



**VERIFICATION AND VALIDATION
GUIDELINES
FOR
ISOTOPIC DETERMINATIONS
BY
ALPHA SPECTROMETRY**

DA-RC01-v1

February 13, 1998

Approved: _____
Analytical Services

ADMIN RECORD

Reviewed For Classification
By: Roger S. Cichorz U/NU
Date: February 13, 1998

1/33

SW-A-006166

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE NO.</u>
1. INTRODUCTION/SCOPE	1
2. DATA REVIEW CHECKLIST (DRC) EXAMINATION INSTRUCTIONS	1
2.1. Examination of NA Replies	1
2.2. Examination of the Sample Narrative	2
3. VERIFICATION AND VALIDATION INSTRUCTIONS.....	2
3.1. Chain of Custody, Holding Times, and Sample Preservation	2
DRC Items 4-a through 4-g	2
3.2. Sample Data Package Narrative Requirements	3
DRC Items 5-a through 5-e	3
3.3. Samples	4
DRC Items 6-a-1 through 6-a-5	4
3.4. QC Samples	6
DRC Items 6-b-1 through 6-b-4	6
3.5. Duplicate Samples	8
DRC Items 6-c-1 through 6-c-4.....	8
3.6. Laboratory Control Sample Analysis	9
DRC Items 6-d-1 and 6-d-2.....	9
3.7. Preparation Blank	10
DRC Items 6-e-1 through 6-e-3.....	10
3.8. Sample Preparation	11
DRC Items 7-a through 7-d.....	11
3.9. Standards Summary	14
DRC Items 8-a through 8e.....	14
3.10. Instrument Calibration Summary	16
DRC Items 9-a through 9c.....	16
3.11. Counting Raw Data Summary	20
DRC Item 10-a through 10-f	20
3.12. Electronic Data Deliverable (EDD)	22
DRC Item 11-a through 11-c.....	22
4. INSTRUMENT CALIBRATION PACKAGE.....	22
4.1. Structural Requirements	22
Calibration DRC Items 1-a through 1-c	22
4.2. Instrument Calibration Package General Requirements	23
Calibration DRC Items 2-a through 2-d	23
4.3. Instrument Calibration Package Energy Calibration	24

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE NO.</u>
Calibration DRC Item 3-a through 3-e.....	24
4.4. Instrument Calibration Package Backgrounds.....	24
Calibration DRC Item 4-a.....	24
4.5. Instrument Calibration Package Efficiency Determinations.....	25
Calibration DRC Item 5-a through 5-e.....	25
5. DATA QUALITY ASSESSMENT REPORT PREPARATION.....	26
6. REFERENCES.....	26
7. REVISION HISTORY.....	26
Attachment 1: Data Quality Assessment Report Template.....	27

1. INTRODUCTION/SCOPE

This procedure presents those data assessment steps which are unique to PSA Module RC01, Isotopic Determinations by Alpha Spectrometry. 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-RC01, until replaced by a more recent version, is applicable to all versions of the PSA Module RC01.

This procedure for the data quality assessment of RC01 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 contained in this section are specific to PSA Module RC01 for Isotopic Determinations by Alpha Spectrometry. 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 \checkmark in the \checkmark 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 Items	Section 4 Items	Section 5 Items	Section 6 Items
1-d	4-b	5-c	6-a-4,5
	4-e	5-d	6-b-3,4
		5-e	6-c-4
			6-e-2,3

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

2.2. Examination of the Sample Narrative

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

3. VERIFICATION AND VALIDATION INSTRUCTIONS

The instructions contained in this section are specific to PSA Module RC01 for isotopic analyses. The instructions in this section are to be used in conjunction with the general instructions for DRC Examination found in Analytical Services Procedure DA-GR01. The remainder of this section includes specific instructions for performing verification and validation activities for Sample Data Packages generated under PSA Module RC01. Each section corresponds to a DRC Checklist section that may contain multiple item numbers. These item numbers are referenced within each section of this procedure.

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

DRC Items 4-a through 4-g

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

Requirement Source: GR01 Exhibit B Section 4.8 and RC01 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 4-d Check for documentation that the sample pH was adjusted to ≤ 2 and/or the temperature was maintained at 4°C prior to receipt by the laboratory.

- If samples were not acid-preserved or 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 Verify that the maximum hold time of 90 days was not exceeded.

- If samples were not analyzed within the 90 day hold time. Do not qualify the data, comment and assign the reason code [101] to all applicable samples.

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-e

Review Items: DRC, 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 addresses 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 of samples and any matrix interferences. Also to include the following:

- ◇ Description of required dilutions
- ◇ Explanations of any QC deficiencies, 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. Samples

DRC Items 6-a-1 through 6-a-5

Review Item: DRC, 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 RC01, 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 PSA module have been analyzed and tested

- If sample 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 the results (for each sample and QC sample) include the following:

- ◇ sample ID
- ◇ analytes
- ◇ analyte activities (see RC01 Exhibit E/Section 4 for Result Reporting Requirements)
- ◇ analyte overall measurement uncertainties (2-sigma) in same units as the reported activity
- ◇ analyte MDAs (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 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 Verify that the MDA for each sample is reported and is \leq the RDL.

If the MDA is $>$ the RDL, and a reduced aliquot size was used due to high or significant activity and the criteria contained in Item 6-a-5 of the DRC were met, then the following does not apply:

- If the MDA exceeds the RDL, comment and qualify all applicable data as [UJ 136].

- If the MDA is > the RDL and the deficiency is not reported in the narrative, assign the reason code [805] to all applicable data.

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 If the MDA is > the RDL and the samples and duplicates were prepared with a reduced aliquot size due to high or significant activity, verify that the following criteria were met:

- ◇ The net counts per second (CPS) for the analyte ROI are > 100 times the background (same units same ROI).
- ◇ Tracer chemical recoveries are within the acceptable limits.
- ◇ The Continuing Calibration Checks for the respective spectra are within the acceptable limits.
- If any of these items are non-compliant, and the MDA > RDL due to laboratory error, use professional judgment to determine if the non compliance affects the data and at a minimum assign the reason code [UJ 136] to all applicable data.

MDA Calculation (No Blank Subtraction): The following equation shall be used to calculate the MDA when blank subtraction is not needed. Except as specified above, the analyte MDAs shall be less than or equal to the respective RDL and the Laboratory shall optimize the below-listed factors. Calculate at least one sample MDA using the following equation:

$$MDA = \frac{2.71 + 4.65\sqrt{BKG}}{T * EFF * V * Y * 2.22}$$

where,

- BKG = total background counts
- T = count time in minutes
- EFF = detector efficiency
- V = sample aliquot size (liters or grams)
- Y = tracer chemical recovery

Units are in pCi/l or pCi/g

MDA Calculation (Filter Program): The following equation shall be used to calculate the MDA for the filter program. Calculate at least one sample MDA using the following equation:

$$MDA = \frac{1}{VF} \left(3.29 \sqrt{\left(\frac{S_{\bar{Y}_{TG}}(CPM)}{EFF * Y} \right)^2 + S_{\bar{Y}_{TG}}(DPM)^2} + \frac{2.71}{EFF * Y * T} \right)$$

where,

- $S_{\bar{Y}_{TG}}$ = standard deviation from the trimmed mean from the blank population

- T = sample count time in minutes
EFF = detector efficiency
VF = volume fraction
Y = tracer chemical recovery

Units are in DPM only

MDA Calculation (Surface Water Program): The following equation shall be used to calculate the MDA for the surface water program. Calculate at least one sample MDA using the following equation:

$$MDA = \frac{1}{2.22 * VF} \left(3.29 \sqrt{\left(\frac{S_{\bar{Y}_{rg}}(CPM)}{EFF * Y} \right)^2 + S_{\bar{Y}_{rg}}(DPM)^2} + \frac{2.71}{EFF * Y * T} \right)$$

where,

- $S_{\bar{Y}_{rg}}$ = standard deviation from the trimmed mean from the blank population
T = sample count time in minutes
EFF = detector efficiency
VF = volume fraction (liters)
Y = tracer chemical recovery

Units are in pCi/l

- If MDAs have been calculated wrong, whether the parameters have been entered wrong or there has been a calculation error issue, 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.

3.4. QC Samples

DRC Items 6-b-1 through 6-b-4

Review Item: DRC, 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 RC01, 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 were reported.

- If this item is non-compliant, address the deficiency in the Data Assessment Report using professional judgment to qualify the data. Omissions or errors which do not affect your ability to review the data shall be documented, at a minimum, with 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.

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

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 Items 6-c-1 through 6-c-4

Review Item: DRC, Sample Data Package Deliverable Section Number 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 RC01 Exhibit E/Section 8

Evaluation: *The following items apply to 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 and assign the reason code [205] to all applicable data.

Item 6-c-3 Verify that the MDA for each duplicate is reported and is < the RDL

- If these items are non-compliant, and the MDA > RDL due to laboratory error, comment and assign [UJ 136] to all applicable data.

Item 6-c-4 Verify that if the MDA for each duplicate is reported and is > the RDL the exception for this deficiency is noted under DRC 6.a)5

- If the explanation for the MDA 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 items apply 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 [$F \leq (E * 1.5)$, or $F/E \leq 1.5$].

$$F = |S - R|$$

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

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_S = 2-sigma measurement uncertainty of sample activity
- E_R = 2-sigma measurement uncertainty of Duplicate activity

- If the duplicate equivalency test does not pass and the sample is homogeneous, qualify the results, comment and assign the reason code [J 235] to all applicable data in analytical batch.

3.6. Laboratory Control Sample Analysis

DRC Items 6-d-1 and 6-d-2

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

Objective: To determine the overall performance of each step during the analysis, including the sample preparation.

Requirement Sources: GR01, Exhibit B/Section 4 and RC01 Exhibit E/Section 9

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

Item 6-d-2 Verify that the LCS met the same tracer recovery requirement as the samples.

- If the laboratory control sample does not meet the required tracer recovery requirements, qualify the results as [R 242].

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

Item 6-d-1 Verify that LCS results are reported and verify by calculation that percent recoveries ($OV \setminus CV$ (certified value)*100) are within the control limits (75%-125%).

- If the laboratory control 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.

Verify that the observed value (OV) is within plus or minus three standard deviations of the standard value (SV).

- If the laboratory control sample does not pass, qualify and assign the reason code [R 132] to all applicable data.

Item 6-d-2 Verify that the following requirements have been met.

- ◇ Check that the LCS is of the same analyte as the sample analyte and is at an appropriate level for the samples in the Analytical Batch.
- ◇ The activity of the LCS aliquot for near background level samples for Pu and Am should not exceed 5 dpm, and for U, it did not exceed 10 dpm for each isotope.
- ◇ The LCS was spiked with an approved tracer (refer to Exhibit D and Exhibit E/Section 11 of RC01) at the same appropriate level as the samples in the Analytical Batch.
- ◇ The LCS was prepared and analyzed in the same manner as the samples.
- ◇ The LCS was counted for the same count duration as the samples.
- If the laboratory control sample does not pass all of these criteria, qualify the results as [J 234] for all applicable data.

3.7. Preparation Blank

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

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

Objective: To assess the extent of contamination introduced through sample preparation, tracer addition, and analysis.

Requirement Sources: RC01 Exhibit E/Section 7, RC01 Exhibit D/Section 7

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

Item 6-e-1 Verify that the preparation blank meets the following requirements:

- ◇ At least one preparation blank consisting of ASTM Type II water was prepared and analyzed with every Analytical Batch of samples prepared at a minimum of 10 % frequency.
- ◇ Preparation blanks met the same tracer chemical recovery and Continuing Calibration Check requirements as the samples.
- If these items are non-compliant, do not qualify any data. Comment and assign the reason code [168] for required frequency not met and/or [234] to samples if blank method requirements were not meet.

If the MDAs for the samples in an Analytical Batch met the analyte RDL, verify that the activity of the preparation blank is less than or equal to the RDL

- If the preparation blank activity is not \leq the RDL, qualify the results [UJ 136] to all applicable data.

Item 6-e-2 Surface water, Effluent air filters and Ambient air filter programs require blank correction of data.

- ◇ Verify that blank correction for Pu-238, Pu-239/240, Am-241, U-234, U-235 and U-238 was performed by establishing a matrix specific blank population for each isotope.
- ◇ Verify that Matrix specific blank population must consist of at least eight but not more than thirty individual blank results (As additional blank samples are analyzed the new values will be added to the population).
- If the blank population was established and not included in the data package issue a Non-Compliance Notification, qualify as [NJ 238] to all applicable data.
- If a preparation blank population has not been established or does not meet the above requirements, comment and assign the reason code [NJ 238] to all applicable data.

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

Item 6-e-1 Verify that the preparation blank meets the following requirements:

- ◇ The Preparation Blank MDA calculation was based on the greatest sample volume or weight for the entire Analytical Batch.
- ◇ Preparation blanks were spiked at the same appropriate level as the samples with an approved tracer.

- ◇ Preparation blanks were counted for at least the same count duration as the samples.
- If these items are non-compliant, do not qualify any data. Comment and assign the reason code [234]

Item 6-e-3 Calculate the Winsorized mean and the standard deviation of the mean blank population using the following:

The Winsorized mean of this population will be computed and subtracted from the sample results prior to reporting the results to RFETS.

The following formula must be used to compute the Winsorized mean:

$$\bar{y}_{wg} = \frac{1}{n} \{ (g + 1) y_{g+1} + \sum_{i=g+2}^{n-g-1} y_i + (g + 1) y_{n-g} \}$$

where

$y_1 \leq y_2 \leq \dots \leq y_n$ is the ordered set of blank values.

\bar{y}_{wg} = the g-times Winsorized Mean

n = number of blanks

g = (n / 4) rounded to nearest integer and truncate integer result

y = nth blank

◇ The Winsorized Mean squared deviations:

$$SSD_{wg} = (g+1)(y_{g+1} - \bar{y}_{wg})^2 + \sum_{i=g+2}^{n-g-1} (y_i - \bar{y}_{wg})^2 + (g+1)(y_{n-g} - \bar{y}_{wg})^2$$

◇ The Standard Deviation of the Trimmed Mean

$$S_{\bar{y}_{TG}} = \sqrt{\frac{SSD_{wg}}{h(h-1)}}$$

h = n-2g

◇ The Standard Deviation of the Trimmed Mean in cpm and dpm

$$SB(\text{dpm}) = S_{\bar{y}_{TG}} \text{ for } y1(\text{dpm}) \qquad SB(\text{cpm}) = S_{\bar{y}_{TG}} \text{ for } y1(\text{cpm})$$

- If the preparation blank population has not been established using the Winsorized mean, does not meet the above requirements or has been calculated wrong, issue a Non-Compliance Notification, comment and qualify all applicable data as [NJ 239].

3.8. Sample Preparation

DRC Items 7-a through 7-d

Review Items: DRC, 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: RC01 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 and QC sample "Net Weight/Volume" (initial net aliquot size and units)
- ◇ sample dilutions, digestion volumes and units (if applicable)
- ◇ "Tracer ID" (unique tracer aliquot identification for each sample and QC sample spiked in the analytical batch that is traceable to the Standards Summary Section)
- ◇ copy of current blank population for samples requiring blank correction
- ◇ "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
- ◇ the ratio of sample weight as received (wet)/sample weight dried
- If any of these items are non-compliant, do not qualify any data. Comment and assign the reason code [240] to all applicable data.

Verify that a copy of the electroplating or microprecipitation preparation log for each sample and QC sample is included in this Deliverable Section and includes the following information:

- ◇ date of preparation
- ◇ sample/QC sample ID
- ◇ planchet or filter paper ID (if different from sample ID)
- ◇ electroplating cell ID (if applicable)
- ◇ methodology SOP
- ◇ analyst and reviewer's signature and date
- If any of 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 [241] to all applicable data.

- Item 7-d** Verify that the volume or weight used to calculate the Preparation Blank activity and MDA (pCi/g or pCi/l) did not exceed the maximum volume or weight of sample for the entire Analytical Batch.
- If this item is non-compliant, do not qualify any data. Comment and assign the reason code [234] to all applicable data.

- Item 7-b** Verify that the analysis of plutonium, uranium, americium, and thorium, for the internal tracer addition methods, are acceptable according to the following:
- ◇ Tracer solutions were prepared so that the overall propagated uncertainty at the 2-sigma confidence level did not increase by more than 3% over the uncertainty of the primary SRM.
 - ◇ The FWHM resolution for each sample and QC sample tracer peak are <80 keV.
 - ◇ The tracer peak centroid is ± 40 keV of the expected.
 - ◇ The following are acceptable tracer isotopes:
U-232, Pu-242 or Pu-236, Am-243 or Cm-244, and Th-229 or Th-234
 - If any of these items are non-compliant, do not qualify any data. Comment and assign the reason code [242] to all applicable data.
 - ◇ The uranium, plutonium, americium and thorium target analytes are chemically separated from each other and from the rest of the sample matrix.
 - Verify that the above analytes have been completely separated from each other by examining the spectral printouts. If the target analytes are not chemically separated for samples and QC samples, qualify, and assign the reason code [R 247] to all applicable data.

Verify that the tracer recovery for U analyses is > 30% but < 110%. The

tracer recovery for Pu and Am analyses is > 20% but < 110%.

Use the following equation to calculate at least one tracer chemical recovery:

$$Y = \frac{DPM}{CV} = \frac{C_T}{T_S * EFF} * \frac{1}{CV}$$

where,

- DPM = observed disintegrations per minute of the tracer
- C_T = total counts in the tracer isotope ROI
- T_S = sample count time in minutes
- EFF = detector efficiency
- CV = certified value decayed to the date of analysis of the tracer aliquot in dpm

- If the tracer recoveries have been calculated wrong, whether the parameters have been entered wrong or there has been a calculation error issue, 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.
- If the tracers for samples and QC samples do not meet the required recoveries, professional judgment should be used to determine the effect this has on the data. At a minimum, qualify, and assign the reason code [J 242] to all applicable data.

Item 7-c Verify that all samples and QC samples in each analytical batch were spiked at the same appropriate tracer level, and were prepared concurrently and in the following manner:

- ◊ The standard material used to prepare the tracer solutions are valid (not expired) and traceable to NIST.
- ◊ The isotopic tracer aliquot has dpm values appropriate for the activity of the sample aliquot analyzed.
- ◊ The activity of the tracer aliquot for near background level samples for Pu and Am do not exceed 5 dpm, and for U, does not exceed 10 dpm for each isotope.
- If any of these items are non-compliant, qualify all applicable data as [J 242].

3.9. Standards Summary

DRC Items 8-a through 8e

Review Items: DRC, 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: RC01 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
- ◇ standard type ("LCS", "Tracer" or "Calibration Source")
- ◇ "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 tracer, LCS or prepared calibration source)
- ◇ "Aliquot Size" and units (net weight/volume of diluted standard used for the tracer, LCS or prepared calibration source)
- ◇ "Aliquot Activity" and units (activity and units for each aliquot of diluted standard used for the tracer, LCS or prepared calibration source 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 tracers, LCS and any in-house prepared calibration sources, the following documentation must be reported:

- ◇ standard isotope
- ◇ standard type ("LCS", "Tracer" or "Calibration Source")
- ◇ "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 tracer, LCS or prepared calibration source)
 - ◊ "Aliquot Size" and units (net weight/volume for each aliquot of primary standard used for the tracer, LCS or prepared calibration source)
 - ◊ "Aliquot Activity" and units (activity and units for each aliquot of primary standard used for the tracer, LCS or prepared calibration source 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 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.
- Items 8-a** Diluted primary standards 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 qualify all applicable data [R 243].
- Items 8-c** Verify that all standard 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 and qualify all applicable data as [R 244].
- Items 8-e** Verify that all standards and sources traceable to NIST have not expired and are valid.
- If standards and tracers have expired, qualify all applicable data as [R 219].

3.10. Instrument Calibration Summary

DRC Items 9-a through 9c

Review Items: DRC, Sample Data Package Deliverable Section Number 9.

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

Requirement Sources: RC01 Exhibit B/Section 4, Exhibit E/Section 8

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

- Item 9-a** Verify the instrument calibration summary includes a summary of the energy calibration, backgrounds and efficiency determinations.
- If any of part of 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 each alpha spectrometry detector used to report results:

Energy Calibration

- ◇ instrument and detector ID
- ◇ date of the energy calibration
- ◇ "Energy Calibration Source ID" and "Expiration Date"
- ◇ energy calibration isotopes
- ◇ energy versus channels "Calibration Curve Equation", including the slope and Y-intercept for linear equations and appropriate corresponding information for higher order equations if applicable
- ◇ alpha spectrometry detector "Energy Range"
- ◇ analyst and reviewer's signature and date
- ◇ copy of instrument run log
- 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.

Backgrounds

- ◇ instrument and detector ID
- ◇ date of the background analysis

- ◇ "Count Time"
- ◇ respective "Start" and "End" "ROI" (in channels or energy)
- ◇ respective ROI "Background Counts" or "Background" counts/unit time
- ◇ "Background Error" and confidence level
- ◇ channel-by-channel printout of the background spectrum
- ◇ analyst and reviewer's signature and date
- ◇ copy of instrument run log
- 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.

Efficiency Calibrations

- ◇ instrument and detector ID
- ◇ date of the efficiency analysis
- ◇ "Efficiency Source ID", "Certified Value" with date of certification, and "Expiration Date"
- ◇ "Count Time"
- ◇ efficiency isotope
- ◇ respective "Start" and "End" "ROI" (in channels or energy)
- ◇ respective ROI "Net Counts" or "Net" counts/unit time
- ◇ "Efficiency", "Efficiency Error" and confidence level
- ◇ channel-by-channel printout of the efficiency spectrum
- ◇ analyst and reviewer's signature and date
- ◇ copy of instrument run log
- ◇ The detector resolution is reported as part of the instrument calibration based on the 239, 240Pu or 241Am peak at ≥ 2000 net counts.
- 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.

Verify all alpha spectrometry detectors were calibrated for the specific analytes of interest and each detector used for analysis of Site samples were calibrated on a monthly basis (at a minimum), or more frequently, if required.

- If the alpha spectrometry system was not calibrated at least monthly, issue a Non-Compliance Notification. Do not qualify any data. Comment and assign the reason code **[129]** to all applicable data.

Verify that the order of performing the Instrument Calibration was (1) Energy Calibration, (2) Background determinations and (3) Detector

Efficiency determinations.

- If the alpha spectrometry system was not calibrated in the above order, issue a Non-Compliance Notification. Do not qualify any data. Comment and assign the reason code [129] to all applicable data.

Item 9-b Verify the energy, background and efficiency calibrations are with-in control limits according to the following:

Energy Calibration Control Limits

- ◇ The energy calibration for each detector shall be performed. A curve shall be fit for Energy (Y-axis) versus Channel (X-axis), and the equation with the slope and Y-intercept for the fit shall be documented.
- ◇ The slope of the equation shall be <13 keV/channel.
- ◇ The energy range of each detector shall include 3 to 6 MeV.
- ◇ The energy calibration shall be performed using NIST traceable calibration sources and at least two isotopes within the energy range of 3 to 6 MeV.
- ◇ The final peak centroid positions of all observed isotopes shall be within ± 40 keV of the expected peak centroid.
- If the alpha spectrometry system was not energy calibrated to meet the above requirements, issue a Non-Compliance Notification. Do not qualify any data. Comment and assign the reason code [245] to all applicable data.

Background Control Limits

- ◇ The Background count time was documented and is as long as the sample count duration.
- ◇ The Background total counts (or counts per unit time) for each target analyte and tracer isotope ROI were analyzed on each detector and documented.
- ◇ The Background for each ROI was sufficiently low to optimize the MDA, so that the RDL can be achieved.
- ◇ The Background error and confidence level is documented.
- If the alpha spectrometry system was not background calibrated to meet the above requirements, issue a Non-Compliance Notification. Do not qualify any data. Comment and assign the reason code [246] to all applicable data.

Efficiency Control Limits

- ◇ The Efficiency determinations were performed on each detector using NIST traceable calibration sources and the isotope used was either be 239, 240Pu or 241Am.
- ◇ If the efficiency source was plutonium and the certified value of the source was based on the total alpha, the ROI used for the efficiency determination covered the range of 4 to 6 MeV to include decay daughters (in-growth of 241Am).
- ◇ If the certified value for the Efficiency calibration source was determined for the specific isotope, the ROI used for the Efficiency determination was specific for that isotope.
- ◇ The Efficiency counts for the ROI were background corrected using the same ROI for the background.
- ◇ The Efficiency is determined on at least 2000 net counts in the ROI (after background correction).

- ◇ The resolution of the Efficiency isotope was <40 keV FWHM at >2000 net counts.
 - ◇ The Efficiency error and confidence level was documented.
 - If the alpha spectrometry system was not calibrated for efficiencies, to meet the above requirements, issue a Non-Compliance Notification. Do not qualify any data. Comment and assign the reason code [177] to all applicable data.
- Item 9-e** Verify all applicable source certificates used for instrument calibrations are NIST traceable.
- If the alpha spectrometry system has been calibrated with invalid sources or sources that are not traceable to NIST or fully documented, issue a Non-Compliance Notification. Comment and qualify all applicable data as [R 164].

3.11. Counting Raw Data Summary

DRC Item 10-a through 10-f

Review Items: DRC, 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.

Requirement Sources: RC01 Exhibit B/Sections 2

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

Item 10-a Verify the counting raw data summary is present.

- If counting 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 item applies to validation only:*

Item 10-a Check that all instrument raw data for the RIN are included and are legible. 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 and detector ID
- ◇ Instrument Calibration Package ID for applicable calibration data
- ◇ "Analytical Batch ID"
- ◇ "Final Sample Aliquot" (i.e., final net weight/volume for the analysis aliquot)
- ◇ "Count Time"
- ◇ analyte isotopes
- ◇ analyte isotopes "Start" and "End" "ROI" (channels or energy)
- ◇ analyte isotopes ROI "Gross Counts" (or "Gross" counts/unit time)

- ◇ analyte isotope ROI "Net Counts" (or "Net" counts/unit time)
 - ◇ tracer isotope
 - ◇ tracer isotope "Start" and "End" "ROI" (channels or energy)
 - ◇ tracer isotope ROI "Gross Counts" (or "Gross" counts/unit time)
 - ◇ tracer isotope ROI "Net Counts" (or "Net" counts/unit time)
 - ◇ "Tracer Chemical Recovery"
 - ◇ alpha spectrometry instrument and analysis SOP(s)
 - ◇ 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 shall be documented for inclusion in a Non-Compliance Notification. Assign reason code **[803]** to all applicable data. Do not continue validation if critical items are missing. Return the SDP to ASD
- Item 10-b** Verify that the individual spectra were reviewed, signed and dated by the alpha spectrometry specialist and found to be acceptable.
- If the spectra have not been reviewed and signed, issue a Non-Compliance Notification to request for missing information, comment and assign the reason code **[804]** to all applicable data.
- Item 10-c** Verify that the tracer chemical recovery for U analyses is > 30% but < 110% and the tracer recovery for Pu and Am analyses are > 20% but < 110%.
- If the tracer recoveries do not meet the above criteria, comment and qualify all applicable data as **[R 242]**.
- Verify the FWHM resolution for each sample and QC sample tracer peak are <80 keV and the tracer peak centroid is ± 40 keV of the expected energy check
- If the sample and tracer peaks do not meet the above requirement, comment and assign the reason code **[NJ 242]** to all applicable data.
- Item 10-d** Verify there is sufficient raw data included to allow manual calculation of the final sample activity, measurement uncertainty, chemical recovery and MDA.
- 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 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-f** Verify all QC samples were counted and 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: DRC, Sample Data Package Deliverable Section Number 11.

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

Requirement Sources: GR01 Exhibit B 4, and RC01 Exhibit B/Sections 2.

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 Items 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: RC01 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 RC01CAL_LabID_Date.

- If this item is non-compliant 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-RC01 Instrument Calibration Package, (3) Narrative, and (4) Instrument Calibration and Raw Data.

- If these items are non-compliant, 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 RC01 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 Items 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: RC01 Exhibit E/Section 10, RC01 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 prior to analyzing samples.

- 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) energy calibration, (2) backgrounds, and (3) efficiencies.

- 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 energy calibrations and efficiency determinations were valid and NIST traceable and the certificates were sent to the CTR upon first use.

- If standards are not traceable to the primary certificate or are not traceable to NIST, issue a Non-Compliance Notification. Comment and qualify all applicable data as **[R 244]**.
- If standards are not valid, issue a Non-Compliance Notification. Comment and qualify all applicable data as **[R 219]**.
- If the certificates have not been sent to the CTR 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 2-d Check that all data has been reviewed, and that 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 **[803]** to all applicable data.

4.3. Instrument Calibration Package Energy Calibration

Calibration DRC Item 3-a through 3-e

Review Items: Deliverable Energy Calibration Instrument Calibration Package Section Number 3

Objective: To ensure that the alpha spectrometry detectors used for sample analysis are capable of producing quality results.

Requirement Sources: RC01 Exhibit E/Section 10, RC01 Appendix B-1

Evaluation *The following items apply to validation only:*

Item 3-a to 3-e

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

- ◇ The energy calibration for each detector was performed. A curve was fit for Energy (Y-axis) versus Channel (X-axis), and the equation with the slope and Y-intercept for the fit is documented.
- ◇ The slope of the equation is <13 keV/channel.
- ◇ The energy range of each detector is include and is 3 to 6 MeV.
- ◇ The energy calibration was performed using NIST traceable calibration sources and at least two isotopes within the energy range of 3 to 6 MeV.
- ◇ The final peak centroid positions of all observed isotopes are within ± 40 keV of the expected peak centroid.
- If the alpha spectrometry system has not been energy calibrated to meet the above requirements, issue a Non-Compliance Notification. Do not qualify any data. Comment and assign the reason code [245] to all applicable data.

4.4. Instrument Calibration Package Backgrounds

Calibration DRC Item 4-a

Review Items: Deliverable Backgrounds Instrument Calibration Package Section Number 4

Objective: To ensure that the alpha spectrometry detectors used for sample analysis are capable of producing quality results.

Requirement Sources: RC01 Exhibit E/Section 10, RC01 Appendix B-1

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

Item 4-a The background raw data are included and the required items documented as follows:

- ◇ The Background count time was documented and was as long as the sample count duration.
- ◇ The Background total counts (or counts per unit time) for each target analyte and tracer isotope ROI were analyzed on each detector and documented.
- ◇ The Background for each ROI is sufficiently low to optimize the MDA, so that the RDL may be achieved.

- ◇ The Background error and confidence level are documented.
- If the alpha spectrometry system has not been background calibrated to meet the above requirements, issue a Non-Compliance Notification. Do not qualify any data. Comment and assign the reason code [246] to all applicable data.

4.5. Instrument Calibration Package Efficiency Determinations

Calibration DRC Item 5-a through 5-e

Review Items: Deliverable Efficiency Determinations Instrument Calibration Package Section Number 4

Objective: To ensure that the alpha spectrometry detectors used for sample analysis are capable of producing quality results.

Requirement Sources: RC01 Exhibit/Section 10, RC01 Appendix B-1

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

Item 5-a to 5-e

The efficiency calibration raw data are included and the required items documented as follows:

- ◇ The Efficiency determinations were performed on each detector using NIST traceable calibration sources and the isotope used were either be 239, 240Pu or 241Am.
- ◇ If the efficiency source was plutonium and the certified value of the source was based on the total alpha, the ROI used for the efficiency determination covered the range of 4 to 6 MeV to include decay daughters (in-growth of 241Am).
- ◇ If the certified value for the Efficiency calibration source was determined for the specific isotope, the ROI used for the Efficiency determination is also specific for that isotope.
- ◇ The Efficiency counts for the ROI are background corrected using the same ROI for the background.
- ◇ The Efficiency is determined on at least 2000 net counts in the ROI (after background correction).
- ◇ The resolution of the Efficiency isotope is <40 keV FWHM at >2000 net counts.
- ◇ The Efficiency error and confidence level are documented.
- If the alpha spectrometry system has not been calibrated for efficiencies, to meet the above requirements, issue a Non-Compliance Notification. Do not qualify any data. Comment and assign the reason code [177] to all applicable data.

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 RC01 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-RC01-v1, prepared by Kip Harward 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 RC01.

Attachment 1: Data Quality Assessment Report Template

RC01

**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		
INSTRUMENT CALIBRATION PACKAGE		
Structural Requirements		
General Requirements		
Energy Calibration		
Backgrounds		
Efficiency Calibration		
Other:		

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

Effective Date:
February 13, 1998

Verification and Validation Guidelines
for PSA Module RC01

Page No.
DA-RC01-v1-28

Action Items:

N/A Item is not applicable to the Line Item

Comments:

Verification/Validation Signature _____
Reviewer Signature _____

Date: _____
Date: _____

Effective Date:
February 13, 1998

Verification and Validation Guidelines
for PSA Module RC01

Page No.
DA-RC01-v1-29

(Validation Only)