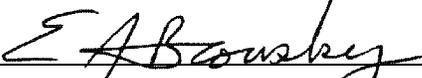


**VERIFICATION AND VALIDATION  
GUIDELINES  
FOR  
RADIONUCLIDES  
BY  
GAMMA SPECTROMETRY**

**DA-GAM-v1**

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## 1. INTRODUCTION/SCOPE

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

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

This version of DA-GAM is applicable to sample data packages for Radionuclides by Gamma Spectrometry Sample Data Packages generated under the National Basic Ordering Agreement (BOA) Statement of Work (SOW), the Rocky Flats Environmental Technology Site (Site) BOA Implementation Requirements documents, GR03 & GR04, and the SOW for "On-Site Radiological Screening by Gamma Spectrometry," RC10.

## 2. VERIFICATION AND VALIDATION INSTRUCTIONS

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

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

**Review Items:** Sample & QC Result Summary, COC record, and sample preparation raw data.

**Objective:** To ascertain the validity of results based on the method required holding times, sample preservation, and the continuity of sample custody.

**Source:** GR01 Exhibit B Section 4.8, GR03 § 6, BOA Attachment 1, § 3.1.2; Attachment J to BOA Attachment 1, § 1.2

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

**Item 1:** Check for documentation that the pH of aqueous samples were adjusted to  $\leq 2$  prior to receipt by the laboratory.

**Action 1:** If aqueous samples were not acid-preserved prior to receipt by the laboratory, comment and assign the reason code [703] to all applicable samples.

**Item 2:** Check for documentation showing aqueous samples were adjusted to a pH of  $\leq 2$  by the laboratory if samples were not adjusted to the proper pH prior to receipt by the laboratory.

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

**Item 3:** Verify the maximum hold time of 180 days was not exceeded.

**Action 3:** If samples were not analyzed within the 180 day hold time, do not qualify the data, comment and assign the reason code [101] to all applicable samples.

## 2.2. Sample Data Package Narrative Requirements

**Review Items:** Sample Case Narrative

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

**Source:** GR01 Exhibit B/Section 4.9, GR03 § 3.2, BOA Attachment 1, § 3.1.6.2

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

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

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

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

## 2.3. Samples & QC Results Summary

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

**Sources:** GR01 Exhibit B/Section 4 and RC10 Exhibit B, Attachment J to BOA Attachment 1; GR03, § 6

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

**Item 1:** Verify all samples and tests that were requested on the COC for radionuclide determinations have been analyzed, tested and appear on the Sample and QC Result Summary.

**Action 1a:** If sample were not analyzed, and documentation identifies a valid reason, do not qualify data. Address the deficiency as a comment in the Data Quality Assessment Report.

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

**Item 2** Verify the Sample and QC Results Summary contains the following information for Site Samples and laboratory QC samples as applicable:

**The following apply to data generated under the BOA and GR03/GR04**

- LABORATORY NAME
- REPORT IDENTIFICATION NUMBER (RIN)
- RFETS SAMPLE ID
- LAB SAMPLE ID
- ANALYTE
- SAMPLE MATRIX
- RESULTS and UNITS
- 2S(total propagated uncertainty)
- MDA
- ALIQUOT SIZE ANALYZED
- ANALYTICAL BATCH ID

**The following apply to data generated under RC10**

- The Site Sample Number(s)
- The Subcontractor Sample ID
- The analytical Batch ID
- Name assigned to sample spectrum file
- Detector ID
- Geometry or name of ISOCS model
- Name of library used
- The length of time the sample was counted?
- Sample weight
- Activity of each nuclide in the library, whether detected or not
- Systematic error
- Total propagated error associated with the activity of each nuclide in the library, whether it was detected or not
- The calculated MDA for each nuclide in the library, whether or not it was detected
- Analyst and reviewer's signatures

- Action 2a:** Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.
- Action 2b:** For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Item 3:** Verify only one result is reported for each requested analyte.
- Action 3:** If more than one result is reported and neither is identified as "Do Not Use data", issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received..
- Item 4:** Verify the MDA for each sample is reported and is  $\leq$  the RDL.
- Action 4a:** If the MDA is  $>$  the RDL, and a reduced aliquot size was used due to high or significant activity, or insufficient sample volume provided, do not qualify the data.
- Action 4b:** If the MDA exceeds the RDL for reasons other than those identified in Action 4a, estimate [UJ 136] all applicable data.
- Action 4c:** If the MDA is  $>$  the RDL and the deficiency is not reported in the narrative, comment and assign reason code [805] to all applicable data.
- Note:** The reviewer should use professional judgement when determining whether this criteria is applicable based on the matrix and method of analysis (ie, analysis by ISOCS detectors). Refer to the BOA Attachment J, Section 2.7 for conditions requiring reanalysis and Section 2.8 for conditions requiring a re-count.

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

- Item 5:** Verify samples requiring reanalysis have been assigned a new analytical batch identification number and appropriate QA/QC is included.
- Action 5:** If data can not be produced to show the batch ID is different and that the appropriate batch QA/QC was included in the reanalysis, reject [R 205] all applicable data.
- Item 6:** Calculate at least one sample MDA using the following equation:

$$MDA = \frac{4.65 * \sqrt{\frac{b}{T}}}{K} + \frac{3}{K * T}$$

where,

b = background count rate in cpm

---

T	=	count time in minutes
K	=	efficiency * e <sup>-λt</sup> * aliquot fraction * tracer recovery*ABN
Efficiency	=	detector efficiency
t	=	time from sample collection to mid-point of count time(or nuclide separation time, as applicable) in the same units as half-life
λ	=	Analyte decay constant = ln2/(half-life)
ABN	=	abundance

**Note 1:** Use of the above equation requires that the background and sample count times are either equivalent, or the background count time is greater than the sample count time. When sample and background counts are different, this must be included in the equation.

**Note 2:** The above equation for MDA has the units of dpm/sample. Any other units will require specification by Site/Project.

Site specific requirements may be provided for other MDA formulations.

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

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

**Action 6:** If MDAs have been calculated wrong, whether the parameters have been entered wrong or there has been a calculation error, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

## 2.4. Batch QC

**Review Item:** Sample and QC Results Summary, Batch QC Summary (QA/QC Data), and Raw Data

**Objective:** Review the QC Sample Results Summary and the Batch QC Summary for compliance to requirements and for information useful to data assessment.

**Sources:** GR01 Exhibit A & B/Section 4, RC10 Exhibit B, and Attachment J to BOA Attachment 1; GR03, § 6

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

**Item 1:** Check that the Batch QC Summary (QA/QC Data) is present and complete. The following information shall be included:

**The following apply to data generated under the BOA and GR03/GR04**

- LAB SAMPLE ID
- COUNT DATE
- QC OBSERVED VALUE with associated two sigma uncertainty
- LCS: Know value and relative Bias
- BATCH BLANK: RDL and MDA
- DUPLICATE: For each duplicate pair, the result of the duplicate equivalency test as defined in *BOA Attachment J, Section 2.3.3*

**The following apply to data generated under RC10**

Results and evaluation of the following QA/QC samples associated with the samples being reported shall be supplied as applicable

- Blank (background of suitable length using uncontaminated matrix similar to the samples)
- LSC sample for Laboratory analysis utilizing standard geometries
- Duplicate for Laboratory analysis utilizing standard geometries
- For ISOCS measurements analyses a statement that the daily source check was analyze prior to the start of the analysis and that all parameters were within acceptable limits.

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

**Item 2:** Verify a Laboratory Control Sample (LCS), Laboratory Duplicate, and Preparation Blank were included for each analytical batch.

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

- Action 2b:** If any single QC sample is missing and data cannot be obtained from the laboratory, qualify applicable data as follows:
- If missing Duplicate only, estimate [J 128] all applicable data.
  - If missing LCS only, reject [R 174] all applicable data.
  - If missing Preparation Blank only, reject [R 175] all applicable data.
- Item 3:** Verify that a set of Batch QC samples were run at least once per analytical batch. An analytical batch shall not contain more than 20 samples (excluding batch QC).
- Action 3:** If this item is non-compliant, qualify as estimated [J 168] all applicable data.
- Item 4:** Verify all QC deficiencies are detailed in the narrative.
- Action 4:** If this item is non-compliant, do not qualify any data. Comment and assign the reason code [805] to all applicable data.
- Item 5:** Verify all sample results, including reanalysis, and the corresponding Analytical Batch QC sample results were reported and that QC samples did not have count rates greater than 1000 counts per second or a dead time greater than 5% to reduce counting errors.
- Action 5a:** If this item is non-compliant, address the deficiency in the Data Assessment Report using professional judgment to qualify the data. Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.
- Action 5b:** For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Evaluation:** *The following items apply to validation only:*
- Item 6:** Verify each QC sample type is clearly identified, i.e., a designator clearly identifies a QC sample as being a LCS, Batch Blank, or Duplicate.
- Action 6:** If QC sample type cannot be clearly identified, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- The following item applies to data generated under the BOA and GR03/GR04**
- Item 7:** Verify the following additional criteria are met for data generated under the BOA and GR03/GR04:
- The QC samples are counted for a sufficient time to meet the required detection limit.

- For each batch duplicate pair, the following additional information is reported:
  - ◊ result of duplicate result equivalency test as defined in Section 2.5, including calculated values for relative Error Ratio.
- 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 Relative Bias
- For the "PB", when applicable, the following additional information is reported:
  - ◊ "MDA"(same units as the reported activity) shall also be reported for each radionuclide detected in all samples that make up the analytical batch

**Action 6a:** Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

**Action 6b:** For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

## 2.5. Duplicate Samples

**Review Item:** Sample and QC Results Summary, Batch QC Summary, and Raw Data

**Objective:** To determine a measure of laboratory precision, or degree of agreement of repeated measurements within acceptable concentration ranges and to assess the homogeneity of the samples.

**Sources:** GR01 Exhibit B/Section 4, RC10 Exhibit E, GR03, § 6, and Attachment J to BOA Attachment 1

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

**Item 1:** Verify the results for the duplicate are reported separately from the corresponding sample.

**Action 1a:** If this item is non-compliant, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

**Action 1b:** If the laboratory is unable to supply the missing data or errors cannot be corrected, do not qualify any data, comment and assign the reason code [205] to all applicable data.

**Item 2:** Verify the MDA for each duplicate is reported and is < the RDL

**Action 2a:** If these items are non-compliant, and the MDA > RDL due to laboratory error, estimate [UJ 136] all applicable data.

- Action 2b:** If the MDA for a reported duplicate is > the RDL and the following criteria are not met, comment and assign reason code [804] to all applicable data.
- The samples and duplicates were prepared with a reduced aliquot size due to high or significant activity.
  - The net counts per second for the analyte ROI are ≤100 times the background (same units, same ROI), and the tracer chemical recovery and Continuing Calibration Checks for the respective spectra are within the acceptable limits

**The following item applies to data generated under the BOA and GR03/GR04**

- Item 3:** Verify that the reported duplicate equivalency test is ≤ 3.

**Action 3a:** If a duplicate exceeds the equivalency test requirement of ≤ 3 due to the difficulty of subsampling and this explanation is described in the Case Narrative, no action is taken.

**Action 3b:** If the duplicate equivalency test does not pass and the sample is homogeneous, estimate [J 235] all applicable data in the analytical batch.

**The following item applies to data generated under RC10**

- Item 4:** Verify that the reported duplicate equivalency test meets the following criteria:

F shall be ≤ 1.5\*E or F/E ≤ 1.5

**Action 4b:** If the duplicate equivalency test does not pass and the sample is homogeneous, estimate [J 235] all applicable data in the analytical batch.

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

**The following item applies to data generated under the BOA and GR03/GR04**

- Item 5:** Calculate the normalized absolute difference between the sample and laboratory duplicate results using the following equation and confirm the value reported:

$$\frac{S - D}{\sqrt{(TPU_s)^2 + (TPU_D)^2}} \leq 3^*$$

where:

S = Sample result

D = Duplicate result

TPUS = 1s Total Propagated Uncertainty of the sample

TPUD = 1s Total Propagated Uncertainty of the duplicate

(\* 2.58 was rounded to 3)

**Action 5:** If the duplicate equivalency test was calculated wrong, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all

other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

**The following item applies to data generated under RC10**

**Item 6:** Calculate the equivalency between a duplicate and respective sample activities with the measurement uncertainties stated at the 2-sigma confidence level using the following equations:

$$F = |S - R|$$

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

where:

- F = The statistical function for testing equivalency
- S = Original sample activity
- R = Duplicate sample activity
- E = Propagated measurement uncertainty, of the difference, at 2-sigma
- ES = 2-sigma measurement uncertainty of sample activity
- ER = 2-sigma measurement uncertainty of Duplicate activity

F shall be  $\leq 1.5 * E$ , or  $F/E \leq 1.5$

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

## 2.6. Laboratory Control Sample Analysis

**Review Item:** Sample and QC Results Summary, Batch QC Summary, and Raw Data

**Objective:** To determine the overall performance of each step during sample preparation and analysis, and to evaluate the LCS relative bias as a means of assessing the accuracy of the analytical method.

**Sources:** GR01 Exhibit B/Section 4, RC10 Exhibit E, GR03, § 6, and Attachment J to BOA Attachment 1

### **ISOCS Measurements**

Laboratory Control Samples are not applicable for ISOCS measurements. The ISOCS technique utilizes an internal standard approach to ensure the ISOCS model was appropriate for the measurement.

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

**The following item applies to data generated under the BOA and GR03/GR04**

**Item 2:**      Verify, by calculation, the LCS relative bias results fall within the range of -.25 to +.25 (Reference: ANSI N13.30, Appendix B) by using the following equation:

$$\text{Relative bias} = \frac{\text{observed} - \text{known}}{\text{known}}$$

**Action 2:**      If the laboratory control sample does not pass the relative bias criteria, professional judgment should be used to determine the effect this has on the data. At a minimum, estimate [J 236] all applicable data.

**The following item applies to data generated under RC10**

**Item 3:**      Verify, by calculation, the percent recovery of the LCS, (OV)/ (CV) \* 100 is within the range of 80% to 100%.

Where

OV = Observed Value of the LCS

SV = Certified Value of the LCS

**Action 3:**      If the laboratory control sample percent recovery is not within limits, professional judgment should be used to determine the effect this has on the data. At a minimum, estimate [J 236] all applicable data.

**Item 4:**      Verify that the following requirements have been met.

- Check that the LCS analytes fall in the same approximate energy region of the spectrum as the analytes in the samples (low, mid-range, or high energy)
- The activity of the LCS shall be at least 5 times but not greater than 20 times the RDL except for RDLs of low activity, in which case the analyte shall be at a level where the random counting error does not exceed 10% in the counting time required to attain the RDL and was approved by the CTR upon first use.
- 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.
- The LCS shall be traceable to the National Institute of Standards and Technology (NIST) or shall be a working reference material as described in ASTM C 1128 and may be used repeatedly for different analytical batches as long as it is appropriate for the matrix and geometry of the batch

**Action 4:**      If the laboratory control sample does meet all criteria, estimate [J 234] all applicable data.

## 2.7. Preparation Blank

**Review Item:** Sample and QC Results Summary, Batch QC Summary, and Raw Data

**Objective:** To assess the extent of contamination introduced through sample preparation and analysis.

**Sources:** GR01 Exhibit B/Section 4, RC10 Exhibit E, GR03, § 6, and Attachment J to BOA Attachment 1

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

### Preparation Blanks

When preparation is not require, a background of suitable length must be obtained for each batch. The background should consist of an uncontaminated matrix similar to the samples whenever possible. When this is not possible, the following shall be used for the preparation Blank:

**TABLE 2-1 PREPARATION BLANK MATRICES**

Sample Type	Blank Matrix Specifications
Water	Distilled or deionized water acidified to $\text{pH} \leq 2$ , radon free
Soil	Empty counting container.
Filters	Physically and chemically identical filter media (supplied by the Site)
Misc. Solids	Empty counting container.

- Item 1:** Verify that the preparation blank meets the following requirements:
- At least one preparation blank meeting the requirements of Table 2-1, analyzed with every Analytical Batch of samples prepared at a minimum of 20 % frequency.
  - Preparation blanks were counted for at least the same count duration as the samples.

**Action 1:** 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.

- Item 2:** 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. This item does not apply to samples which have significantly greater activity than the RDL. In this case the MDA for the analysis shall be a maximum of 10% of the sample activity.

**Action 2:** If the sample results are not greater than the RDL and the preparation blank activity is not  $\leq$  the RDL, estimate [UJ 136] all applicable data.

## 2.8. Sample Preparation

**Review Items:** Preparation Raw Data

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

**Sources:** GR01 Exhibit B/Section 4, RC10 Exhibits B & E, GR03, § 6, and Attachment J to BOA Attachment 1

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

**Item 1:** Verify that benchsheets and/or preparation logs are included in the SDP.

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

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

**Item 2:** Verify that benchsheets and/or preparation logs are included and document the following as applicable:

- ANALYTICAL BATCH IDENTIFIER
- DATE OF PREPARATION
- IDENTIFIER FOR THE LABORATORY SOP for the preparation
- IDENTIFIERS FOR ALL SAMPLE AND QC SAMPLES in the batch
- CONCENTRATION OF WORKING STANDARDS used for LCS.
- VOLUMES OR WEIGHTS OF LCS ADDED (if the concentration is given in activity per unit weight then the weight added shall be reported; if the concentration is given in activity per unit volume, then the volume added shall be reported)
- BALANCE IDENTIFIERS WITH DATES OF USE (if applicable)
- INITIAL AND FINAL WEIGHTS AND VOLUMES for all samples and QC samples including gross weights, tare weights, and aliquot weights where applicable
- PIPETTE IDENTIFIERS AND DATES OF USE (if applicable)
- COMMENTS describing any significant sample changes or reactions which occur during preparation
- SIGNATURES AND DATES of all analysts and reviewers

### Soils, Sediments, Sludges and Solid Waste

- APPROXIMATE SAMPLE VOLUME RECEIVED, THE ALIQUOT SIZE HOMOGENIZED

**Action 2a:** Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

**Action 2b:** For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

**Item 3:** 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.

**Action 3:** If this item is non-compliant, do not qualify any data. Comment and assign the reason code [234] to all applicable data.

## 2.9. Standards Summary

**Review Items:** Standard Summary Raw Data

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

**Sources:** GR01 Exhibit B/Section 4, RC10 Exhibits B & E, GR03, § 6, and Attachment J to BOA Attachment 1

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

**The following item applies to data generated under the BOA and GR03/GR04**

**Item 1:** Verify that the standard summary raw data is included in the in the SDP.

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

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

**The following item applies to data generated under the BOA and GR03/GR04**

**Item 2:** For primary standards that were used for all Lab Control Samples, Check Sources, Instrument Calibration Sources and Internal Standard Sources used during the analysis, the following documentation must be reported:

- STANDARD ID. (Working Standard) that was used traced back to the PRIMARY STANDARD ID. (All identifiers must be traceable to standard reference material certificates. Submit only the first page of the NIST certificate to establish primary standard ID. and/or traceability.
- STANDARD ISOTOPES, CONCENTRATION, AND ERROR IN THE WORKING STANDARD USED

- EXPIRATION DATE of analytes in the standard
- USE for this standard (tracer, LCS, efficiency, etc.)
- DATE OF PREPARATION
- SUFFICIENT DILUTION data to provide for calculation of the activity

**Action 2a:** Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

**Action 2b:** For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

**The following item applies to data generated under RC10**

**Items 3:** Verify that all standard certificates have been forwarded to the CTR upon first use.

**Action 3:** If standard certificates have not been forwarded to the CTR, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

**The following items apply to all data**

**Items 4:** Verify that all standard identifications are traceable to the primary certificate, which are traceable to NIST.

**Action 4:** If standards are not traceable to the primary certificate or are not traceable to NIST, issue a Non-Compliance Notification and reject [R 244] all applicable data.

**Items 5:** Verify that all standards and sources traceable to NIST have not expired and are valid.

- If standards and tracers have expired, reject [R 219] all applicable data.

## 2.10. Calibration Raw Data

**Review Items:** Calibration Raw Data

**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.

**Sources:** GR01 Exhibit B/Section 4, RC10 Exhibits B & E, GR03, § 6, Attachment J to BOA Attachment 1, and ANSI N42.12 and N42.14

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

**ISOCS Type Detectors**

Instrument calibration does not apply to ISOCS type detectors in the same way that it does to standard HPGe detectors. ISOCS type detectors are supplied with a "characterization report" from the vendor and only need to be calibrated for energy and peak shape.

**Item 1:**            Verify the instrument calibration summary includes a summary of the energy calibration, background, detector resolution, and efficiency determinations with the exception of ISOCS detectors which only require an energy and peak shape calibration.

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

**Item 2:**            When ISOCS type detectors are used to analyze a sample and a model is developed to obtain the efficiency coefficients, the laboratory must maintain a file or note book that documents the selection of model parameters. The model parameters selected for each sample will be presented in the Raw Data section of the data package.

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

**Item 3**             Verify the Calibration Package identification and/or file name is included in the Calibration Raw Data.

**Action 3:**        If the Calibration Package identification and/or file name is not included in the Instrument Calibration Summary, 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 4:**            Verify the required following items are included for each gamma spectrometry detector used to report results:

**Energy Calibration (required for both standard and ISOCS type detectors regardless of the type of measurements performed)**

- Instrument and Detector ID
- Date of the Energy Calibration
- "Energy Calibration Source ID" and "Expiration Date" (**RC10 Only**)
- Energy calibration isotopes and gamma energy or energies used for calibration
- Calibration Equation
- Gamma spectrometry detector Energy Range and total number of channels that span the energy range (**RC10 Only**)

- Plot of energy Vs channel for each detector used (**RC10 Only**)
- Analyst and reviewer's signatures and dates. (**RC10 Only**)

**Action 4a:** Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of errors or omissions.

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

**Item 5:** Verify the required following items are included for each gamma spectrometry detector used to report results:

**Backgrounds**

- Instrument and Detector ID
- File Name (**RC10 Only**)
- Date of the Background Analysis
- "Count Time"
- Respective ROI "Background Counts" or "Background" counts/unit time when the determination of a specific radionuclide is requested
- Respective Start and End ROI (in channels or energy) for specific isotope analyses (**RC10 Only**)
- Background Error and confidence level (**RC10 Only**)
- Analyst and reviewer's signature and date (**RC10 Only**)

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

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

**Item 6:** Verify the required following items are included for each gamma spectrometry detector used to report results:

**Efficiency Calibrations (required for standard detectors only)**

(ICOCs efficiencies are dependent on the ISOCS model parameters and are reported with individual sample results)

- Instrument and Detector ID
- File name (**RC10 Only**)
- Date of the Efficiency Analysis
- Efficiency Isotope
- Efficiency Equation
- "Efficiency Source ID", "Certified Value" with date of certification, and "Expiration Date" (**RC10 Only**)

- "Count Time" (**RC10 Only**)
- Plot of efficiency vs. energy for each detector used (**RC10 Only**)
- Analyst and reviewer's signature and date (**RC10 Only**)

- Action 6a:** Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.
- Action 6b:** For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Item 7:** Verify all gamma detectors were calibrated over the energy range of interest and for each detector a daily source check was performed at the beginning and end of the shift each day samples were counted.
- Action 7:** If daily source checks were not run at the beginning and end of each shift that samples were counted issue a NCN. Do not qualify any data. Comment and assign the reason code [129] to all applicable data.
- Item 8:** Verify the energy calibrations are within control limits according to the following:
- The energy calibration for each detector was performed. A linear curve was fit for Energy (Y-axis) versus Channel (X-axis) of the curve, and the constants for the equation are documented. The goodness of fit ( $r^2$ ) has been provided (**RC10 only**).
  - The slope of the equation is approximately equal to 0.25 keV/channel.
  - The energy range of each detector is 59 to 1836 keV (**RC10 only**).
  - The energy calibration was performed using NIST traceable calibration sources and at least two isotopes are within the energy range of 59 to 1836keV. Americium 241 was included in the energy calibration source (**RC10 only**).
- Action 8:** If the gamma spectrometry system was not energy calibrated to meet the above requirements, issue a NCN. Do not qualify any data. Comment and assign the reason code [245] to all applicable data.
- Item 9:** Verify the background calibrations are with-in control limits according to the following:
- Background Control Limits are performed as part of the initial calibration and are verified at least quarterly following the initial calibration, or after any counting chamber changes have been made
  - The background for each detector did not contain any target analytes above the RDL.
  - The background was collected at the beginning of each analytical batch.
  - The background count time was documented and is at least as long as the sample count duration.
  - The background spectrum from each detector was saved and processed using the latest efficiency calibration.

- Action 9:** If the gamma 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.
- Item 10:** Verify the efficiency calibrations are with-in control limits according to the following:
- The Efficiency determinations were performed on each detector using matrix and geometry specific NIST traceable calibration source(s), if applicable.
  - Americium-241 was included in the efficiency calibration (**RC10 Only**).
  - The certified value for each isotope in the efficiency standard was determined at a specific energy, therefore, the efficiency determination must also use that specific energy (**RC10 Only**).
  - The laboratory has documented the reason why any of the peaks present in the original efficiency calibration source are not used to determine the efficiency curves above or below the knee (**RC10 Only**).
  - The efficiency error and confidence level are documented (**RC10 Only**).
  - When ISOCS type detectors are used and a model is developed to obtain the efficiency coefficients, the laboratory must maintain a file or notebook that documents the selection of the model parameters. The model shall be accepted by the CTR prior to use (**RC10 Only**).
  - The model selected for use in an ISOCS analysis shall be presented in the Case Narrative and shall include a discussion of actual and predicted peak ratios for isotopes with multiple gamma energies present in the samples (**RC10**).
- Action 10:** If the gamma system was not calibrated for efficiencies, to meet the above requirements, issue a NCN. Do not qualify any data. Comment and assign the reason code [177] to all applicable data.

## 2.11. Daily Check Source & Background Counts

- Review Items:** Calibration Raw Data
- Objective:** Verify that the daily source checks deliverable parameters are within control limits to establish and document instrument stability
- Sources:** GR03, § 6, Attachment J to BOA Attachment 1, RC10, Exhibit B
- Evaluation:** *The following items apply to both verification and validation:*
- Item 1:** Verify the Daily Check Source & Background Count data are present and includes a counting and evaluation of a daily source check at the beginning of each day that samples are analyzed and at the end of the day, or not longer than after 16 hours of continuous analysis, to demonstrate that the detector was performing satisfactorily during the entire sample analysis time.
- Action 1:** If the daily check source and background data are not included, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any

deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

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

**Item 2:** Verify that the required daily source check information is provided for all gamma detectors used to analyze the Site samples for the RIN and the associated Analytical Batch QC and includes the following:

- Instrument Used
- File Name of data collected
- Date of calibration checks
- Output of daily energy calibration check with acceptance criteria or complete calibration if recalibration was necessary.
- Output of daily efficiency check source with control charts showing 2 & 3 sigma limits.
- FWHM values of efficiency peaks & associated control chart

**Action 2a:** Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code [804]. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

**Action 2b:** For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

**Item 3:** Determine if check source is within limits

**Action 3a:** Use professional judgement to evaluate data associated with check source results that fall between 2 & 3 sigma control limits.

**Action 3b:** If check source exceeds the 3 sigma control limit, issue a NCN and reject [R 141] all applicable data.

## 2.12. Sample Analysis Raw Data

**Review Items:** Sample Analysis Raw Data

**Objective:** To verify 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.

**Sources:** GR01 Exhibit B/Section 4, RC10 Exhibits B, GR03, § 6, Attachment J to BOA Attachment 1, and ANSI N42.12 and N42.14

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

**Item 1:** Verify Sample Analysis raw data is present.

**Action 1:** If sample analysis raw data is missing, issue a NCN, comment and assign reason code [801] to all applicable data. Inspect all other SDP deliverables

for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

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

**Item 2:** Check that all instrument raw data for the RIN are included and are legible. Check that sample analysis raw data are included for all analyses performed and include the following:

- SAMPLE ID (Site / Laboratory)
- DATE AND TIME of analysis
- COUNT TIME
- DATA FILE NAME
- INSTRUMENT AND DETECTOR ID
- FILE NAME OF BACKGROUND USED
- APPROPRIATE DETECTOR BACKGROUND
- DETECTOR EFFICIENCY FOR THIS SAMPLE
- ANALYTICAL BATCH ID
- SAMPLE ALIQUOT SIZE
- ANALYTE ISOTOPE(S)
- START AND END CHANNELS FOR ALL APPLICABLE ROIS
- ANALYTE(S) GROSS COUNTS
- BACKGROUND COUNTS (IDENTIFY COUNT TIME OF BACKGROUND)
- ANALYTE(S) NET COUNTS
- FWHM AND PEAK ENERGY where applicable
- INSTRUMENT RUN LOG for applicable count dates(copy is acceptable)
- LIBRARY IDENTIFICATION

**Action 2a:** Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804]**. An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

**Action 2b:** For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

**Item 3:** Verify that the individual spectra were reviewed, signed and dated by the gamma spectrometry specialist and found to be acceptable.

**Action 3:** If the spectra have not been reviewed and signed, issue a NCN to request for missing information, comment and assign the reason code **[804]** to all applicable data.

**Item 4:** Verify the peak resolution (FWHM) and energy calibration check (peak centroid position) were found to be acceptable according to BOA Attachment J, Part 6, Section 1.1 (ANSI N42.12 and N42.14) criteria for all samples and QC samples:

- Action 4a:** If the energy calibration check (peak centroid position or FWHM) does not meet the above requirements, comment and assign the reason code [245] to all applicable data.
- Item 5:** Verify there is sufficient raw data included to allow manual calculation of the final sample activity, measurement uncertainty, and MDA.
- Action 5:** If this item is non-compliant, issue a NCN, comment and assign reason code [803] to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.
- Item 6:** 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.
- Action 6:** If these items are non-compliant, do not qualify any data. Comment and assign the reason code [234] to all applicable data.

### **3. DATA QUALITY ASSESSMENT REPORT PREPARATION**

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

### **4. 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 Laboratory Requirements, GR01 Version B.3
- On-Site Radiological measurements by Gamma Spectrometry, RC10, Version B
- RFETS BOA Implementation Requirements, GR03 Version A.5
- RFETS BOA Implementation Requirements, GR04 Version A
- Basic Ordering Agreement (BOA) for Laboratory Analytical Services administered by Westinghouse Savannah River Company on behalf of the Department of Energy.

### **5. REVISION HISTORY**

DA-GAM , prepared by Jana Dawson and J. P. Garrett of TechLaw Inc. is the first issue of the Verification and Validation Guidelines for Radionuclides by Gamma Spectrometry.

## ATTACHMENT 1: DATA QUALITY ASSESSMENT REPORT TEMPLATE

### GAM

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

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

Sample Numbers: \_\_\_\_\_

Quality Control Items	Reviewed (Y or N)	Non-Compliance Identified
General (Cover Page, General SDP, Narrative)		
Chain of Custody, Preservation, and Holdings		
Sample Results		
QC Sample Results		
Duplicate Sample Results		
Laboratory Control Results		
Preparation Blank Results		
Preparation Raw Data		
Standards Summary Raw Data		
Calibration Raw Data		
Sample Analysis Raw Data		
Electronic Data Deliverable EDD		
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  
 N/A    Item is not applicable to the Line Item

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

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

**Action Items:**

**Comments:**

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

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

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

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

(Validation Only)