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(formerly **SOP-15.06-R1 ICN1**
and **SOP-15.07-R1 ICN1**)

Revision: **0**



Effective Date: 6/30/08

Next Review Date: 6/30/13

Waste & Environmental Services

Standard Operating Procedure

for **ROUTINE VALIDATION OF GAMMA SPECTROSCOPY, CHEMICAL SEPARATION ALPHA SPECTROMETRY, GAS PROPORTIONAL COUNTING, AND LIQUID SCINTILLATION ANALYTICAL DATA**

APPROVAL SIGNATURES:

| Subject Matter Expert: | Organization | Signature | Date |
|-------------------------------|--------------|-------------------|---------|
| Bill Hardesty | WES-EDA | Signature on file | 4/21/08 |
| Quality Assurance Specialist: | Organization | Signature | Date |
| Laura Ortega | QA-IQ | Signature on file | 5/14/08 |
| Responsible Line Manager: | Organization | Signature | Date |
| Craig Eberhart | WES-EDA | Signature on file | 4/21/08 |

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1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine radionuclide analytical data, including:

- Gamma-emitting isotopes by gamma spectroscopy;
- Alpha-emitting isotopes (americium-241; uranium-234, -235, and -238; thorium -230, -232, and -234; and plutonium-238 and -239/-240) by chemical separation alpha spectrometry;
- Strontium-90 by gas proportional counting (GPC);
- Gross alpha and beta analyses by GPC; and
- Tritium by liquid scintillation.

2.0 BACKGROUND AND PRECAUTIONS

2.1 Background

This procedure conforms to the requirements of Environmental Protection Agency (EPA) Methodologies. LANL data validation is performed according to procedures based upon the NNSA Model Data Validation Procedure. Data qualifiers and reason codes are assigned according to the specifications in this method specific procedure.

2.2 Precautions

Nothing in this procedure precludes the data validator from going beyond the minimum requirements specified within this procedure. If additional directions are required, the data validator shall reference NNSA Model Data Validation Procedure, EPA method specific guidelines. Implementation of this procedure may be followed by a more focused and data use-specific evaluation of the data by the project chemist, especially if the implementation of this procedure indicates the data may contain technical deficiencies.

3.0 EQUIPMENT AND TOOLS

None.

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1 Qualifications for Data Validators

| | | |
|----------------|----|--|
| Data Validator | 1. | Possess a minimum of a bachelor's degree in chemistry, or one of the physical sciences AND either two (2) years of experience in generating analytical data in an environmental analytical laboratory AND two (2) years of data validation experience. |
| | 2. | Complete Attachment 1, Data Validation Cover Sheet, and Attachment 2, Rad Analytical Data Validation Checklist, during data validation. |

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Data Validator (Continued) 3. Refer to Attachment 3, Guidance for the Qualifier and Reason Code Application, for additional guidance.

4.2 Records

Data Validator 1. Submit the following records generated by this procedure to the Records Processing Facility:

- Completed Data Validation Cover Sheets; and
- Completed Rad Analytical Data Validation Checklists.

5.0 PROCESS FLOW CHART

For specific validation criteria follow the NNSA Model for Data Validation.

6.0 ATTACHMENTS

Attachment 1 5166-1 Data Validation Cover Sheet (1 page)

Attachment 2 5166-2 Rad Analytical Data Validation Checklist (3 pages)

Attachment 3 5166-3 Guidance for the Qualifier and Reason Code Application (3 pages)

7.0 REVISION HISTORY

Author: Bill Hardesty

| Revision No. <i>[Enter current revision number, beginning with Rev.0]</i> | Effective Date <i>[DCC inserts effective date for revision]</i> | Description of Changes <i>[List specific changes made since the previous revision]</i> | Type of Change <i>[Technical (T) or Editorial (E)]</i> |
|--|--|---|---|
| 0 | 6/30/08 | New Document | T |

[Using a CRYPTOCARD, click here to record "self-study" training to this procedure.](#)

If you do not possess a CRYPTOCARD or encounter problems, contact the EP training specialist.

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ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

5166-1

Example of a Data Validation Cover Sheet

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Section I.

REQUEST NUMBER: _____ VALIDATION DATE: _____ LAB CODE: _____

CONTRACT LABORATORY NAME: _____

VALIDATOR: _____ ORGANIZATION: _____

ANALYTICAL SUITE (CHECK ALL THAT APPLY):

- | | | | |
|--|--|--|---|
| <input type="checkbox"/> TPH-GRO | <input type="checkbox"/> HIGH EXPLOSIVES | <input type="checkbox"/> DIOXIN FURANS | <input type="checkbox"/> LCMSMS PERCHLORATES |
| <input type="checkbox"/> TPH-DRO | <input type="checkbox"/> METALS | <input type="checkbox"/> PCB CONGENERS | <input type="checkbox"/> ORGANOCHLORINE PESTICIDES/POLYCHLORINATED |
| <input type="checkbox"/> GENERAL CHEMISTRY | <input type="checkbox"/> RADIOCHEMISTRY | <input type="checkbox"/> LCMSMS HIGH EXPLOSIVES | <input type="checkbox"/> BIPHENYLS |

OTHER (DESCRIBE): _____

Section II. Completeness Check

| YES | NO | N/A | (CHECK ONE) | YES | NO | N/A | (CHECK ONE) |
|--------------------------|--------------------------|--------------------------|-----------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 1. CHAIN-OF-CUSTODY FORM(S) | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 6. RAW/BSS DATA |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 2. CASE NARRATIVE | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 7. QUALITY CONTROL FORMS |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 3. SAMPLE RESULT FORMS | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 8. QUANTITATION REPORTS |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 4. SAMPLE CHROMATOGRAMS | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 9. TICS FORMS |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 5. STANDARD CHROMATOGRAMS | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 10. TICS MASS SPECTRA |

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

VALIDATOR'S SIGNATURE: _____ DATE: _____

SOP-5166, Revision 0.0

LOS ALAMOS
Environmental Restoration Project

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ATTACHMENT 2: RAD ANALYTICAL DATA VALIDATION CHECKLIST

5166-2

Rad Analytical Data Validation Checklist

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| Yes No N/A (Check One) | | | | Assign Qualifier Listed Below If Criterion = Yes | |
|-------------------------------|--------------------------|--------------------------|---|---|---------------------|
| | | | | Non-detected Analyte | Detected Analyte |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 1. The holding time was >1 and ≤2 times the applicable holding time requirement. | UJ, R9 | J-, R9 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 2. The holding time was >2 times the applicable holding time requirement. | R, R9a | J-, R9a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 3. The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to the MDC. | N/A | U, R5 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 4. The analyte should be regarded as rejected because spectral interferences prevent positive identification of the analytes. | R, R5a | R, R5a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 5. The MDC and/or TPU documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, R5b | R, R5b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 6. The results for the affected analytes should be regarded as not detected (U) because the associated sample concentration was less than 3X the 1 sigma TPU. | N/A | U, R11 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 7. The sample result is ≤5X the concentration of the related analyte in the method blank. | N/A | U, R4 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 8. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5X. | N/A | J+, R4a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 9. The sample result is ≤5X the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank. | N/A | U, R4d |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 10. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, R4e | R, R4e |

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| Yes No N/A (Check One) | | | | Assign Qualifier Listed Below If Criterion = Yes | |
|-------------------------------|--------------------------|--------------------------|---|---|---------------------|
| | | | | Non-detected Analyte | Detected Analyte |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 11. The tracer is <10%R. Follow the external laboratory limits located within the associated data package. Tracer%R is not applicable for Gamma Spectroscopy. | R, R3 | R, R3 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 12. The tracer is < the Lower Acceptance Level (LAL) but ≥10%R. Follow the external laboratory limits located within the associated data package. Tracer%R is not applicable for Gamma Spectroscopy. | UJ, R3a | J-, R3a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 13. The Tracer%R value is > the Upper Acceptance Limit (UAL). Follow the external laboratory limits located within the associated data package. Tracer%R is not applicable for Gamma Spectroscopy. | N/A | J+, R3b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 14. Required tracer information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. Tracer%R is not applicable for Gamma Spectroscopy. | R, R3d | R, R3d |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 15. The LCS percent recovery was <10%. Follow the external laboratory limits located within the associated data package. | R, R12 | R, R12 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 16. The LCS percent recovery was < the LAL but >10%. Follow the external laboratory limits located within the associated data package. | UJ, R12a | J-, R12a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 17. The LCS percent recovery was > the UAL. Follow the external laboratory limits located within the associated data package. | N/A | J+, R12b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 18. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, R12c | R, R12c |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 19. Associated duplicate sample has DER or RER > the analytical laboratory's acceptance limits. | R, R10 | J, R10 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 20. The duplicate sample was not prepared and/or analyzed with the samples for unspecified reasons. The duplicate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, R6 | R, R6 |

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| Yes No N/A (Check One) | | | | Assign Qualifier Listed Below If Criterion = Yes | |
|-------------------------------|--------------------------|--------------------------|--|---|---------------------|
| | | | | Non-detected Analyte | Detected Analyte |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 21. The associated matrix spike recovery was <10%. Follow the external laboratory limits. MS/MSD is not applicable to Gamma Spectroscopy. | UJ, R6a | R, R6a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 22. The associated matrix spike recovery was less than the LAL but greater than 10%. Follow the external laboratory limits. MS/MSD is not applicable to Gamma Spectroscopy. | UJ, R6b | J-, R6b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 23. Required matrix spike information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. If LCS information is present, do not Reject. Qualify data based on LCS information. MS/MSD is not applicable to Gamma Spectroscopy. | R, R6c | R, R6c |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 24. Duplicate, dilution, or reanalysis. | UJ, R88 | J, R88 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 25. The LANL project chemist identified quality deficiencies in the reported data that require further qualification. This code can ONLY be used and/or under advisement by the LANL project chemist. | UJ, R, R19 | J, R, R19 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 26. Quantification of data via data validation did not occur based on Quality Control requirements in this procedure. Adhere to the external laboratory qualifiers found within the Form I analytical data summary sheets generated by the external laboratory. | U, U_LAB | J, J_LAB NQ, NQ |

ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

5166-3

Guidelines for the Qualifier and Reason Code Application

Records Use only



| No. | Valid Flag Code Nondetect | Valid Flag Code Detect | Valid Reason Code | Valid Reason Description |
|-----|---------------------------|------------------------|-------------------|--|
| 1 | UJ | J- | R9 | The holding time was >1 and ≤2 times the applicable holding time requirement. |
| 2 | R | J- | R9a | The holding time was >2 times the applicable holding time requirement. |
| 3 | N/A | U | R5 | The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to the MDC. |
| 4 | R | R | R5a | The analyte should be regarded as rejected because spectral interferences prevent positive identification of the analytes. |
| 5 | R | R | R5b | The MDC and/or TPU documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |
| 6 | N/A | U | R11 | The results for the affected analytes should be regarded as not detected (U) because the associated sample concentration was less than 3X the 1 sigma TPU. |
| 7 | N/A | U | R4 | The sample result is ≤5X the concentration of the related analyte in the method blank. |
| 8 | N/A | J | R4a | The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5X. |
| 9 | N/A | U | R4d | The sample result is ≤5X the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank. |

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| No. | Valid Flag Code Nondetect | Valid Flag Code Detect | Valid Reason Code | Valid Reason Description |
|-----|---------------------------|------------------------|-------------------|---|
| 10 | R | R | R4e | Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |
| 11 | R | R | R3 | The tracer is <10%R. Follow the external laboratory limits located within the associated data package. Tracer%R is not applicable for Gamma Spectroscopy. |
| 12 | UJ | J- | R3a | The tracer is < the Lower Acceptance Level (LAL) but ≥10%R. Follow the external laboratory limits located within the associated data package. Tracer%R is not applicable for Gamma Spectroscopy. |
| 13 | N/A | J+ | R3b | The Tracer%R value is > the Upper Acceptance Limit (UAL). Follow the external laboratory limits located within the associated data package. Tracer%R is not applicable for Gamma Spectroscopy. |
| 14 | R | R | R3d | Required tracer information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. Tracer%R is not applicable for Gamma Spectroscopy. |
| 15 | R | R | R12 | The LCS percent recovery was <10%. Follow the external laboratory limits located within the associated data package. |
| 16 | UJ | J- | R12a | The LCS percent recovery was < the LAL but >10%. Follow the external laboratory limits located within the associated data package. |
| 17 | N/A | J+ | R12b | The LCS percent recovery was > the UAL. Follow the external laboratory limits located within the associated data package. |
| 18 | R | R | R12c | The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |
| 19 | R | J | R10 | Associated duplicate sample has DER or RER > the analytical laboratory's acceptance limits. |
| 20 | UJ | J | R10d | The duplicate sample was not prepared and/or analyzed with the samples for unspecified reasons. The duplicate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |

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| No. | Valid Flag Code Nondetect | Valid Flag Code Detect | Valid Reason Code | Valid Reason Description |
|-----|---------------------------|------------------------|-------------------|--|
| 21 | R | R | R6 | The associated matrix spike recovery was <10%. Follow the external laboratory limits. MS/MSD is not applicable to Gamma Spectroscopy. |
| 22 | UJ | J- | R6a | The associated matrix spike recovery was <10%. Follow the external laboratory limits. MS/MSD is not applicable to Gamma Spectroscopy. |
| 23 | UJ | J+ | R6b | The associated matrix spike recovery was above the UAL. Follow the external laboratory limits. MS/MSD is not applicable to Gamma Spectroscopy. |
| 24 | R | R | R6c | Required matrix spike information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. If LCS information is present, do not Reject. Qualify data based on LCS information. MS/MSD is not applicable to Gamma Spectroscopy. |
| 25 | UJ | J | R88 | Duplicate, dilution, or reanalysis. |
| 26 | UJ, R | J, R | R19 | The LANL project chemist identified quality deficiencies in the reported data that require further qualification. This code can ONLY be used and/or under advisement by the LANL project chemist. |
| 27 | U | J, NQ | U_LAB, J_LAB, NQ | Quantification of data via data validation did not occur based on Quality Control requirements in this procedure. Adhere to the external laboratory qualifiers found within the Form I analytical data summary sheets generated by the external laboratory. |

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| | | |
|--|---------------|---------------|
| Title: Routine Validation of Gamma Spectroscopy, Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data | No.: SOP-5166 | Page 11 of 11 |
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