



# **VERIFICATION AND VALIDATION GUIDELINES**

**FOR**

## **TOTAL URANIUM BY LASER INDUCED PHOSPHORESCENCE**

**DA-RC06-v1**

February 16, 1998

Approved: \_\_\_\_\_  
Analytical Services Division

Reviewed For Classification  
By: Roger S. Cichorz U/NU  
Date: February 16, 1998



## TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE NO.</u>
1. INTRODUCTION/SCOPE.....	1
2. DATA REVIEW CHECKLIST (DRC) EXAMINATION INSTRUCTIONS .....	1
<b>2.1. Examination of NA Replies</b> .....	1
<b>2.2. Examination of the Sample Narrative</b> .....	2
3. VERIFICATION AND VALIDATION INSTRUCTIONS .....	2
<b>3.1. Chain of Custody and Sample Preservation</b> .....	2
DRC Items 4-a through 4-g.....	2
<b>3.2. Sample Data Package Narrative Requirements</b> .....	2
DRC Items 5-a through 5-e.....	2
<b>3.3. Sample and QC Sample Results Summary</b> .....	3
DRC Item 6-a-1 through 6-a-5 .....	3
<b>3.4. QC Samples</b> .....	5
DRC Item 6-b-1 through 6-b-4.....	5
<b>3.5. Duplicate Samples</b> .....	6
DRC Item 6-c-1 through 6-c-4 .....	6
<b>3.6. Laboratory Control Sample Analysis</b> .....	7
DRC Item 6-d-1 and 6-d-2.....	7
<b>3.7. Preparation Blank</b> .....	8
DRC Item 6-e-1 through 6-e-3 .....	8
<b>3.8. Sample Preparation</b> .....	9
DRC Items 7-a through 7-d .....	9
<b>3.9. Standards Summary</b> .....	10
DRC Items 8-a through 8e.....	10
<b>3.10. Instrument Calibration Summary</b> .....	13
DRC Items 9-a through 9f.....	13
<b>3.11. KPA Raw Data Summary</b> .....	14
DRC Items 10-a through 10-e.....	14
<b>3.12. Electronic Data Deliverable (EDD)</b> .....	15
DRC Item 11-a through 11-c .....	15
4. INSTRUMENT CALIBRATION PACKAGE.....	16
<b>4.1. Structural Requirements</b> .....	16
Calibration DRC Item 1-a through 1-c .....	16
<b>4.2. Instrument Calibration Package General Requirements</b> .....	16
Calibration DRC Item 2-a through 2-d.....	16
<b>4.3. Instrument Calibration Package Calibration Curve</b> .....	17
Calibration DRC Item 3-a through 3-c .....	17

## TABLE OF CONTENTS

<b><u>SECTION</u></b>	<b><u>PAGE NO.</u></b>
<b>4.4. Instrument Background</b> .....	18
Calibration DRC Item 4-a .....	18
<b>4.5. Calibration Check</b> .....	18
Calibration Check DRC Item 5-a .....	18
5. DATA QUALITY ASSESSMENT REPORT PREPARATION.....	18
6. REFERENCES .....	19
7. REVISION HISTORY .....	19
Attachment 1: Data Quality Assessment Report Template.....	21

## 1. INTRODUCTION/SCOPE

This procedure presents those data assessment steps which are unique to PSA Module RC06, Total Uranium by Laser Induced Phosphorescence. This procedure is to be used in conjunction with the general guideline 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-RC06, until replaced by a more recent version, is applicable to all versions of the PSA Module RC06.

This procedure for the data quality assessment of RC06 Sample Data Packages is organized into the following Sections:

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

## 2. DATA REVIEW CHECKLIST (DRC) EXAMINATION INSTRUCTIONS

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

It is possible that several items in the DRC may not be applicable to a given data package, and those items may be marked as NA (not applicable). For the following items given in Table 2-1, enter ✓ in the ✓ column of the DRC to indicate that the NA response is accepted but not verified:

**Table 2-1 Non Applicable DRC Items**

Section 1 Items	Section 2 Items	Section 5 Items	Section 6 Items	Section 8 Items
1-d	4-b	5-c	a-4	a*
	4-e	5-d	b-3	b*
		5-e	c-4	

\*Note that not both items 8a and 8b can be NA, but either one may be.

2.1.1. For all other items with NA marked in the Reply column, enter X in the ✓ column to indicate that verification is required for this item.

## 2.2. Examination of the Sample Narrative

Read the sample narrative for information which may indicate additional items to be verified. Items to check include statements about data qualifiers, blank or reagent contamination, instrument calibration, variation of results from the method of standard additions, and/or sample handling problems.

## 3. VERIFICATION AND VALIDATION INSTRUCTIONS

The instructions contained in this section are to be used in conjunction with the general instructions for DRC Examination in procedure DA-GR01 which covers the sections common to all of the modules. This section includes specific instructions for performing verification and validation for Sample Data Packages for RC06 and corresponds to a DRC item.

### 3.1. Chain of Custody and Sample Preservation

#### ***DRC Items 4-a through 4-g***

**Review Items:** DRC, Deliverable Section Number 4; Deliverable Section Number 6: Form 1D, COC record, sample preparation/extraction log.

**Requirement Source:** GR01 Exhibit B Section 4.8 and RC06 Exhibit D Section 3.

**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 sample data package (SDP).

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

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

**Item 4d** Check for documentation that the sample pH was checked and if necessary adjusted to  $\leq 2$  and when adjusted the sample was allowed to remain in the original container for at least 24 hours prior to commencement of analysis.

- If the samples were not acid-preserved prior to receipt by the laboratory, comment and assign the reason code [703] to the applicable samples.
- Check for documentation that the sample pH was adjusted to  $\leq 2$  and the samples allowed to remain in the original container for at least 24 hours prior to commencement of analysis. If this was not done qualify all results as estimated[J 201].

### 3.2. Sample Data Package Narrative Requirements

#### ***DRC Items 5-a through 5-e***

**Review Items:** Sample Data Package Deliverable Section Number 5

**Objective:** Review the narrative for compliance to requirements, problems or unusual circumstances encountered in the analytical processing of samples and for information useful for validation of data.

**Requirement Source:** GR01 Exhibit B Section 4.9

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

Check that the SDP Narrative is present and at a minimum, the narrative shall address each of the following items, even if no deficiencies or unusual occurrences were experienced:

- Item 5-a** Synopsis of the methodology and analysis, including all standard operating procedures used and revisions.
- If this item is non-compliant, do not qualify any results. Comment and assign the reason code [805].
- Item 5-b** Descriptions of samples and any matrix interferences.
- If this item is non-compliant, do not qualify any results. Comment and assign the reason code [805].
- Item 5-c** Descriptions of all anomalies, caveats, deficiencies, interferences, reanalysis, and deviations from approved SOPs related to the analysis samples and any matrix interferences. Also to include the following:
- ◇ Description of required dilutions
  - ◇ Explanations of any QC deficiencies, missed holding times, or inability to achieve the RDLs
  - ◇ Explanations and descriptions of all deviations from routine protocols, including deviations from approved SOPs, detection limit modifications, etc.
  - ◇ Explanations for each item marked "N," on the Data Review Checklist
  - ◇ All other information that might affect data validation
  - If any of the above are non-compliant, do not qualify any results. Comment and assign the reason code [805].
- Item 5-d** Samples requiring reanalysis are identified with a reason for reanalysis, the original and reanalysis Analytical Batch Identification Numbers are included.
- If this item is non-compliant, do not qualify any results. Comment and assign the reason code [805].
- Item 5-e** 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.
- If this item is non-compliant, do not qualify any results. Comment and assign reason codes [227] and/or [805] as appropriate.

### 3.3. Sample and QC Sample Results Summary

#### ***DRC Item 6-a-1 through 6-a-5***

**Review Item:** Sample Data Package Deliverable Section Number 6 & 10.

**Objective:** Review the Samples section of Samples and QC Sample Results Summary for compliance to requirements and for information useful for validation of data.

**Requirement Sources:** GR01, Exhibit B/Section 4 and RC06, Exhibit B/Section 2

**Evaluation**

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

- Item 6-a-1** Verify that all samples and tests that were requested on the COC for this module have been analyzed and tested.
- If samples were not analyzed do not qualify any data. Address the deficiency in the Data Assessment Report.
- Item 6-a-2** Verify that sample results are arranged by Site sample number and for each sample and QC sample the results include the following:
- ◇ sample ID
  - ◇ analytes
  - ◇ analyte activities (see RC06 Exhibit E/Section 4 and Section 5.1 for Result Reporting Requirements)
  - ◇ analyte overall measurement uncertainties (2-sigma) in same units as the reported activity
  - ◇ "Analytical Batch ID" (Analytical Batch Identification Number)
  - Omissions or errors which do not affect your ability to review the data shall be documented with a comment and reason code [804].
  - Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [803] to all applicable data.
- Item 6-a-3** Verify that only one result is reported for each requested analyte.
- If more than one result is reported and neither is identified as "Do Not Use data", contact the CTR for instructions.
- Item 6-a-5** If the MDC is > the RDC, then for a given quarter, the only variable for this analysis is the sample size. If the sample size was not sufficiently large to meet the RDC, it will most likely be due to matrix interference's or the fact that the activity of the sample was so high, it was necessary to use a small volume of sample.
- If the RDC was not met because of small sample size due to matrix interferences, then the data is conditionally acceptable and assign the reason code [UJ 136].
  - If the activity of the sample was high and it exceeds the RDL, the data is acceptable.

**Evaluation**

*The following items apply to validation only:*

- Item 6-a-4** Verify that samples requiring reanalysis have been assigned a new analytical batch identification number and appropriate QA/QC is included.
- If data can not be produced to show the batch ID is different, reject those samples, comment and qualify all applicable data as [R 205].

**Item 6-a-5** MDC Calculation

Refer to Sample and QC Sample Results Summary of the SDP for the 1s (**SD**) of the blank population. Calculate at least one sample MDC according to the following:

$$MDC = \frac{[(4.65 * SD) * DIL]}{V}$$

where:

*SD* = Standard Deviation (1s) of the Previous Quarter Matrix Blanks

*DIL* = Dilution Factor

*V* = Sample Volume or Weight (Liters or Grams)

- If the calculated MDCs do not agree with the MDCs given in the data package, discontinue validation. Inspect all other SDP deliverables for other missing or incomplete information. Issue a Non-Conformance Notification for all noted deficiencies and assign reason code [**803**] to all applicable data. Return the SDP to ASD with the Non-Compliance Notification.

**Item 6-a-6** Verify that the sample results are within the calibration ranges of the calibration curves given and the data was collected on the same day.

- If non compliant, assign reason code [**J 228**] to all applicable data

3.4. **QC Samples**

***DRC Item 6-b-1 through 6-b-4***

**Review Item:** Sample Data Package Deliverable Section Number 6 & 10.

**Objective:** Review the Samples and QC Sample Results Summary for compliance to requirements and for information useful for validation of data.

**Requirement Sources:** GR01, Exhibit A & B/Section 4 and RC06, Exhibit B/Section 2

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

**Item 6-b-2** Verify that the required QC samples were included for each batch.

- If the Laboratory Control Sample, Laboratory Duplicate, or Preparation Blank were not run, at a minimum, comment and assign the reason code [**R 230**] to all applicable data. If any one single item is missing, qualify applicable data as follows:
  - If missing Duplicate only, qualify data as [**J 128**]
  - If missing LCS only, qualify data as [**R 174**]
  - If Missing Preparation Blank only, qualify data as [**R 175**]

Verify that a set of QC samples were run at 10% frequency per analytical batch.

- If this item is non-compliant, do not qualify any data. Comment and assign the reason code [**J 168**] to all applicable data.

- Item 6-b-3** Verify that all QC deficiencies are detailed in the narrative.
- If this item is non-compliant, do not qualify any data. Comment and assign the reason code [805] to all applicable data.

- Item 6-b-4** Verify that all sample results, including reanalysis, and the corresponding Analytical Batch QC sample results are acceptable and were reported.
- If this item is non-compliant, do not qualify any data. Comment and assign the reason code [801] to all applicable data.

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

- Item 6-b-1** Verify that for each QC sample (Duplicate, LCS and Preparation Blank), the QC type, QC Sample Identification, and the following required items are included:

The QC sample type is clearly identified and designated as follows:

- ◇ duplicate is the corresponding sample identification + "D", or "Duplicate"
- ◇ laboratory control sample is designated as "LCS"
- ◇ preparation blank is designated as "PB"

For each batch duplicate pair, the following additional information is reported:

- ◇ result of duplicate result equivalency test as defined in Item 6-c-2 below, including calculated values for F and E

For the "LCS", the following additional information is reported:

- ◇ LCS "SV" (standard value (SV) of the LCS, decayed to analysis date, if applicable)
- ◇ Uncertainty of LCS standard value (2-sigma)
- ◇ LCS "% Recovery"
- Omissions or errors in any of the 6-b-1 items which do not affect your ability to review the data shall be documented with a comment and reason code [804].
- Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [803] to all applicable data.

### 3.5. Duplicate Samples

#### **DRC Item 6-c-1 through 6-c-4**

**Review Item:** Sample Data Package Deliverable Section Numbers 6 & 10.

**Objective:** To determine a measure of laboratory precision, or degree of agreement of repeated measurements within acceptable concentration ranges.

**Requirement Sources:** GR01, Exhibit B Section 4 and RC06 Exhibit E Section 8

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

- Item 6-c-1** Verify that the results for the duplicate are reported separately from the corresponding sample.
- If this item is non-compliant, issue a Non-Compliance Notification to request the correct data. Do not qualify any data. Comment and assign the reason code [205] to all applicable data.
- Item 6-c-3** Verify that the MDC for each duplicate is reported and is < the RDC
- If these items are non-compliant, and the MDC > RDL due to laboratory error, comment and assign [UJ 136] to all applicable data.
- Item 6-c-4** Verify that if the MDC for each duplicate is reported and is > the RDC the exception for this deficiency is noted under DRC 6.c)4
- If the explanation for the MDC being greater than the RDL is not explained in DRC 6.a)5, do not qualify the results comment and assign the reason code [804] to all applicable data.

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

- Item 6-c-2** Confirm the results for the duplicate and the corresponding sample were equivalent, using the following RC01 equivalency test and control criteria [(F ≤ E \* 1.5), or F/E ≤ 1.5].

$$F = |S - R|$$

$$E = \sqrt{E_S^2 + E_R^2}$$

where,

- F = The absolute difference of the sample and duplicate activities
- S = Original sample activity
- R = Duplicate sample activity
- E = Propagated measurement uncertainty, of the difference, at 2-sigma
- E<sub>S</sub> = 2-sigma measurement uncertainty of sample activity
- E<sub>R</sub> = 2-sigma measurement uncertainty of Duplicate activity
- If the duplicate equivalency test does not pass and the sample is homogeneous, qualify the results and assign the reason code [J 235] to all applicable data.

### 3.6. Laboratory Control Sample Analysis

#### **DRC Item 6-d-1 and 6-d-2**

**Review Item:** Sample Data Package Deliverable Section Number 6.

**Objective:** To evaluate the Laboratory Control Sample (LCS) and determine if it serves as a monitor of the overall performance of each step during the analysis, including the sample preparation.

**Requirement Sources:** GR01, Exhibit B/Section 4 and RC06 Exhibit E/Section 9

**Evaluation** *The following items apply to verification and validation.*

- Item 6-d-1** Verify that LCS results are reported. Verify by calculation that percent recoveries (OV/CV (certified value)\*100) are within the control limits (90%-110%).
- If the laboratory control sample was not reported, issue a Non-Compliance Notification and assign the reason code [R 803] to all applicable data.
  - If the laboratory control sample does not pass the percent recovery qualify and assign the reason code [R 236] to all applicable data.

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

- Item 6-d-2** Verify that the following requirements have been met.
- ◇ Check that the LCS is of natural uranium and at an appropriate level for the samples in the Analytical Batch.
  - ◇ The LCS was prepared and analyzed in the same manner as the samples.
  - If the laboratory control sample does not pass all of these criteria, qualify the results and assign the reason code [J 234] to all applicable
- Item 6-d-3** Verify that the LCS is measured in the same calibration range as the samples.
- Assign reason code [J 234] if non compliant.

### 3.7. Preparation Blank

#### ***DRC Item 6-e-1 through 6-e-3***

**Review Item:** Sample Data Package Deliverable Section Number 6 & 10.

**Objective:** To verify that the Preparation Blank (PB) serves as a proper monitor to assess the extent of contamination introduced through sample preparation, tracer addition, and analysis.

**Requirement Sources:** GR01 Exhibit E/Section 7, RC06 Exhibit E/Section 7

**Evaluation** *The following items apply to verification and validation.*

- Item 6-e-1** Verify that at least one preparation blank consisting of distilled water was prepared and analyzed with every Analytical Batch of samples prepared, at 10 %frequency.
- If this item is non-compliant, do not qualify any data. Comment and assign the reason code [J 168] to all applicable data.

Verify that if the MDCs for the samples in an Analytical Batch met the analyte RDC, the activity of the preparation blank is less than or equal to the RDC

- If the preparation blank is not equal to or less than the RDL, qualify the results and assign the reason code [UJ 237] to all applicable data.

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

- Item 6-e-2** Verify that the data package contains the following information for the Blank population used for determining MDC:
- ◇ date of MDC calculation
  - ◇ a unique sample ID for each blank used

- ◇ date of analysis of each blank
- ◇ the calculated 1s (standard deviation) of the entire blank population
- ◇ the resulting MDC
- Omissions or errors which do not affect your ability to review the data shall be documented with a comment and reason code [804].
- Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [803] to all applicable data.

**Item 6-e-3** Unless specified in an applicable Addendum, verify that Samples and QC samples are NOT blank corrected.

- If this item is non-compliant, issue a Non-Compliance Notification to request the appropriate calculations and assign the reason code [804] to all applicable data.

### 3.8. Sample Preparation

#### ***DRC Items 7-a through 7-d***

**Review Items:** Sample Data Package Deliverable Section Number 7.

**Objective:** To determine that bench sheets and run logs have been filled out properly and to determine that proper sample preparation methods were performed.

**Requirement Sources:** RC06 Exhibit B/Section 2

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

**Item 7-a** Verify that benchsheets and/or preparation logs are included in the SDP.

- If benchsheets and/or preparation logs are not included, request the missing data in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [801] to all applicable data.

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

**Item 7-a** Verify that benchsheets and/or preparation logs are included and document the required items as follows:

- ◇ preparation start date
- ◇ "Analytical Batch ID"
- ◇ sample identifications
- ◇ QC sample type and identifications (unique LCS identification traceable to the Standards Summary Section)
- ◇ sample and QC sample "Gross Weight" and units (if applicable)
- ◇ "Tare Weight" and units, for the tare weight of any beakers (if applicable)
- ◇ sample dilutions, digestion volumes and units (if applicable)

- ◇ sample and QC aliquot
- ◇ final sample aliquot(i.e. sample aliquot taken for measurement in KPA cell)
- ◇ "Pipette ID" and dates of use (if applicable)
- ◇ "Balance ID" and dates of use (if applicable)
- ◇ methodology SOP #
- ◇ signatures and dates of all analysts and reviewers
- Omissions or errors in any of the above items which do not affect your ability to review the data shall be documented with a comment and reason code [804].
- Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [803] to all applicable data.

For soils, sediments, sludges and waste (which require homogenizing the sample prior to analysis), verify that the following additional information is reported:

- ◇ the approximate sample volume of the gross sample (as received)
- ◇ the aliquot size homogenized
- ◇ the aliquot size of dried, homogenized sample digested
- If any of these items are non-compliant, do not qualify any data. Comment and assign the reason code [240] to all applicable data.

### 3.9. Standards Summary

#### ***DRC Items 8-a through 8e***

**Review Items:** Sample Data Package Deliverable Section Number 8.

**Objective:** To verify that all standards meet the requirements of documentation and traceability to ensure reliable data.

**Requirement Sources:** RC06 Exhibit B/Section 2.11; GR01 Exhibit E/Section 6

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

**Item 8-a** Verify that the standard summary is included in the in the SDP.

- If the standard summary is not included, request the missing data in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [801] to all applicable data.

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

**Items 8-a** For primary standards that were diluted and used for tracers, LCS and any in-house prepared instrument calibration sources, verify that the following information for the diluted standard preparation is reported:

- ◇ date of preparation

- ◇ standard isotope is natural uranium
- ◇ standard type (LCS, Reference Cell solution, Calibration Source or Calibration Check)
- ◇ "Primary STD ID" (traceable to the certificate)
- ◇ "Primary SV" and units (certified value of the primary standard with the date of decay or certification)
- ◇ "Dilution" (e.g., 5/1000, for 5 mls diluted to 1 liter)
- ◇ "Diluted STD ID" (unique identification of the diluted standard)
- ◇ "Diluted SV" and units (certified value of the diluted standard with the date of decay)
- ◇ "Aliquot ID" (unique identification of each aliquot of diluted standard used for the LCS, prepared calibration standards, Reference Cell solution or calibration check)
- ◇ "Aliquot Size" and units (net weight/volume of diluted standard used for the LCS, prepared calibration standards, Reference Cell solution or calibration check)
- ◇ "Aliquot Concentration" and units (concentration and units for each aliquot of diluted standard used for the LCS, prepared calibration standards, Reference Cell solution or calibration check standard with the date of decay)
- ◇ "Primary STD Exp. Date" (expiration date)
- ◇ "Diluted STD Exp. Date" (expiration date)
- ◇ "Pipette ID" and dates of use (if applicable)
- ◇ "Balance ID" and dates of use (if applicable)
- ◇ methodology SOP
- ◇ signatures and dates of all analysts and reviewers
- Omissions or errors in any of the above items which do not affect your ability to review the data shall be documented with a comment and reason code [804].
- Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [803] to all applicable data.

**Item 8-b** For standards that were not diluted and used for LCS in-house prepared calibration standards, Reference Cell solution or calibration check standards, the following documentation must be reported:

- ◇ standard isotope
- ◇ standard type (LCS, Calibration standard, Reference Cell solution or Calibration Check))
- ◇ "Primary STD ID" (traceable to the certificate)
- ◇ "Primary SV" and units (certified value of the primary standard with date of decay or certification)
- ◇ "Primary STD Exp. Date" (expiration date)

- ◇ "Aliquot ID" (unique identification of each aliquot of primary standard used for the LCS, prepared calibration standards, Reference Cell solution of Calibration Check)
  - ◇ "Aliquot Size" and units (net weight/volume for each aliquot of primary standard used for the LCS, prepared calibration standard, Reference Cell solution, or Calibration Check.)
  - ◇ "Aliquot Activity" and units (activity and units for each aliquot of primary standard used for the LCS, prepared calibration standard,, Reference Cell solution, or Calibration Check with the date of decay)
  - ◇ "Pipette ID" and dates of use (if applicable)
  - ◇ "Balance ID" and dates of use (if applicable)
  - ◇ methodology SOP
  - ◇ signatures and dates of all analysts and reviewers
  - Omissions or errors in any of the above items which do not affect your ability to review the data shall be documented with a comment and reason code **[804]**.
  - Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code **[803]** to all applicable data.
- Items 8-a** Primary standard and secondary standard calculation should be performed to verify the values.
- If these calculations do not coincide with the standard values, issue a Non-Compliance Notification and assign the reason code **[R 243]** to all applicable data.
- Items 8-c** Verify that all standards certificates have been forwarded to the CTR upon first use
- If standard certificates have not been forwarded to the CTR, issue a Non-Compliance Notification to request the standard certificates. Do not qualify the data and assign the reason code **[801]** to all applicable data.
- Items 8-d** Verify that all standard identifications are traceable to the primary certificate, which are traceable to NIST.
- If standards are not traceable to the primary certificate or are not traceable to NIST, issue a Non-Compliance Notification, qualify and assign the reason code **[R 244]** to all applicable data.
- Items 8-e** Verify that all standards and sources traceable to NIST have not expired and are valid.
- Standards and tracers that have expired are not valid under any circumstances. Qualify and assign the reason code **[R 219]** to all applicable data.

### 3.10. Instrument Calibration Summary

#### ***DRC Items 9-a through 9f***

**Review Items:** Sample Data Package Deliverable Section Number 9.

**Objective:** Verify that the instrument calibration parameters are within control limits and establish an analytical curve relating the response of an instrument to a quantifiable characteristic of the analyte in known standards.

**Requirement Sources:** RC06 Exhibit B/Section 2.12, Exhibit E/Section 10

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

**Item 9-a** Verify the instrument calibration summary is included.

- If the instrument calibration summary is missing, do not qualify any data, comment and assign reason code **[801]** to all data.

**Item 9-c** Verify that the monthly calibration package has been forwarded to the CTR.

- If no documentation is available indicating the monthly calibration package has been received by the CTR, contact the CTR for instructions.

**Item 9-d** Verify the monthly Calibration Package identification is included in the Instrument Calibration Summary.

- If the Calibration Package identification is not included in the Instrument Calibration Summary or included on the DRC, contact the CTR for instructions. At a minimum, comment and assign reason code **[804]** to all data.

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

**Item 9-a** Verify the required following items are included for the KPA:

- ◇ Instrument Calibration Package identification with the syntax RC06CAL\_Lab ID\_ Date. Verify that calibration was done on each day samples were analyzed on the KPA.
- ◇ Date of Reference Cell solution preparation
- ◇ Standard ID of the Reference Cell solution, the Calibration Standards and the Calibration Check Standard
- ◇ Starting Background
- ◇ Print-out of the Calibration Curve clearly labeled
- ◇ Calibration Equation
- ◇  $R^2$ , linear regression coefficient of the Calibration Curve
- ◇ % Recovery of the Calibration Check standard
- If any of these items are con-compliant, initiate a Non-Compliance Notification to request the missing information. Do not qualify any data. Comment and assign the reason code **[803]** to the applicable data.

**Item 9-b** Verify that the calibration check standard is within the control limits (95% - 105%) each time it was measured.

- Assign reason code **[J 104]** if non compliant

- Item 9-e** Verify that  $R^2$ , the linear regression coefficient for the Calibration Curve is  $\geq .99$ .
- Assign reason code [J 103] if non compliant.
- Item 9-f** Verify that at least three calibration standards were used in each calibration range.
- Assign reason code [J 106] if non-compliant.

### 3.11. KPA Raw Data Summary

#### ***DRC Items 10-a through 10-e***

**Review Items:** Sample Data Package Deliverable Section Number 10.

**Objective:** Verify that sample raw data deliverable requirements have been met and that raw data are present in a form suitable for verification and validation. 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.

**Requirements Sources:** RC06 Exhibit B/Section 2

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

- Item 10-a** Check that KPA raw data summary for the RIN is present.
- If the KPA raw data summary is missing, issue a Non-Compliance Notification to request the missing data. Do not qualify any data, comment and assign reason code [801] to all data.

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

- Item 10-a** Check that preparation raw data (benchsheets and/or preparation logs) are included for all analyses performed and include the following:
- ◇ Site sample ID and/or respective Laboratory ID
  - ◇ date of analysis
  - ◇ data filename, if applicable
  - ◇ instrument ID
  - ◇ Instrument Calibration Package ID for applicable calibration data
  - ◇ "Analytical Batch ID"
  - ◇ sample "Final Aliquot Size"
  - ◇ number of laser pulses used
  - ◇ Reference Ratio
  - ◇ Intensity
  - ◇  $R^2$
  - ◇ Standard ID and aliquot added for each measurement by Standard Additions.
  - ◇ Report the last four items for all measurements of the method of Standard Additions.
  - ◇ analyst and reviewer's signature and date

- Omissions which do not affect your ability to review the data shall be documented with a comment and reason code [804].
  - Other omissions or errors essential for data assessment shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [803] to all applicable data.
- Item 10-b** Verify that the individual sample data were reviewed, signed and dated by the responsible analytical chemist and found to be acceptable.
- If the data have not been reviewed and signed, comment and assign the reason code [804] to all applicable data.
- Item 10-c** Verify there is sufficient raw data included to allow manual calculation of the final sample concentration, measurement uncertainty, MDC, and chemical recovery..
- If this item is non-compliant, issue a Non-Compliance Notification to request the missing information. Do not qualify any data. Comment and assign the reason code [803] to all applicable data.
- Item 10-d** Verify that the Instrument Calibration Package identification or the dates of the calibration are included with the raw data.
- If this item is non-compliant, issue a Non-Compliance Notification to request the missing information. Do not qualify any data. Comment and assign the reason code [803] to all applicable data.
- Item 10-e** Verify all QC samples were analyzed in the same manner as the samples in the Analytical Batch, in the same time frame, and using the same instrument calibration parameters, and instrument analysis algorithms.
- If these items are non-compliant, do not qualify any data. Comment and assign the reason code [234] to all applicable data.

### 3.12. Electronic Data Deliverable (EDD)

#### ***DRC Item 11-a through 11-c***

**Review Items:** Sample Data Package Deliverable Section Number 11.

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

**Requirement Sources:** GR01 Exhibit B 4, and RC06 Exhibit B/Section 2.14.

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

#### **Item 11-a through 11-c**

See DA-GR01 for evaluation.

## 4. INSTRUMENT CALIBRATION PACKAGE

### 4.1. Structural Requirements

#### ***Calibration DRC Item 1-a through 1-c***

**Review Items:** Deliverable Instrument Calibration Package Section Number 1

**Objective:** Ensure that the instrument calibration data package is provided for all reported data and that the data is consistent with the results reported.

**Requirement Sources:** RC06, Exhibit B, Section 4.1 and Appendix B-1

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

**Item 1-a** Check that the instrument calibration package was assigned an identification using the syntax of RC06CAL\_LabID\_Date.

- If this item is non-compliant, initiate a Non-Compliance Notification to request the missing information. Do not qualify any data. Comment and assign the reason code [804] to all applicable data.

**Item 1-b** Check that the instrument calibration package contains the following sections in the following order. (1) Cover Page, (2) Data Review checklist-RC06 Instrument Calibration Package, (3) Narrative, and (4) Instrument Calibration and Raw Data.

- If these items are non-compliant, issue a Non-Compliance Notification to request the missing information. Do not qualify any data. Comment and assign the reason code [804] to all applicable data.

**Item 1-c** Check that the structural requirements specified in RC06 have been met. All discrepancies were identified and documented, accordingly.

- If these items are non-compliant, issue a Non-Compliance Notification to request the missing information. Do not qualify any data. Comment and assign the reason code [804] to all applicable data.

### 4.2. Instrument Calibration Package General Requirements

#### ***Calibration DRC Item 2-a through 2-d***

**Review Items:** Deliverable Instrument Calibration Package Section Number 2

**Objective:** Ensure that the instrument calibration data package is provided for all reported data and that the data is consistent with the results reported.

**Requirement Sources:** RC06, Exhibit E, Section 10, RC06 Appendix B-1

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

**Item 2-a** Check that the instrument calibration package was completed and verified to be acceptable.

- If this item is non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data. Comment and assign the reason code [803] to all applicable data.
- Item 2-b** Check that the instrument calibration was performed in the order (1) background (2) calibration curve (3) calibration check
- If these items are non-compliant, do not qualify any data. Comment and assign the reason code [129] to all applicable data
- Item 2-c** Check that all sources used for the calibration curve and calibration check were valid and NIST traceable and were sent to the CTR upon first use.
- If the standard is expired, issue a Non Compliance Notification. Comment and assign the reason code [NJ 219].
  - If standards are not traceable to the primary certificate or are not traceable to NIST, issue a Non-Compliance Notification. Comment and assign the reason code [NJ 244] to all applicable data.
- Item 2-d** Check that all data has been reviewed and certified as accurate. The date and signatures of all analyst and reviewers of the data are included.
- If these items are non-compliant, issue a Non-Compliance Notification to request the missing deficiency report in the narrative. Do not qualify any data. Comment and assign the reason code [804] to all applicable data.

#### 4.3. Instrument Calibration Package Calibration Curve

##### ***Calibration DRC Item 3-a through 3-c***

**Review Items:** Deliverable Calibration Curve Instrument Calibration Package Section Number 3

**Objective:** To ensure that the calibration curves used for sample analysis meet requirements.

**Requirement Sources:** RC06, Exhibit E, Section 10, RC06 Appendix B-1

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

##### **Item 3-a to 3-c**

The calibration curve raw data are included and document the required items as follows:

- ◇ The range of the calibration curves is appropriate for samples analyzed..
- ◇ At least three standards were used to generate the calibration curve for each range.
- ◇ The calibration curve passes through zero.
- ◇ The  $R^2$  of the calibration curve is  $\geq .99$ .
- If any or all of the above items are missing assign reason code [803] and initiate a Non-Compliance Notification to request the data..
- If the calibration curve data is present but does not meet any or all of the criteria, the reviewer will exercise professional judgment to determine if the calibration is rejected [R 228] or if this calibration will produce results that are estimates only[J 228].

#### 4.4. Instrument Background

##### ***Calibration DRC Item 4-a***

- Review Items:** Deliverable Backgrounds Instrument Calibration Package  
Section Number 4
- Objective:** To ensure that the KPA used for sample analysis IS capable of  
producing quality results.
- Requirement Sources:** RC06 , Exhibit E, Section 10, RC06 Appendix B-1
- Evaluation** *The following items apply to validation only:*
- Item 4-a** The background raw data are included and document the required items as  
follows:
- ◇ The Background was collected prior to analysis and is acceptable with  
respect to the desired detection limit. Backgrounds will have to be  
examined by requesting the control chart of instrument backgrounds for  
the previous quarter.
  - ◇ The instrument background determined should fall within 3sigma limits of  
the mean background from the control chart.
  - If the KPA has not been background calibrated to meet the above  
requirements, initiate a Non-Compliance Notification. Do not qualify any  
data. Comment and assign the reason code [R246] to all applicable data.

#### 4.5. Calibration Check

##### ***Calibration Check DRC Item 5-a***

- Review Items:** Deliverable Calibration Check, Exhibit B, Section 4.2.3
- Objective:** To ensure that the KPA used for sample analysis is capable of  
producing quality results.
- Requirement Sources:** RC06 , Exhibit E, Section 10.6.3, RC06 Appendix B-1
- Evaluation** The following items apply to validation only:
- Item 4-a** The calibration check standard raw data are included and document the  
following:
- The Calibration Check Standard must be within 5% of the known value.
- If the Calibration Check Standard is not with  $\pm 5\%$  of the
  - known value, assign the reason code [R 104].

### 5. DATA QUALITY ASSESSMENT REPORT PREPARATION

Prepare a Data Quality Assessment Report in accordance with the criteria established in the General Data Assessment guidelines presented in DA-GR01. The template to be used for all Data Quality Assessment Reports for RC06 is presented as Attachment 1.

## 6. 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

## 7. REVISION HISTORY

- *DA-RC06-v1*, prepared by Mary Ely of Kaiser-Hill Analytical Services, is the first issue of this procedure. *DA-RC01-v1* was used for verification and validation of the first SDPs received according to PSA Module RC06.

This Page Intentionally Left Blank

**Attachment 1: Data Quality Assessment Report Template**  
**RC06**  
**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		
QC Sample Results		
Duplicate Sample Results		
Laboratory Control Results		
Preparation Blank Results		
Standards Summary		
Instrument Calibration Summary		
Counting Raw Data Summary		
EDD		
<b>INSTRUMENT CALIBRATION PACKAGE</b>		
Structural Requirements		
General Requirements		
Calibration		
Backgrounds		
Check Source		
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

**Action Items:**

**Comments:**

Verification/Validation Signature \_\_\_\_\_

Date: \_\_\_\_\_

Reviewer Signature \_\_\_\_\_

Date: \_\_\_\_\_

*(Validation Only)*