-4 to indicate that verification is required for this item due to information provided in the Narrative.
- If the narrative states that "E Flags" are present, enter "X" in the \checkmark column of item 6-k-4 to indicate that verification is required for this item due to information provided in the Narrative.

3. VERIFICATION AND VALIDATION INSTRUCTIONS

The instructions contained in this section are specific to PSA Module SS05 for inorganic analyses. The instructions in this section are to be used in conjunction with the general instructions for verification and validation found in the ASD procedure DA-GR01. The following subsections include specific instructions for performing verification and validation activities that are specific to PSA Module SS05. Each of the following subsections corresponds to a SS05 DRC Checklist section that may contain multiple Item numbers. These Item numbers are associated with instructions for assessing each DRC checklist Item within each of the following subsections:

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

DRC Items 4-a through 4-g

Review Items: Deliverable Sections 3, 4, and 5, Form 7, Chain-of-Custody Documentation, and raw data .

- Requirement Source:** GR01 Exhibit B Section 4.8 and GR01 Exhibit D Section 3, SS05 Exhibit D Section 3 and Table 3-1 below.
- Objective:** To ascertain the validity of results based on the holding time and preservation of the sample and to check that Sample COC documentation is included in the SDP.
- Note: The holding time is based on the date when collection was completed, rather than the verified time of sample receipt (VTSR).
- Evaluation:** *The following items apply to both verification and validation:*
- Technical requirements for sample holding times and sample preservation for SS05 are listed in the following Table 3-1, Holding Time and Preservation Criteria:

Table 3-1 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
All metals other than Hg	180 days	Storage at 4°C	pH <2 w/HNO ₃

Items 4-a, b, c, & e Follow instructions in DA GR01.

- Item 4-d** Check for documentation that the sample pH was adjusted to ≤ 2 and the temperature was maintained at 4°C prior to receipt by the laboratory.
- If samples were not acid-preserved and not maintained at 4°C prior to receipt by the laboratory, comment and assign the reason code [703] to all applicable samples.
- Item 4-f** 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, qualify all results according to the following guidelines:
- 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 initiate a Non-Conformance Notification and assign the qualifier [J 101]. If the hold-time violation is not attributed to the laboratory, initiate a Non-Conformance Notification and assign qualifier [J 701].
 - Qualify all non-detects 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, initiate a Non-Conformance Notification and assign qualifier [R 102]. If the hold-time violation is not attributed to the laboratory, initiate a Non-Compliance Notification and assign qualifier [R 702].
 - 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, initiate a Non-

Conformance Notification and assign the qualifier **[J 101]**. If the hold-time violation is not attributed to the laboratory, initiate a Non-Conformance Notification and assign qualifier **[J 701]**.

- Item 4-g** 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.
- If an aqueous sample was not adjusted to the proper pH by the laboratory, when required, initiate a Non-Compliance Notification and qualify all results as estimated **[J 201]**.

3.2. Sample Data Package Narrative Requirements

DRC Items 5-a through 5-f

- Review Items:** Deliverable Section Numbers 3, 5 and 6.
- Objective:** To determine compliance to Narrative requirements and obtain information useful for validation of data.
- Requirement Source:** GR01 Exhibit B § 4.9 and SS05 Exhibit B § 2.7
- Evaluation:** *The following items apply to both verification and validation:*

Item 5-a through Item 5-f

Check that the SDP Narrative is present and that each Item 5-a through Item 5-d are compliant.

- If the Narrative or any of the following items are non-compliant and needed for data assessment, initiate a Non-Compliance Notification for the missing information and assign the reason code **[801]** for a missing narrative, and the reason code **[805]** for information missing from the narrative to all applicable data. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.
- If any of the following items are non-compliant and not required for data assessment, comment and assign the reason code **[804]** to all applicable data.

- Item 5-a** There is a synopsis of approved sample preparation and analytical methods. The base method of each SOP is identified and deviations from the base methods are identified.
- Item 5-b** There are physical descriptions of the samples and a statement about which samples are of the same matrix.
- Item 5-c** There is a synopsis of the original Analytical Batch QC assessment.
- Item 5-c-1** There is a statement about items assigned “N”, “*”, and “E” Flags.
- Item 5-c-2** There is a statement indicating if dilutions were required, what dilutions were performed, and the reason for all dilutions (except ICP serial dilution).
- Item 5-c-3** There is a statement indicating if RDLs were or were not met for all analytes.
- Item 5-c-4** There is an explanation for any RDLs that were not met.

- Item 5-c-5** There is a discussion on all sample results that were reported using Method of Standard Additions.
- Item 5-d** There is identification of any samples requiring reanalysis with the reason for reanalysis, the original and reanalysis Analytical Batch Identification Numbers, and a synopsis of the reanalysis Analytical Batch QC assessment.
- Item 5-e** If any deviations required CTR approval, check for documentation of this approval.
- Item 5-f** There is a statement for all holding-time compliances and deviations with a description and explanation of any holding-time violations.

3.3. Sample Results (Summary Forms 1)

DRC Items 6, 6-a, 6-a-1 through 6-a-8

Review Items: Deliverable Section Number 6, and Forms 1, 5A, 6, and 9.

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

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.

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

Item 6 Check that the Sample and QC Results Summary Package is present.

- If the Sample and QC Results Summary Package is not present, initiate a Non-Compliance Notification for the missing deliverable and assign the reason code **[801]**. Do not qualify data without any missing Result Summary Forms. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 6 through Item 6-a-8

Check that each following Item 6 through Item 6-a-8 is compliant.

- If any of the following items are non-compliant and required for data assessment, initiate a Non-Compliance Notification and assign the reason code **[803]**. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.
- If any of the following items are non-compliant and not required for data assessment, comment and assign the reason code **[804]**.

Item 6 Check that the Sample and QC Sample Results Summary Package Forms are labeled with the Lab Code and the RIN.

Item 6-a Check that Form 1s are present for each sample in the RIN for this PSA Module.

Item 6-a-1 Check that one and only one result is reported on Forms 1 for each requested analyte.

Item 6-a-2 Check that results for samples analyzed as aqueous liquids (including

- TCLP) are reported in $\mu\text{g/L}$ and solids are reported in mg/Kg .
- Item 6-a-3** 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.
 - Item 6-a-4** Check that results for non-detected analytes are reported at the IDL and factored for any dilutions.
 - Item 6-a-5** 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 .
 - Item 6-a-6** Check that C, and M qualifiers are entered correctly for each analyte.
 - Item 6-a-7** 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 Summary 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.
 - Item 6-a-8** Check that the detection limit of each diluted sample ($\text{IDL} \times \text{dilution factor}$) for a non-detected analyte is \leq the specified RDL (see Table 3-2).

3.4. Calibration Verification (Summary Form 2A)

DRC Items 6-b, 6-b-1 through 6-b-7

Review Items: Deliverable Section Number 6, Form 2, Form 8, 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.

Note: 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.

Requirement Sources: SS05 Exhibit B §2.8, SS05 Exhibit D §§4.9, 5.6, and SS05 Exhibit D §2 (base method requirements).

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

Item 6-b Check that Form 2As are present.

- If a Form 2A is not provided, initiate a Non-Compliance Notification to request the missing Form 2s and assign reason code **[801]** to all applicable data. Do not qualify data without the Form 2. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 6-b-1. Check that ICV and CCV results are reported for all requested analytes.

- If ICV or CCV results are not reported for an element, initiate a Non-Compliance-Notification and qualify all results for the element as rejected **[R 104]**.

Note: that the ICV may be designated as QCS due to naming conventions in the EPA-600 methods. Evaluate QCS results by the same criteria listed below for ICV.

Item 6-b-4 Verify that all ICV and CCV percent recoveries (%R) are between 80 and 120% for mercury and 90-110% for all other metals.

Item 6-b-5 If the ICV %R for any element falls outside the acceptance windows, initiate a Non-Compliance-Notification 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 111-125% (Hg, 121-135%), results less than the IDL are acceptable. However, assign reason code **[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]**

If the CCV %R for any element falls outside the acceptance windows of 90-110% (Hg 80-120%), check for reanalysis of the affected samples bracketed by CCV analyses with %R values within the acceptance window. If any reported data is Not bracketed by acceptable calibration verifications (ICV or CCV), initiate a Non-Compliance-Notification and qualify all data, not bracketed by acceptable calibration verifications for that element, 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 ICV %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 items apply to validation only:*

Item 6-b-2 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).

- If an ICV was not analyzed or site samples were analyzed before the ICV, initiate a Non-Compliance-Notification and qualify all samples analyzed before the first calibration verification sample as Rejected **[R 129]**.

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

- If a CCV was not analyzed after the last site samples were analyzed, initiate a Non-Compliance-Notification and qualify all samples analyzed after the last acceptable CCV as Rejected **[R 129]**.

Item 6-b-3 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).

- 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.

Check the time between calibration verifications is not more than two hours

- If more than two hours elapsed between consecutive calibration verifications, initiate a comment and assign the following reason code **[129]**.

Item 6-b-4 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.

- If the raw data and reported data do not agree, comment and assign the reason code **[152]**.

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.

- If the results do not agree, assign the reason code **[153]** for calculation errors.
- Do not recalculate the data for qualification. Initiate a Non-Compliance Notification for the discrepancies. Do not qualify data without a revised Form 2 and SDP, if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Note: When there are calculation errors for the ICV and CCV %R values reported on the Form 2A, the verification and validation assessment activities for the items 6-b-4 and 6-b-5 must be completed with the corrected ICV and CCV %R values and new qualifiers assigned, if required.

- Item 6-b-6** 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.
- 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 6-b-7** Verify that the correlation coefficient for all analyses other than ICP was greater than 0.995.
- If the correlation coefficient is less than 0.995, qualify all associated results as estimated **[J 103]**.
- Item 6-b-8** Check standard logs for sources of standards used for calibration and ICV for at least three elements (not to include uranium). Assure that the sources for calibration are independent of the sources for the ICV. If the information provided is not adequate to identify independent sources for the initial three elements, check all other elements for use of independent sources.
- If the information provided is not adequate to identify independent sources, initiate a Non-Compliance Notification for the missing information and assign reason code **[801]**.
 - If the information is not available, the reviewer must assume that standards are not independent. All results for any element for which independent calibration verification cannot be demonstrated, shall be qualified as estimated **[J 140]**. Initiate a Non-Compliance Notification to notify the lab of the deficiency.

3.5. **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.

DRC Items 6-c, 6-c-1 through 6-c-3

Review Items: Deliverable Section Number 6, and Form 2B

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

Requirement Sources: SS05 Exhibit B §2.8, SS05-A Exhibit D §§4.11 5.6, and SS05 Exhibit D §2 (base method requirements).

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

Item 6-c Check that Form 2Bs are present.

- If the Form 2Bs are not in the SDP, initiate a Non-Compliance Notification to request the missing forms and assign the reason code **[801]**. Do not qualify data without the Form 2Bs. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 6-c-3 Verify that the CRDL %R values were within the limits of 80 to 120% for ICP analyses.

- 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 initiate 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 initiate 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]**. Initiate a Non-Compliance Notification for the unacceptable recoveries.

Evaluation: *The following items are examined for validation only:*

Item 6-c-1 Verify that the CRDL Standard was analyzed at the proper concentration (2 times the CRDL or IDL, whichever is greater) for each analyte.

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

Item 6-c-2 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.

- Qualify all ICP results as estimated **[J 129]** if the CRDL frequency requirement was not met.

3.6. Blanks (Summary Form 3)

DRC Items 6-d, 6-d-1 through 6-d-8

Review Items: Deliverable Section Number 6, Forms 3 and 10, 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.

Note: 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.

Requirement Sources: SS05 Exhibit D Sections 4.9 and 5.6, and SS05 Exhibit D Section 2 (base method requirements). Specific references to base method requirements include ILM 4.0, Exhibit B Section III F (page B-25).

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

Item 6-d Check that Form 3s are present.

- If Form 3s are not provided, initiate a Non-Compliance Notification to request the missing forms and assign the reason code **[175]**. Do not qualify data without the Form 3. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 6-d-1 Check that ICB, CCB and PB results are reported for all requested analytes.

- If any ICB, CCB, and PB results are not reported, initiate a Non-Compliance Notification to request appropriate corrective action and assign the reason code **[175]** to all sample results without associated blank results. Do not qualify data without a revised Form 3. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Check that Form 3 results are reported to the IDLs listed on Form 10.

- If the Form 3 results are not reported to the IDLs listed on the Form 10, initiate a Non-Compliance Notification and assign the reason code **[215]**. Do not qualify until revised data package is delivered. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Items 6-d-4 through 6-d-8

Verify that the absolute value of any ICB, CCB, or PB result is not greater than the IDL for the element. Check for the reanalysis of all samples associated with unacceptable blanks, if applicable.

- All sample results for an element which are greater than 10 times the absolute value of the blank result may be accepted without qualification.

Comment and qualify other results with non compliant blanks 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 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 less than 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 less than the negative IDL, but greater than 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 remainder of items are performed for validation only:*

Item 6-d-1 Obtain blank results from the instrument raw data for at least three elements with blank results less than the IDL, less than the negative IDL, and greater than the IDL. Compare the results to those reported on Form 3.

- 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, all reported blank results must be checked against raw data. Comment and assign the reason code **[152]** to all applicable data.

Item 6-d-2 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.

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

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

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

Item 6-d-3 Check that no more than 10 solutions were analyzed between any two consecutive analyses of CCB (include all solutions analyzed except ICBs and CCBs).

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

Check that no more than two hours elapsed between consecutive analyses of calibration verification blanks (ICBs or CCBs).

- If more than 2 hours have elapsed between consecutive calibration blank analyses, comment and assign the reason code **[129]** to all affected samples.

Note: The analysis should be terminated when the absolute value of any blank result

exceeds the RDL.

Note: 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: 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:

- ◇ Correct Preparation blank action levels for the percent moisture content of the sample
- ◇ Correct blank action levels for ICB and CCB analyses by the percent moisture content of the sample as well as the weight and volume adjustments of the soil sample.

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

DRC Items 6-e, 6-e-1 through 6-e-3

Review Items: Deliverable Section Number 6, and Form 4.

Objective: To assess the impact of the contract laboratory ICPES interelement interferences on sample results with high levels of interferent elements.

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 exhibit D Section 2.

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

Item 6-e Check that Form 4A and 4B are present for ICP analyses.

- If a Form 4A or 4B are not present, initiate a Non-Compliance Report and assign the reason code [801]. Do not qualify data if Form 4A or 4B are missing. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 6-e-1 Check that ICSA and ICSAB results are reported for every requested analyte determined by ICPES.

- For all elements in a sample without associated ICSA or ICSB 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 6-e-2 Verify that an ICSA and ICSB are analyzed at the beginning and end of each analytical sequence (or every 8 hours, whichever is more frequent).

- If the ICSA and ICSB are not analyzed in the correct sequence and frequency, comment and qualify all results except aluminum, iron, calcium, and magnesium as estimated [J 250].

Evaluation: *The remainder of items are performed for validation only:*

Item 6-e-3 If a sample does not have aluminum, calcium, iron, or magnesium results

that exceed the ICSAB found value no further action is necessary.

For samples that have aluminum, calcium, iron, or magnesium values that exceed the found value in the ICSAB solution, verify the ICSAB %R for each element on Form 4A is within 80 to 120%. Comment and qualify the 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, these data are considered valid **[V]**.
- 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]**.

Evaluate the ICSA results for all requested elements, **except** Al, Ca, Fe, and Mg, to verify that the absolute values are not greater than the IDL for those requested elements which are not present in the ICSA solution. Comment and 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 $< IDL$, do not qualify the data.
- If the ICSA results are $>(2*IDL)$ and the sample results are $> IDL$ and $<(10)(\text{the ICSB reading})$, 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]**.

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

- If the raw data and reported data do not agree, comment and assign the reason code **[152]**.

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%.

- If the results do not agree and do not result in the need for reassessment of the Item 6-e-3, comment and assign the reason code **[153]** for the calculation errors.
- If the results do not agree and reassessment of the Item 6-e-3 is required, initiate a Non-Compliance Notification and assign the reason code **[153]** for the calculation errors. Do not recalculate the data for qualification or qualify data without a revised Form 4 and SDP, if required. Continue assessment, when

possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

3.8. Matrix Spike Analysis (Summary Forms 5A)

DRC Items 6-f, 6-f-1 through 6-f-4

Review Items: Deliverable Section Number 6, Form 5 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.

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2. For SS08 Line Item codes (TCLP Extracts) see SS08-B Exhibit D §§ 9.2.2 and 9.2.3.

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

Item 6-f Check that Form 5As are present.

- If Form 5As are not provided, initiate a Non-Compliance Notification and assign the reason code **[801]**. Do not qualify data without a Form 5A and revised SDP, if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 6-f-1 Check that at least one Form 5A is present for each method, matrix, waste type, and analytical batch.

- If a matrix spike was not analyzed at the proper frequency, initiate a Non-Compliance Notification and qualify all samples associated with the missing matrix spike as estimated **[J 168]**.
- If no matrix spike was analyzed, regardless of frequency, initiate a Non-Compliance Notification for the missing QC and qualify all samples associated with the missing matrix spike as estimated **[J 230]**.

Item 6-f-2 Check that control limits are correctly assigned.

- 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 6-f-3 Check that the matrix spike %R is reported for all requested analytes.. For SS08 Line Item Codes (TCLP Extracts) the following elements must be spiked at the levels specified in the analytical method:

- | | |
|-------------|------------|
| ◇ Antimony | ◇ Mercury |
| ◇ Arsenic | ◇ Nickel |
| ◇ Barium | ◇ Selenium |
| ◇ Beryllium | ◇ Silver |
| ◇ Cadmium | ◇ Thallium |
| ◇ Chromium | ◇ Vanadium |

◇ Lead

◇ Zinc

- If spike results and subsequent %R value are not reported for an element that was required to be spiked, initiate a Non-Compliance Notification for the element not reported and qualify all associated results as estimated [J 230].

Item 6-f-4 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, and aluminum in soil samples.
- ◇ calcium, magnesium, potassium, and sodium in water samples.
- ◇ when the sample result for an element is >4(the spike level).

- If an “N Flag” is not present when required, comment and assign reason code [804].

Comment and qualify sample results according to the following spike criteria:

- If the spike recovery is >125% and the reported sample results are < IDL, the data are valid.
- If 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 remainder of item 6-f-3 is performed for validation only:

Item 6-f-3 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.

- If the raw data and reported data do not agree, comment and assign the reason code [152].

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

$$\%R = (SSR - SR) \times 100 / SA$$

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.

Verify that the recalculated value agrees with the laboratory reported value on Form 5A to within 0.1%. If the results cannot be verified, recalculate all spike recoveries.

- If the %R values do not compare and do not result in the need for reassessment of the items 6-f-3 and 6-f-4, comment and assign the reason code **[153]** for the calculation errors.
- If the %R values do not compare and reassessment of the items 6-f-3 and 6-f-4 are required, initiate a Non-Compliance Notification and assign the reason code **[153]** for the calculation errors. Do not recalculate the data for qualification or qualify data without a revised Form 5 or SDP, if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

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

DRC Item 6-g

Review Items: Deliverable Section Number 6, and Form 5B.

Objective: To evaluate the potential sources of poor matrix spike recoveries determined with a post digestion spike.

Note: 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 is similar, then matrix effects are suspected.

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.

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

Item 6-g Check that Form 5B results are present for each pre-digestion matrix spike element which did not meet the matrix spike recovery criteria.

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

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.

- If information is not available in the Narrative, comment and assign the reason code **[804]** 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 remainder of item 6-g is performed for validation only:*

Item 6-g 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.

- If the raw data and reported data do not agree, comment and assign the reason

code [152].

Recalculate from the raw data one or more of the spiked sample percent recoveries (%R) using the equation found in Section 3.9 for pre-digestion matrix spike evaluation.

Verify that the recalculated value agrees with the laboratory reported value on Form 5B to within 0.1%. If the results cannot be verified, recalculate all spike recoveries.

- If the %R values do not compare, comment and assign the reason code [153] for the calculation errors.

3.10. Laboratory Duplicate Analysis (Summary Forms 6)

DRC Items 6-h, 6-h-1 through 6-h-4

Review Items: Deliverable Section Number 6, Form 6 and raw data.

Objective: To verify acceptable precision of sample results for the sample matrix, laboratory preparation, and analysis procedure.

Note: Duplicate sample determinations are used to measure variability due to a combination of factors including laboratory precision, method precision, and sample homogeneity. (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.)

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.

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

Item 6-h Check that Form 6s are present.

- If the Form 6s are not available, initiate a Non-Compliance Notification and assign the reason code [801]. Do not qualify data without a Form 6 and revised SDP, if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 6-h-1 Verify that at least one duplicate analysis was performed for each matrix, waste type, and analytical batch.

- If a duplicate analysis was not at the appropriate frequency, initiate a Non-Compliance Notification and qualify all associated samples as estimated [J 168].
- If an element for a duplicate analysis was not analyzed, initiate a Non-Compliance Notification and qualify the element for all associated samples as estimated [J 230].

Item 6-h-2 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.

- 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 6-h-3 Check that the original and duplicate results, and subsequent RPD when required are reported for each requested element..

- If duplicate results and subsequent RPD are not reported for a required element, initiate a Non-Compliance Notification and qualify all associated results as estimated [J 230].
- If duplicate results were reported but the subsequent RPD was not entered on the Form 6 when required, comment and assign reason code [803].

Item 6-h-4 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.

- If an "* Flag" is not present when required, comment and assign reason code [803].

Comment and qualify sample results according to the following duplicate criteria:

- 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].
- 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 remainder of item 6-h-3 is performed for validation only:*

Item 6-h-3 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.

- If the raw data and reported data do not agree, comment and assign the reason code [152].

Recalculate 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)

Verify that the recalculated value agrees with the laboratory reported value on Form 6 to within 0.1%. If the results cannot be verified, recalculate all duplicate RPDs.

- If the RPD values do not compare and do not result in the need for reassessment of the items 6-h-2 and 6-h-4, comment and assign the reason code **[153]** for the calculation errors.
- If the RPD values do not compare and require reassessment of the items 6-h-2 and 6-h-4, initiate a Non-Compliance and assign the reason code **[153]** for the calculation errors. Do not recalculate the data for qualification or qualify data without a revised Form 6 or SDP if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

3.11. Laboratory Control Sample Analysis (Summary Forms 7)

DRC Items 6-i, 6-i-1 through 6-i-2

Review Items: Deliverable Section Number 6, Form 7 and raw data.

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

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.

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

Item 6-i Check that Form 7s are present.

- If the Form 7s are not present initiate a Non-Compliance Notification and assign the reason code **[801]**. Do not qualify data without a Form 7 or SDP if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 6-i-1 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.

- If an LCS was not present or an LCS with the incorrect matrix was used, initiate a Non-Compliance Notification and qualify all results as estimated **[J 230]**.
- If an LCS was present but not at the appropriate frequency, comment and qualify all results as estimated **[J 168]**.

Check that LCS results are reported for all requested elements except antimony and silver, and percent recoveries are within the control limits.

Note: The required element lists for LCS are identical to the required list for matrix spike analyses. See Section 3.9, Item 6-f-3 for required TCLP elements.

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

Item 6-i-2

For aqueous LCS recoveries, comment and qualify sample results according to the criteria following:

- 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]**.

For solid LCS recoveries, comment and qualify sample results according to the criteria following:

- If the solid LCS recovery for any analyte falls outside 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 action is taken.
- If the LCS results are lower than the control limits, then qualify all sample results < IDL as estimated **[J 110]**.

Evaluation:

The remainder of item 6-i-2 is performed for validation only:

Item 6-i-2

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%.

- If the raw data and reported data do not agree, comment and assign the reason code **[152]**.

Recalculate one or more of the reported recoveries (%R) according to 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

Verify that the recalculated values agree with the laboratory reported values on Form 7 to within 0.1%. If the results cannot be verified, recalculate all LCS percent recoveries.

- If the LCS %R values do not compare and do not result in the need for reassessment of item 6-i-2, comment and assign the reason code **[153]** for the calculation errors.
- If the %R values do not compare and require the reassessment of Item 6-i-2, Initiate a Non-Compliance Notification and assign the reason code **[153]** for the calculation errors. Do not recalculate the data for qualification or qualify data without a revised Form 7 or SDP, if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

3.12. Furnace AA Quality Assurance (Summary Forms 8)

DRC Item 6-j

Review Items: Deliverable Section Number 6 Form 2A, 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.

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.

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

Item 6-j Verify that a Form 8 is present for each sample with results marked S in the Form 1 Q column.

Note: GFAA quality control procedures apply only to elements that are analyzed using the GFAA instrumentation.

- If a Form 8 is not present when required, initiate a Non-Compliance Notification and assign the reason code **[801]**. Do not qualify data without a Form 8 or SDP if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Evaluation: *The remainder of item 6-j is performed for validation only:*

Item 6-j Verify that 2 replicate injections were performed and the readings agree within 20% Relative Standard Deviation (RSD) for sample concentrations greater than the RDL.

- 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]**.
- If the RSD of the reanalyzed sample is greater than 20% qualify the sample results as estimated **[J 130]**.

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

- If the post-digestion spike was not run immediately after the sample or the spike concentration was not 2x the RDL, initiate a Non-Compliance Notification and assign the reason code [114].

For a sample with an absorbance or concentration that is less than 50% of the analytical spike value, comment and qualify the sample result according to the following criteria:

- 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 [J 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, qualify data as valid [V 216].

For a sample with an absorbance or concentration that is greater than 50% of the spike concentration and the analytical spike recovery is less than 40%, comment and qualify the sample result according to the following criteria:

- 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 [J 216].
- If the analytical spike recovery is less than 10% and the result is a non-detect, then the result is rejected [R 217].

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%. Comment and qualify the sample result according to the following criteria:

- 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 (5), 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].

3.13. ICP Serial Dilution (Summary Forms 9)

DRC Items 6-k, 6-k-1 through 6-k-4

Review Items: Deliverable Section Number 6, and Form 2A.

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

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.

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

- Item 6-k** Check that Form 9s (serial dilution results) are present if any reported results were determined by ICPES.
- If the results are not present when required, then initiate a Non-Compliance Notification and assign the reason code **[801]**. Do not qualify data without a Form 9 or SDP if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.
- Item 6-k-1** Verify that at least one serial dilution was reported for each analytical batch and matrix analyzed by ICPES.
- If a serial dilution was not analyzed with an ICP analytical batch, qualify all detected results as estimated **[J 230]**.
 - 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 6-k-2** Verify that control limits for % Difference (%D) on the Forms are either 90-110%, or blank. The control limits on the Form 9 are to be listed as 90-110% whenever the original sample concentration is > 50(IDL).
- If the control limits are not assigned correctly, comment and assign the reason code **[232]**. Continue to evaluate the serial dilution results using the correct limits
- Item 6-k-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).
- If %D results are not reported for a required element, initiate a Non-Compliance Notification and qualify all associated results as estimated **[J 230]**.
 - If serial results were reported but the subsequent %D was not entered on the Form 9 when required, comment and assign reason code **[803]**.
- Item 6-k-4** Check that an "E Flag" is present if the sample result or serial dilution result is > 50(IDL) and the %D is > 10%.
- If an "E Flag" is not present as required, assign reason code **[803]**.
 - If the sample result or serial dilution result is > 50(IDL) and the %D is > 10%, comment and qualify all detected results for the element as estimated **[J 117]**.

Evaluation: *The remainder of item 6-k-3 is performed for validation only:*

- Item 6-k-3** 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%.
- If the raw data and reported data do not agree, comment and assign the reason code **[152]**.

Recalculate 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

Verify that the results have been correctly reported on Form 9 to within 0.1%. If the results do not agree, recalculate all Serial Dilution results.

- If the serial dilution %R results do not compare and do not result in the need for reassessment of the items 6-k-2 and 6-k-4, comment and assign the reason code **[153]** for calculation errors.
- If the serial dilution %R results do not compare and require reassessment of items 6-k-2 and 6-k-4, initiate a Non-Compliance Notification and assigned the reason code **[153]** for calculation errors if required. Do not recalculate the data for qualification qualify data without a revised Form 7 or SDP, if required. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

3.14. Instrument Detection Limit (Summary Form 10)

DRC Items 6-I, and 6-I-1

Review Items: Deliverable Section Number 6, and Form 10

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

Requirement Sources: SS05 Exhibit D §2.8.

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

Item 6-I 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 PSA Module Line Item Code.

- If an IDL is not provided, initiate a Non-Compliance Notification for the missing IDL values and assign the reason code **[212]** to all results associated with the missing IDL values. Do not qualify data without a Form 10. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Verify that the IDL for each element is equal to or less than the RDL listed in SS05 Exhibit C Table C3 for the Line Item Code. See the following Note and Table 3-2 for RDL information.

- If the IDL is greater than the RDL, initiate a Non-Compliance Notification and qualify all non-detected results as rejected **[R 213]**.

Note: The RDL is 1 µg/L for the following Line Item Codes : SS05*033, SS05*034, SS05*035, SS05*056, SS05*058, and SS05*059. The RDL for the Line Item Code SS05*036 is 150 µg/L

The following Table 3-2, identifies the appropriate RDL Column to be used in SS05 Exhibit C, Table C3 for the Line Item Codes specified:

Table 3-2 RDL Lists for Line Item Codes

RDL Column from SS05 Exhibit C, Table C3	For Line Item Codes
RDL-1 Aqueous	SS05*001 & 002 SS05*007, & 008 SS05*013, & 014 SS05*017, & 018 SS05*025, & 026 SS05*027, & 028 SS05*029, & 030 SS05*031, & 032 SS05*037, & 038 SS05*039, & 040 SS05*051, & 052 SS05*053, & 057
RDL-2 Aqueous	SS05*003, & 004 SS05*009, & 010 SS05*015, & 016 SS05*019, & 020 SS05*023, & 024 SS05*041, & 042 SS05*045, & 046 SS05*049, & 050
RDL-3 Aqueous	SS05*005, & 006 SS05*011, & 012 SS05*021, & 022 SS05*032, & 052 SS05*043, & 044 SS05*047, & 048 SS05*054, & 055

* Denotes the latest revision of the PSA Module SS05

Note: If RDLs listed on Form 10 are not those required by the Line Item Code requested, check the RIN file for additional information, which may explain the deviation.

Evaluation: *The remainder of item 6-l is performed for validation only:*

Item 6-l-1 Verify that the effective date for IDLs is within 3 months of the run date. No qualification is taken if the IDL effective date has exceeded 3 months. However, comment and assign reason code [214] to all affected results.

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

DRC Item 6-m, 6-n, & 6-n-1

- Review Items:** Deliverable Section Number 6 Forms 11 and 12
- 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.
- Requirement Sources:** SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.
- Evaluation:** *The following items apply to both verification and validation:*
- Item 6-m** Check that Form 11s are present for each ICPES instrument used to report data.
- If the Form 11 is not included, initiate a Non-Compliance Notification and assign the reason code **[801]**.
- Item 6-n** Check that Form 12 is present for each ICPES instrument used to report data.
- If the Form 12 is not included, initiate a Non-Compliance Notification and assign the reason code **[801]**.
- Evaluation:** *The following item applies to validation only:*
- Item 6-n-1** Verify that all uncorrected results (for dilutions) are within the linear range of the instrument.
- If a reported result is above the linear range of the instrument, comment and qualify the result as estimated **[J 155]**.

3.16. Preparation Logs (Summary Forms 13)

DRC Items 6-o, 6-o-1 through 6-o-6

- Review Items:** Deliverable Section Number 6 Form 2A.
- Objective:** Confirm completeness and accuracy of the preparation summary form.
- Requirement Sources:** SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.
- Evaluation:** *The following item applies to both verification and validation:*
- Item 6-o** Check that Form 13 is present for each analytical batch reported, if preparation is applicable..
- If the Form 13 is required and not present, initiate a Non-Compliance Notification and assign reason code **[801]**. Do not qualify data without a Form 13. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.
- Evaluation:** *The following items apply to validation activities only:*
- Item 6-o-1** 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.

- If a sample was not prepared in an acceptable preparation batch, initiate a Non-Compliance Notification and qualify all results for the sample as rejected [R 207].
- If a sample was prepared in an acceptable preparation batch but not included on a Form 13, comment and assign reason code [804].

Item 6-o-2

- If any of the following for Items 6-o-2 are not in compliance, comment and assign the reason code [804].

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

Item 6-o-3 through Item 6-o-6

- All of the following items are subject to qualification in other verification and validation activities. Therefore, no action is taken or qualifiers added to the data for non-compliance to method requirements for the items 6-o-1 through 6-o-6. However, comment and assign reason code [804] for noncompliance to the other SOW contract requirements for Form 13.

Item 6-o-3 Check that samples are clearly linked to an associated spiked sample, lab duplicate sample, lab control sample, and preparation blank through the following:

- ◇ Compare all preparation dates and sample identifiers reported on Form 13 with raw data preparation bench sheets.

Item 6-o-4 Check for at least one set of duplicates, spikes, PBs, and LCSs for each analytical batch.

Note: A replicate spiked sample may be substituted for a duplicate sample.

Item 6-o-5 Check that 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.

Item 6-o-6 Check that no digestion batch exceeds 20 analytical samples.

Note: Do not count PBs, LCSs, lab duplicates, spikes, or spike replicates as analytical samples.

3.17. **Instrument Run Logs (Summary Forms 14)**

DRC Items 6-p, 6-p-1 through 6-p-4

Review Items: Deliverable Section Number 6, and Form 2A.

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

Requirement Sources: SS05 Exhibit B §2.8 and requirements of base methods cited in SS05 Exhibit D Section 2.

Evaluation: *The following item applies to both verification and validation:*

- Item 6-p** 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
 - If the Form 14's are not present, initiate a Non-Compliance Notification and assign the reason code **[801]**.
 - If the Form 14's are present but are not complete, comment and assign the reason code **[803]**.

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

Item 6-p-1 through Item 6-p-3

- If any of the following items are non-compliant, evaluate the analytical spikes, dilution factors, and run times from the raw data. Comment and assign the reason code **[152]**.

Item 6-p-1 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.

Item 6-p-2 For each GFAAS run, check that at least three analytical spike recoveries are correctly entered.

Item 6-p-3 Check that reported data for each analyte are indicated by X's.

3.18. Sample Preparation Method

DRC Item 7-c

Review Items: Deliverable Section Numbers 4, 5, and 7.

Objective: To determine if the proper preparation method was performed according to line item code, analyte, sample matrix, and analytical method utilized.

Requirement Sources: SS05 Exhibit C and required analysis methods

Evaluation: *The following item applies to both verification and validation:*

Item 7-c Compare the sample digestion procedure reported in the narrative to that indicated on the Table 3-3, *Digestion Methods by Line Item Code and Analysis Technique*.

- If the incorrect method was used for sample preparation and a CTR approved deviation was not documented, initiate a Non-Compliance Notification and qualify all associated data as estimated [J 207].

Evaluation: *The remainder of item 7-c is performed for validation only:*

Item 7-c Compare the sample digestion procedure listed on the preparation raw data to that indicated on the Table 3-3 below.

If the incorrect method was used for sample preparation and a CTR approved deviation was not documented, initiate a Non-Compliance Notification and qualify all associated data as estimated [J 207].

Table 3-3 Digestion Methods by Line Item Code and Analysis Technique

Line Item Code	Analysis Description	Aqueous Samples by ICPEs	Soils and Solids by ICPEs	Aqueous Samples by AA	Soils and Solids by AA
SS05*001 SS05*002 SS05*003 SS05*004 SS05*005 SS05*006 SS05*025 SS05*027 SS05*035	CLP-SOW for Total Metals of unfiltered aqueous	CLP Water ICP variation	Not Applicable	CLP Water, GFAAS and CVAA variations	Not Applicable
SS05*039 SS05*040 SS05*041 SS05*042 SS05*043 SS05*044 SS05*052 SS05*053 SS05*060	CLP-SOW for Total Metals of soils/sediments	Not Applicable	CLP Soil, ICP variation	Not Applicable	CLP Soil, GFAAS and CVAA variations
SS05*007 SS05*008 SS05*009 SS05*010 SS05*011 SS05*012 SS05*026 SS05*051 SS05*059	CLP-SOW for Dissolved Metals of filtered aqueous	CLP Water ICP variation— (Digest, unless stated otherwise on COC)	Not Applicable	CLP Water, GFAAS and CVAA variations	Not Applicable
SS05*013 SS05*014 SS05*015 SS05*016 SS05*028 SS05*030 SS05*034 SS05*038	EPA-600 for Total Recoverable Metals of unfiltered aqueous	200.2 or 200.7 for Total Recoverable	Not Applicable	200.2 or 200.9 total recoverable	Not Applicable
SS05*017 SS05*018 SS05*019 SS05*020 SS05*029 SS05*031 SS05*037 SS05*058	EPA-600 for Dissolved Metals of filtered aqueous	200.7 with acidified sample	Not Applicable	200.9 with acidified sample	Not Applicable
SS05*036	EPA-600 for Total Metals	200.7	Not Applicable	Not Applicable	Not Applicable

Table 3-3 Digestion Methods by Line Item Code and Analysis Technique (continued)

Line Item Code	Analysis Description	Aqueous Samples by ICPEs	Soils and Solids by ICPEs	Aqueous Samples by AA	Soils and Solids by AA
SS05*021 SS05*022 SS05*023 SS05*024 SS05*032 SS05*033	SW-846 for Total Metals of unfiltered aqueous	3010A	Not Applicable	As 7060A Pb 3020A Se 7740A Tl 3020A Sb 3005 Hg 7470A	Not Applicable
SS05*045 SS05*046 SS05*054 SS05*056	SW-846 for Dissolved Metals of filtered aqueous	3005A	Not Applicable	As 7060A Pb 3020A Se 7740A Tl 3020A Sb 3005 Hg 7470A	Not Applicable
SS05*047 SS05*048 SS05*049 SS05*050 SS05*055 SS05*057	SW-846 for Total Metals of solids	Not Applicable	3050A	Not Applicable	3050A Hg 7471A

* Denotes the latest revision of the PSA Module SS05.

Note: this table does not include microwave methods which may be acceptable for some of these determinations. These additional methods have not been included in this table because none of the audited labs used this technique for metals preparation.

3.19. Sample Preparation Raw Data

DRC Items 7-a and 7-b

Review Items: Deliverable Section Number 7.

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

Requirement Sources: GR01 Exhibit B § 4.11, GR01 Exhibit F § 4, SS05 Exhibit B §2.9, and requirements of base methods cited in SS05 Exhibit D Section 2.

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

Item 7-a and Item 7-b

Verify that Item 7-a and Item 7-b are each in compliance by performing the checks below.

- If omissions exist and they do not affect the ability to review the data, comment and qualify all applicable data with reason code [804].
- If omissions exist and they affect the ability to review the data, initiate a Non-

Compliance Notification and assign all applicable data with reason code [803]. Do not qualify data without the required information. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Item 7-a 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

Item 7-b Check that sufficient raw data are included in the SDP to allow manual calculations of the final reported sample results. Check this item as complete if raw data were sufficient to perform calculations for all previous items (e.g. 6-i).

3.20. Standards

DRC Items 8-a through 8-c

Review Items: Deliverable Section Number 8.

Objective: To confirm that reported data can be linked to traceable standards.

Requirement Sources: GR01 Exhibit B § 4.12, GR01 Exhibit F § 4 and SS05 Exhibit B § 2.10.

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

Item 8-a through Item 8-c

Verify that each Item 8-a through 8-c is in compliance by performing the Checks below.

- If omissions exist and they do not affect the ability to review the data, comment and qualify all applicable data with reason code [804].
- If omissions exist and they affect the ability to review the data, initiate a Non-Compliance Notification and assign all applicable data with reason code [803]. Do not qualify data without the required information. Continue assessment, when possible, of other Deliverable Sections. Return the SDP to ASD with Non-Compliance Notification(s) for all missing or revised information needed to complete data assessment.

Choose at least one standard out of each of the following categories (where applicable) for the following checks:

- ◇ Non-zero calibration standard for ICPES
- ◇ Non-zero calibration standard for GFAAS
- ◇ Non-zero calibration standard for CVAA
- ◇ Laboratory Control Sample

Perform the following Checks on each of the chosen standards:

- ◇ Find the tracking identifier on the raw data
- ◇ Locate the standard summary sheet for that identifier
- ◇ Check that the standard tracking identifiers on these standard summary sheets match those listed on instrument and preparation raw data.
- ◇ Check that all analyte solutions in these standard summary sheets include clear identification of the primary standard.
- ◇ Check that primary standards used to prepare each ICV are different from primary standards used to prepare calibration standards
- ◇ Check that standard summaries include expiration dates for secondary standards.

3.21. Instrument Raw Data

DRC Items 9-a through 9-d

Review Items: Deliverable Section Number 9.

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.

Requirement Source : GR01 Exhibit B § 4.13, GR01 Exhibit F § 4, and SS05 Exhibit B §2.11.

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

Item 9-a through Item 9-d

Verify that each Item 9-a through 9-c are in compliance by performing the Checks below for each Item 9-a through Item 9-d.

- If omissions exist and they do not affect the ability to review the data, comment and assign all applicable data with reason code **[804]**.
- If omissions exist and they affect the ability to review the data, initiate a Non-Compliance Notification and assign all applicable data with reason code **[803]**. Do not qualify data without the required information. If omissions affect the ability to continue with the data assessment, determine if other data or information are missing and return the SDP to ASD with a Non-Compliance Notification.

Item 9-a Verify that Item 9-a is in compliance by performing the Checks below.

- ◇ 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 the identification of five standards on the raw instrument data to determine if they are identified by standard tracking identifiers. Choose the five standards, including at least one from each of the following categories:
 - ⇒ Calibration standard
 - ⇒ ICV
 - ⇒ CCV
- ◇ Check that preparation blank and LCS data are clearly linked to the digestion batch.

Item 9-b Check instrument raw data printouts for area spectroscopist review, signature, and date on each instrument batch.

Item 9-c Check this item as complete if raw data were sufficient to perform calculations for all previous items (e.g. 6-i).

Item 9-d Record any information that indicates the batch QC samples were systematically prepared and analyzed differently from samples in the Analytical Batch.

3.22. **Electronic Data Deliverable (EDD)**

DRC Items 10-a through 10-c

Review Items: Deliverable Section Number 10.

Objective: To ensure that electronically-reported data are accurate.

Requirement Sources: GR01 Exhibit B 4, and SS05 Exhibit B §2.12.

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

Item 10-a through 10-c

See DA-GR01 for evaluation.

4. 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 SS05 is presented as Attachment 1.

5. REFERENCES

- Guidance for Radiochemical Data Validation, Draft RD4, October 4, 1995, prepared by Office of Transportation, Emergency Management & Analytical Services (EM 26), Office of Compliance and Program Coordination, Environmental Management, U.S. Department of Energy.
- Reason Codes for Data Assessment, Analytical Services Document
- General Data Quality Assessment Guidelines, DA-GR01-A-1

6. REVISION HISTORY

- The first draft of DA-SS05 was prepared by Carol Gies of Kaiser-Hill Analytical Services. This version followed an order considered logical for data assessment rather than the order of items on the DRC. Also, several sections had not yet been drafted.
- The first draft was then reviewed and edited by QuantaLex for completeness and accuracy of evaluation and action criteria
- The order of the DA-SS05 verification and validation sections was changed to follow the order of the SDP deliverable sections on the DRC to facilitate the referencing of items in the procedure.
- Final drafting of DA-SS05-v1 was completed by Norm Ross of Kaiser-Hill Analytical Services on December 6, 1997. This revision included: formatting for consistency with DA-GR01-v1, formatting to separate evaluation and action criteria, inclusion of new and revised reason codes, corrections and additions of evaluation and action criteria, and general editing.
- DA-SS05-v1 is to used for DRC examination and verification and validation of the first SDPs received according to PSA Module SS05.

Attachment 1: Data Quality Assessment Report Template

**SS05
Data Quality Assessment Report
Rocky Flats Environmental Technology Site**

RIN Number	Analytical Method/PSA Line Item	Validation Level

Analytical Laboratory	Assessment Performed by	Number of Samples/ Matrix.

Sample Numbers: _____

Quality Control Item	Reviewed (Y or N)	Non-Compliance Identified
General (Cover Page, Table of Contents, DRC Checklist, General SDP Requirements 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		

Y Item was reviewed or non-compliance was identified
N Item was not reviewed or non-compliance was not identified

Effective Date:
December 18, 1997

Verification and Validation Guidelines
for PSA Module SS05

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DA-SS05-v1-39

Action Items:

Comments:

Verification/Validation Signature _____

Date: _____

Reviewer Signature _____
(Validation Only)

Date: _____

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