



VERIFICATION AND VALIDATION GUIDELINES

FOR

RADIOMETRIC STRONTIUM

BY GPC

DA-RC05-v1

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1. INTRODUCTION/SCOPE

This procedure presents those data assessment steps which are unique to PSA Module RC05, Radiometric strontium by Gas Flow Proportional Counting (GPC). 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-RC05, until replaced by a more recent version, is applicable to all versions of the PSA Module RC05.

This procedure for the data quality assessment of RC05 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 RC05 for Radiometric Strontium by Gas Flow Proportional Counting (GPC). 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-5
	4-e	5-d	6-b-3
		5-e	

2.1.1. For all other items with *NA* marked in the *Reply* column, enter **X** in the **Ö** 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 RC05 for radiometric strontium 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 RC05. 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 1D, COC record, sample preparation/extraction log.

Requirement Source: GR01 Exhibit B Section 4.8 and RC05 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 There are no technical requirements for holding times for radiometric strontium samples. Therefore this item is not evaluated at either the verification or validation level.

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, comment 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 Verify that samples requiring reanalysis are identified with a reason for reanalysis and 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 RC05, 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 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 for each sample and QC sample the results include the following:
- ◇ sample ID
 - ◇ analytes
 - ◇ analyte activities (see RC05 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 a comment and reason code [804].
 - Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [803] to all applicable data.
- Item 6-a-3** Verify that only one result is reported for each requested analyte.
- If more than one result is reported and neither is identified as "Do Not Use data", contact the CTR for instructions.

Item 6-a-5 Verify that the MDA for each sample is reported and is \leq the RDL.

If the MDA is $>$ the RDL, 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 measurement uncertainty is not greater than 15% of the sample activity
- ◇ The MDA for the analysis is a maximum of 20% of the sample activity
- If 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 qualify and assign the reason code [UJ 136] to all applicable data.

The following equation shall be used to calculate the MDA. Except as specified above, the analyte MDAs shall be less than or equal to the respective RDL. Calculate at least one sample MDA using the following equation:

$$MDA = \frac{\frac{2.71}{T_S} + 3.29 \sqrt{\frac{BKG}{T_S} + \frac{BKG}{T_B}}}{2.22 \times EFF \times AMT \times Y \times e^{-\lambda t}}$$

where,

- BKG = Background count rate (cpm)
- T_B = Background count time (minutes)
- T_S = Sample count duration (minutes)
- EFF = Detector efficiency
- AMT = Analysis Aliquot (liters or grams)
- e^{-λt} = Decay correction (for specific radionuclide)
- Y = Chemical recovery obtained from gravimetric stable carrier.

- If MDA has been calculated wrong, whether the parameters have been entered wrong or there has been a calculation error, 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 Sections 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 RC05, Exhibit B/Section 2, Exhibit E/Section 6, 7, and 8

Evaluation: *The following items apply to both 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.

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

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

Item 6-b-3 Verify that all QC deficiencies are detailed in the narrative.

- If this item is non-compliant, do not qualify any data. Comment and assign the reason code [805] to all applicable data.

Item 6-b-4 Verify that all sample results, including reanalysis, and the corresponding Analytical Batch QC sample results are acceptable and were reported.

- If this item is non-compliant, 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 and 6-c-2

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 RC05 Exhibit E/Section 7

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. Comment and assign the reason code [205] 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 RC05 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, comment and qualify the results as [J 235] to all applicable data.

3.6. Laboratory Control Sample Analysis

DRC Items 6-d-1 through 6-d-3

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 RC05 Exhibit E/Section 8

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

Item 6-d-1 Verify that the LCS certified activity and uncertainty at the 2-sigma, decayed to analysis date, are reported.

- If the laboratory control was not reported and/or decay corrected to the analysis date, comment, qualify and assign the reason code [R 132] to all applicable data.

Verify by calculation that percent recoveries (OV/CV (certified value)*100) are within the control limits (75%-125%).

- If the laboratory control sample does not pass the percent recovery, comment, 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 is not within plus or minus three standard deviations of the standard value, comment, qualify and assign the reason code [R 236] to all applicable data.

Verify that the LCS meets the following criteria:

- ◇ The isotopes for the LCS shall be $^{90}\text{Sr}/^{90}\text{Y}$.
 - ◇ An aqueous LCS was used for all matrices.
 - ◇ The units for reporting the LCS are up to the discretion of the laboratory as long as the units are specified, and are the same for both the observed and certified values.
 - ◇ The LCS shall be counted for the same count duration as the samples
 - ◇ The LCS was prepared and analyzed in the same manner as the samples.
- If the laboratory control sample does not pass all of these criteria, qualify the results and assign the reason code [J 234] to all applicable data.

Item 6-d-2 Verify the activity level in the LCS is at an appropriate level for the samples in the Analytical Batch.

The activity in the analysis aliquot should be sufficiently high to produce statistically sound data. However, the activity level should not be so high as to create a potential for sample or laboratory cross-contamination.

- ◇ The LCS for low level environmental water samples, low level soil samples, and low level waste samples shall be less than 5 pCi /aliquot.
 - ◇ For higher level samples, is the activity in the analysis aliquot (not the concentration of the activity in the sample) used to determine the appropriate LCS level
- If the laboratory control sample does not pass all of these criteria, comment, qualify the results and assign the reason code [J 234] to all applicable data.

3.7. Preparation Blank

DRC Item 6-e-1

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

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

Requirement Sources: RC05 Exhibit E/Section 6

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 least a 10 % frequency.
- ◇ An aqueous Preparation Blank was used for all aqueous and non-aqueous matrices.
- If these items are non-compliant, do not qualify any data. Comment and assign the reason code [234]

The Preparation Blank Data shall be evaluated according to following criteria:

- ◇ If the MDAs for the samples in an Analytical Batch meet the analyte RDLs and the sample activities are less than 5 times the RDL (RC05, Exhibit C), the activity of the preparation blank shall be equivalent to zero when the measurement uncertainty is considered and shall be less than or equal to the RDL.
- If the preparation blank is not equivalent to zero when the measurement uncertainty is considered and is not less than or equal to the RDL, then this item is non-compliant. Qualify the data and assign the reason code [J 237]
 - ◇ If the MDAs for the samples in an Analytical Batch meet the analyte RDLs and the sample activities are greater than or equal to 5 times the RDL (RC05, Exhibit C), the activity of the preparation blank shall be equivalent to zero when the measurement uncertainty is considered.
- If the preparation blank is not equivalent to zero when the measurement uncertainty is considered, then this item is non-compliant. Qualify the data and assign the reason code [J 237].
 - ◇ If the MDAs for the samples in an Analytical Batch do not meet the RDL due to high analyte activity and reduced sample size (as per Section 5 of this Exhibit), the Preparation Blank analyte activity shall be less than 1% of the sample analyte activity.
- If the Preparation Blank analyte activity is not less than 1% of the sample analyte activity, then this item is non-compliant. Qualify the data and assign the reason code [J 237]

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

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

- ◇ Preparation blanks were counted for at least the same count duration as the samples unless the samples had to be counted longer than the routine count time in order to meet the RDL.
- ◇ The Preparation Blank MDA calculation is based on the greatest sample volume or weight for the entire Analytical Batch.
- If these items are non-compliant, do not qualify any data. Comment and assign the reason code [234]

3.8. Sample Preparation

DRC Items 7-a through 7-g

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: RC05 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 as described in the Standards Summary Section)
- ◇ sample and QC sample aliquots
- ◇ sample dilution, digestion, or dissolution volumes, if applicable
- ◇ "Final Sample Aliquot" - calculated net aliquot on planchet, if applicable (required only if dilution, digestion, or dissolution was required for sample preparation)
- ◇ "Tare Weight" and "Gross Weight", for each planchet
- ◇ "Net weight" of Sr and/or Y precipitate on counting planchet
- ◇ "Pipette ID" and dates of use, if applicable
- ◇ amount and concentration of stable Sr, and Y carriers added, if applicable
- ◇ measured amount of stable Sr in samples before carrier is added
- ◇ analysis technique that was employed to determine the amount of stable Sr present in the sample before the carrier is added, if applicable
- ◇ date and time for the start of the Yttrium ingrowth
- ◇ date and time for the end of the Yttrium ingrowth
- ◇ "Balance ID" and dates of use
- ◇ methodology SOP, including revision number or date
- ◇ 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.

Item 7-b Verify that if stable strontium carrier was used and the stable strontium content of the sample was unknown, that the stable strontium content of the sample was determined by AA or ICP.

- If the stable strontium content of the sample was not known and was not determined then this item is non-compliant, qualify and assign the reason code [R 207] to all applicable data.

Item 7-c Verify that the weight of the preparation blank was subtracted from the residue weight of the sample to get the net residue weight.

- If the weight of the blank was not subtracted from the residue weight of the sample then this item is non-compliant, qualify and assign the reason code [R 207] to all applicable data.

Item 7-d Verify that the sample was counted within three hours after the sample was stripped from the column.

- If the sample is not counted within the three hour time limit an error will be introduced from Y90 and this item will be non-compliant, qualify, comment and assign the reason code [NJ 207] to all applicable data.

Item 7-e Verify that the Yttrium ingrowth time allowed was at least 7 days if applicable.

- If the ingrowth time for yttrium was not at least seven this item is non-compliant, qualify, comment and assign the reason code [NJ 207] to all applicable data.

Item 7-f Verify that all start and stop times for all ingrowths were recorded and reported.

- If the start and stop times for all ingrowth times were not reported then this item is non-compliant, issue a Non-Compliance Notification to request the

missing data. Do not qualify any data and assign the reason code [803] to all applicable data.

- If the start and stop times for all ingrowth times were not recorded then this item is non-compliant, qualify, comment and assign the reason code [R 207] to all applicable data.

Item 7-g 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.

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: RC05 Exhibit B/Section 4; 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 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" 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 LCS or prepared calibration source)

- ◇ "Aliquot Size" and units (net weight/volume of diluted standard used for the LCS or prepared calibration source)
- ◇ "Aliquot Activity" and units (activity and units for each aliquot of diluted standard used for the 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 LCS and any in-house prepared calibration sources, the following documentation must be reported:

- ◇ standard isotope
- ◇ standard type ("LCS 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 LCS or prepared calibration source)
- ◇ "Aliquot Size" and units (net weight/volume for each aliquot of primary standard used for the LCS or prepared calibration source)
- ◇ "Aliquot Activity" and units (activity and units for each aliquot of primary standard used for the 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 above items which do not affect your ability to review the data shall be documented with a comment and reason code **[804]**.
- Other omissions or errors shall be documented for inclusion in a Non-Compliance Notification. Inspect all other SDP deliverables for any other

missing or revised information needed for data assessment and return the SDP to ASD with Non-Compliance Notification. Assign reason code [803] to all applicable data.

- Items 8-a** Primary standard and secondary standard calculation should be performed to verify the values.
- If these calculations do not coincide with the standard values, then this item is non-compliant, qualify, comment and assign the reason code [R 243] to all applicable data.
- 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, comment, qualify and assign the reason code [R 244] to all applicable data.
- Items 8-e** Verify that all standards and sources traceable to NIST have not expired and are valid.
- Standards and tracers that have expired are not valid under any circumstances. Comment, qualify and assign the reason code [R 219] to all applicable data.

3.10. Instrument Calibration Summary

DRC Items 9-a through 9d

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: RC05 Exhibit B/Section 4, Exhibit E/Section 10

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

- Item 9-a** Verify the instrument calibration summary is included.
- If the instrument calibration summary is missing, do not qualify any data, comment and assign reason code [801] to all data.
- Item 9-b** 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-c** 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 specified items are included and have been met for each gas proportional counter used to report results:
- ◇ instrument and detector ID(s)
 - ◇ date(s) of efficiency calibration
 - ◇ “Standard IDs” of sources used for efficiency calibrations (alpha and beta)
 - ◇ isotopes for efficiency calibration standards
 - ◇ “Efficiency” and “Efficiency Uncertainty” (2 sigma) for alpha and beta counting efficiencies
 - ◇ date(s) of self-absorption/crosstalk curve calibration
 - ◇ “Standard IDs” of sources used for self-absorption/crosstalk curve calibration
 - ◇ isotopes for self-absorption/crosstalk curve calibration planchets
 - ◇ curve coefficients for alpha and beta self-absorption curves
 - ◇ curve coefficients for crosstalk curves, or crosstalk factors, as applicable
- If these items are non-compliant, issue a Non-Compliance Notification to request the missing information. Do not qualify any data and assign the reason code [803] to all applicable data.
- Items 9-d** Verify that all applicable source certificates used for the instrument calibration are traceable to NIST and were 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 [803] to all applicable data.

3.11. Continuing Calibration Summary

DRC Items 10-a through 10-c

Review Items: DRC, Sample Data Package Deliverable Section Number 10.

Objective: Verify that the continuing calibration summary deliverable parameters are within control limits to establish and document instrument stability

Requirement Sources: RC05 Exhibit B/Sections 2

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

Item 10-a Verify the continuing calibration summary is present.

- If the continuing calibration 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 Verify that the required continuing calibration information is provided for all GPC detectors used to analyze the Site samples for the RIN and the associated Analytical Batch QC.

- ◇ instrument and detector ID(s)
- ◇ date of the background analysis
- ◇ data filename, if applicable
- ◇ individual alpha and beta background counts or count rates
- ◇ “Count Time” - background count time
- ◇ “Alpha Background” and “Beta Background” - average alpha and beta count rates
- ◇ “Alpha Background Error” and “Beta Background Error” and confidence limits for each
- ◇ signatures and dates of all analysts and reviewers
- ◇ analyst and/or reviewers comments regarding resolution of abnormal background counts
- ◇ methodology SOP(s) with revision number or date
- 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 10-b Verify the following information is reported for the detector background determinations:

- ◇ instrument and detector ID(s)
- ◇ date of the background analysis
- ◇ data filename, if applicable
- ◇ individual alpha and beta background counts or count rates
- ◇ “Count Time” - background count time
- ◇ “Alpha Background” and “Beta Background” - average alpha and beta count rates
- ◇ “Alpha Background Error” and “Beta Background Error” and confidence limits for each
- ◇ signatures and dates of all analysts and reviewers

- ◇ analyst and/or reviewers comments regarding resolution of abnormal background counts
- ◇ methodology SOP(s) with revision number or date
- 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 10-c Verify the following information is reported for the detector check source determinations:

- ◇ instrument and detector ID
- ◇ date of check source analysis
- ◇ data filename, if applicable
- ◇ check source ID(s)
- ◇ alpha and beta counts, count rates, or calculated efficiencies, as appropriate. If the check source test requires that more than one count is collected, but only the averages of the data are evaluated, only the averages are required to be reported.
- ◇ control limits
- ◇ result of check source data assessment (pass, fail, etc.)
- ◇ signatures and dates of all analysts and reviewers
- ◇ analyst and/or reviewers comments regarding resolution of “failed” check source data
- ◇ methodology SOP(s) with revision number or date
- 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.

3.12. Counting Raw Data Summary

DRC Items 11-a through 11-c

Review Items: DRC, Sample Data Package Deliverable Section Number 11.

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: RC05 Exhibit B/Sections 2

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

Item 11-a Verify the instrument raw data for the RIN is present and includes benchsheets and/or preparation logs and a copy of the instrument run log.

- If the counting raw data summary is missing or is missing one or more of the items above, 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 11-a Verify that all instrument raw data for the RIN are included and are legible. Verify that preparation raw data (benchsheets and/or preparation logs) are included for all analyses performed and include the following:

- ◇ sample ID
- ◇ date of analysis
- ◇ data filename, if applicable
- ◇ instrument and detector ID(s)
- ◇ "Analytical Batch ID"
- ◇ "Count Time"
- ◇ analyte
- ◇ analyte "Gross Counts" (or "Gross Count Rate")
- ◇ methodology SOP(s) with revision number or date
- ◇ 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 11-b Verify that if the planchets were counted more than once the following is reported:

- ◇ If the planchets were counted more than once, the required information is provided for each count.
- ◇ If more than one count was used to calculate the reported results, the worksheet, spreadsheet, etc., used to calculate average activities, MDAs, and propagated uncertainties is included.
- ◇ If not all of the counts taken were used, the data which were not used is clearly labeled as "Data Not Used".
- 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 11-c** Verify that a copy of the instrument run log is included in this Deliverable Section. It should contain the following information, at a minimum:
- ◇ date of analysis
 - ◇ data filename, if applicable
 - ◇ Site sample ID or respective laboratory ID
 - ◇ instrument and detector ID
 - ◇ 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

3.13. Electronic Data Deliverable (EDD)

DRC Items 12-a through 12-c

Review Items: DRC, Sample Data Package Deliverable Section Number 12.

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

Requirement Sources: GR01 Exhibit B 4, and RC05 Exhibit B/Sections 2.

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

Item 12-a through 12-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: RC05 Exhibit E/Section 11, Exhibit B/Section 4, RC05 Appendix B-1

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

Item 1-a Verify that the instrument calibration package was assigned an identification using the syntax of RC05CAL_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** Verify that the instrument calibration package contains the following sections in the following order. (1) Cover Page, (2) Data Review checklist-RC05 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** Verify that the structural requirements specified in RC05 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 and assign the reason code [804] to all applicable data.

4.2. General Requirements

Calibration DRC Items 2-a through 2-i

Review Items: Deliverable Instrument Calibration Package Section Number 2

Objective: Ensure that the instrument calibration data package and all general requirements are provided, and for all reported data that the data is consistent with the results reported.

Requirement Sources: RC05 Exhibit E/Section 11, Exhibit B/Section 4, RC05 Appendix B-1

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

- Item 2-a** Verify that all the required items in the Instrument Calibration Package section are legible, clearly identified, and appear exactly as given, or the identification is cross referenced on the cover sheet.
- If this item is non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data and assign the reason code [803] to all applicable data.
- Item 2-b** If there was an item identification discrepancy, verify the specific item is listed under a header titled “Specific Item”, and the laboratories cross referenced identification appears under an adjacent header titled “Identifier Used”.
- If this item is non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data and assign the reason code [803] to all applicable data.
- Item 2-c** Verify that all sources used for calibration and efficiency determinations are valid and NIST traceable.
- If sources are not valid or have expired, all data compiled using these standards should be rejected. Comment, qualify and assign the reason code [R 219].

- If sources are not traceable to NIST all data compiled using these standards should be rejected. Comment, qualify and assign the reason code [R 244] to all applicable data.
- Item 2-d** Verify that the geometry of the efficiency and self absorption curve planchets are the same as the planchets used in the sample analysis.
- If the geometry of the efficiency and self absorption curve planchets are not the same as the planchets used in the sample analysis professional judgment should be used to evaluate how this discrepancy affects the data. Comment, qualify and assign the reason code [R 228] to all data if applicable.
- Item 2-e** Verify that the matrices of the calibration sources used for efficiency and self-adsorption/crosstalk curves were the same as that of the prepared sample and QC sample planchets.
- If the matrices were not the same, all data compiled using these standards should be rejected. Comment, qualify and assign the reason code [R 228] to all applicable data.
- Item 2-f** Verify that commercially prepared sources have not been used past their expiration dates which may be based on the radionuclide half-life or physical form of the standard.
- If sources are expired all data compiled using these standards should be rejected. Comment, qualify and assign the reason code [R 219] to all applicable data.
- Item 2-g** Verify that if alpha calibration requirements were disregarded, all alpha emitters were removed from the strontium and yttrium fractions.
- If alpha calibration requirements were disregarded and all alpha emitters were not removed from the strontium and yttrium fractions, all data compiled should be rejected. Comment, qualify and assign the reason code [R 207] to all applicable data.
- Item 2-h** Verify that all data has been reviewed and certified as accurate and includes the signatures of all analysts and reviewers.
- If this item is non-compliant do not qualify any data. Comment and assign the reason code [804] to all applicable data.
- Item 2-i** Verify that standardization of strontium and yttrium carriers has been performed.
- If the carriers have not been standardized professional judgment should be used to evaluate the data, checking all QA/QC and at a minimum comment, qualify and assign the reason code [NJ 166] to all applicable data.

4.3. Voltage Plateau

Calibration DRC Items 3-a through 3-d

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

Objective: To ensure that the gas proportional counters used for sample
analysis are capable of producing quality results.

Requirement Sources: RC05 Exhibit E/Section 11, Exhibit B/Section 4, RC05
Appendix B-1

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

Item 3-a Verify that all required data and information specified below are included in
the Instrument calibration Package:

- ◇ date(s) of plateau determinations
 - ◇ data filename(s), if applicable
 - ◇ instrument and detector ID(s)
 - ◇ “Standard ID” of source(s) used for plateau determination
 - ◇ counts obtained for each voltage setting
 - ◇ plot of voltage plateau curve
 - ◇ length and slope of the plateau
 - ◇ selected operating voltage
 - ◇ analyst’s and reviewer’s signature and date
 - ◇ methodology SOP with revision number or date
 - ◇ copy of instrument run log
- If these items are non-compliant, issue a Non-Compliance Notification to request the missing information. Do not qualify any data and assign the reason code [803] to all applicable data.

Item 3-b Verify that the voltage plateaus met the following criteria for length and
number of counts at each voltage setting on the plateau as specified:

- ◇ The voltage range used to prepare the counting plateau was wide enough to fully define the “knee” of the plateau and the end of the plateau.
 - ◇ The source was counted long enough to obtain 10,000 counts or more for each point on the plateau portion of the curve.
- If these items are non-compliant, professional judgment should be used to evaluate how this discrepancy affects the data based on the instrument calibrations not meeting the Statement of Work, comment, qualify and assign the reason code [R 228] to all data if applicable.

Item 3-c Verify that the length and slope of the plateau met the manufacturers
specifications.

- If the length and slope of the plateau did not meet the manufacturers specifications do not qualify any data. Comment and assign the reason code [228] to all applicable data.

Item 3-d Verify that the if the operating voltage setting is common to more than one detector in the system, that the optimum operating voltage for all common detectors was selected.

- If this item is non compliant do not qualify any data. Comment and assign the reason code [228] to all applicable data.

4.4. Discriminator Window Settings

Calibration DRC Items 4-a and 4-b

Review Items: Deliverable Discriminator Window Setting Section Number 4

Objective: To ensure that the discriminator window settings have been set correctly in order to achieve the proper alpha and beta crosstalk.

Requirement Sources: RC05 Exhibit E/Section 11, Exhibit B/Section 4, RC05 Appendix B-1

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

Item 4-a Verify that all required data and information is included according to the following:

- ◇ date(s) of discriminator window determination
 - ◇ data filename(s), if applicable
 - ◇ instrument and detector ID(s)
 - ◇ “Standard ID” of source(s) used for setting discriminator windows
 - ◇ beta upper level discriminator and alpha lower level discriminator settings
 - ◇ calculated beta to alpha crosstalk and alpha to beta crosstalk (percent or fraction)for selected discriminator settings
 - ◇ analyst’s and reviewer’s signature and date
 - ◇ methodology SOP with revision number or date
 - ◇ copy of instrument run log
- If these items are non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data and assign the reason code [803] to all applicable data.

Verify that the discriminator windows were set for each individual detector.

- If the discriminator windows were not set on individual detectors used to count samples or QC samples, professional judgment should be used to evaluate how this discrepancy affects the data. It may be necessary that these samples must be rejected. Comment, qualify and assign the reason code [R 228] to any sample data that was counted on these detectors if applicable.

Item 4-b Verify that the discriminator windows were set to achieve <1% beta to alpha crosstalk.

- If this item is non-compliant, professional judgment should be used to evaluate how this discrepancy affects the data based on the instrument calibrations not meeting the Statement of Work, comment, qualify and assign the reason code [R 228] to all data if applicable.

4.5. Efficiency Calibrations

Calibration DRC Items 5-a and 5-b

Review Items: Deliverable Efficiency Calibration Package Section Number 5

Objective: To ensure that the gas proportional counters used for sample analysis are capable of producing quality results.

Requirement Sources: RC05 Exhibit E/Section 11, Exhibit B/Section 4, RC05 Appendix B-1

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

Item 5-a Verify that separate counting efficiencies and errors were determined for Sr-89 and Sr-90 using known amounts of the respective radioactive standards and strontium carrier.

- If the separate counting efficiencies and errors were not determined for Sr-89 and Sr-90 using known amounts of the respective radioactive standards and strontium carrier comment, qualify and assign the reason code [R 228] to all data.

Verify the efficiency calibration raw data are included and document the required items as follows:

- ◇ date(s) of efficiency calibration
- ◇ data filename(s), if applicable
- ◇ instrument and detector ID(s)
- ◇ "Standard ID" of sources used for efficiency calibration
- ◇ isotopes on the efficiency calibration standards
- ◇ "SV", "SV Uncertainty", and date of certified activity for efficiency calibration sources
- ◇ count time
- ◇ alpha and beta counts or count rate for each count collected
- ◇ calculated "Efficiency", "Efficiency Error", and confidence level
- ◇ analyst's and reviewer's signature and date
- ◇ methodology SOP with revision number or date
- ◇ copy of instrument run log
- If these items are non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data and assign the reason code [803] to all applicable data.

- Item 5-b** Verify that each alpha and beta calibration standard was counted to a minimum accumulation of 10,000 counts.
- If this item is non compliant do not qualify any data. Comment and assign the reason code [172] to all applicable data.
- Verify that the error and confidence level for each efficiency is included.
- If these items are non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data and assign the reason code [804] to all applicable data.

4.6. Self-Absorption/Crosstalk Curve Calibrations

Calibration DRC Items 6-a through 6-f

Review Items: Deliverable Self-Absorption/Crosstalk Curve Calibrations Package Section Number 6

Objective: To ensure that the gas proportional counters used for sample analysis are capable of producing quality results.

Requirement Sources: RC05 Exhibit E/Section 11, Exhibit B/Section 4, RC05 Appendix B-1

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

- Item 6-a** Verify that the self-absorption/crosstalk curve raw data are included and include the following information:
- ◇ date(s) of calibration
 - ◇ data filename(s), if applicable
 - ◇ instrument and detector ID(s)
 - ◇ “Standard ID” of sources used for calibration
 - ◇ isotopes of the self-absorption curve calibration planchets
 - ◇ “SV”, “SV Uncertainty”, and date of certified activity for calibration sources
 - ◇ residue weight of each source used for calibration
 - ◇ alpha and beta counts or count rate for each count collected
 - ◇ count time
 - ◇ calculated "Self-Absorption Factor" for each count collected
 - ◇ calculated “Crosstalk Factor” for each count collected
 - ◇ coefficients of best-fit curve for self-absorption curve with goodness of fit test result (correlation coefficient)
 - ◇ coefficients of best-fit curve for crosstalk curve(s) with goodness of fit test result
 - ◇ curve plots
 - ◇ analyst’s and reviewer’s signature and date
 - ◇ methodology SOP with revision number or date
 - ◇ copy of instrument run log

- If these items are non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data and assign the reason code [803] to all applicable data.
- Item 6-b** Verify that the curves were generated with 8 planchets well distributed over the expected mass range of sample planchets.
- If these items are non-compliant, professional judgment should be used to evaluate how this discrepancy affects the data based on the instrument calibrations not meeting the Statement of Work, comment, qualify and assign the reason code [R 106] to all data if applicable.
- Item 6-c** Verify that each alpha and beta calibration standard was counted to an accumulation of 10,000 counts.
- If this item is non-compliant, professional judgment should be used to evaluate how this discrepancy affects the data based on the instrument calibrations not meeting the Statement of Work, comment, qualify and assign the reason code [R 172] to all data if applicable.
- Item 6-d** Verify that the sources used for the determination of self absorption and cross-talk were of similar isotope content to that of the analytical sample.
- If this item is non-compliant, professional judgment should be used to evaluate how this discrepancy affects the data based on the instrument calibrations not meeting the Statement of Work, comment, qualify and assign the reason code [R 228] to all data if applicable.
- Item 6-e** Verify that the standard activity was decay corrected (if applicable) prior to calculation of instrument efficiencies.
- If this item is non-compliant, professional judgment should be used to evaluate how this discrepancy affects the data based on the instrument calibrations not meeting the Statement of Work, comment, qualify and assign the reason code [R 228] to all data if applicable.
- Item 6-f** Verify that the curve coefficients and goodness of fit statistics are reported for alpha and beta self absorption curve and alpha to beta crosstalk curves.
- If these items are non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data and assign the reason code [804] to all applicable data.

4.7. Check Source Requirement

Calibration DRC Items 7-a through 7-d

- Review Items:** Deliverable Check Source Requirement Package Section Number 7
- Objective:** To ensure that the gas proportional counters used for sample analysis are capable of producing quality results.
- Requirement Sources:** RC05 Exhibit E/Section 11, Exhibit B/Section 4, RC05 Appendix B-1

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

- Item 7-a** Verify that check sources were analyzed following gas changes, before samples were counted and/or daily.
- If check sources were not analyzed following gas changes, before samples were counted and/or daily these items are non-compliant, comment, qualify and qualify all applicable data as [R 141].
- Item 7-b** Verify that if the daily check source count results exceeded the tolerance limits or ± 3 sigma control limits that the laboratory recounted the check source to verify the out of control condition.
- If check sources were not recounted to verify out of control conditions this item is non-compliant, comment and qualify all applicable data as [R 141].
- Item 7-c** Verify that if the recount result exceeded the control limits again that the detectors were taken out of service until the system was brought back into control.
- If check source recount result exceeded the control limits again and the detectors were not taken out of service until the system was brought back into control this item is non-compliant, comment and qualify all applicable data as [R 141].
- Item 7-d** Verify that check source data was submitted with each reporting batch. If samples within a reporting batch are from separate counting batches verify that check source data is included for all counting batches.
- If check source data was not submitted this item is non-compliant, issue a Non-Compliance Notification to request any missing information. Do not qualify any data and assign the reason code [803] to all applicable data.

4.8. Instrument Backgrounds

Calibration DRC Items 8-a through 8-c

Review Items: Deliverable Instrument Background Requirement Package
Section Number 8

Objective: To ensure that the gas proportional counters used for sample analysis are capable of producing quality results.

Requirement Sources: RC05 Exhibit E/Section 11, Exhibit B/Section 4, RC05
Appendix B-1

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

- Item 8-a** Verify that Instrument Backgrounds for both alpha and beta were determined weekly.
- If Instrument Backgrounds were not determined weekly this item is non-compliant, do not qualify any data. Comment and assign the reason code [246] to all applicable data.
- Item 8-b** Verify that the count time for the backgrounds were the same as the routine sample count time.

- If Instrument Backgrounds count times were not the same as the routine sample count time this item is non-compliant, do not qualify any data. Comment and assign the reason code [246] to all applicable data.
- Item 8-c** Verify that the instrument backgrounds and uncertainties used in calculating sample analyte activities were based on the average of at least 5 background counts.
- If Instrument Backgrounds count times were not based on an average of at least 5 background counts this item is non-compliant, do not qualify any data. Comment and assign the reason code [246] 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 RC05 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-RC05-v1, prepared by Kip Harward of Kaiser-Hill Analytical Services, is the first issue of this procedure. DA-RC05-v1 was used for verification and validation of the first SDPs received according to PSA Module RC05.

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Attachment 1: Data Quality Assessment Report Template

RC05

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		
Preparation Summary		
Standards Summary		
Instrument Calibration Summary		
Continuing Calibration Summary		
Counting Raw Data Summary		
EDD		
INSTRUMENT CALIBRATION PACKAGE:		
Structural Requirements		
General Requirements		
Voltage Plateau		
Discriminator Window Settings		
Efficiency Calibration		
Self-Absorption/Crosstalk Curve Calibration		
Check source Requirements		
Instrument Backgrounds		
Other:		

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

Action Items:

Comments:

Verification/Validation Signature _____

Date: _____

Reviewer Signature _____

Date: _____

(Validation Only)