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Environment, Safety, and Health Directorate

OIO Operations Integration Office

Technical Procedure

Routine Validation of Gamma Spectroscopy, Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Analytical Data

Subject Matter Expert:

Name:	Organization:	Signature:	Date:
Keith Greene	OIO-DO		

Derivative Classifier: ☐ **Unclassified or** ☒ **DUSA ENVPRO**

Name:	Organization:	Signature:	Date:
Linda Salazar	ADESH/SI-DC		

Approval Signatures:

Quality Assurance Reviewer:	Organization:	Signature:	Date:
Doris Quintana	QPA-IQ		
Responsible Line Manager:	Organization:	Signature:	Date:
Ellen Gammon	WM-PROG		

REVISION HISTORY

Document Number and Revision *[Include revision number, beginning with Revision 0]*	**Effective Date** *[Document Control Coordinator inserts effective date]*	**Description of Changes** *[List specific changes made since the previous revision]*
OIO-TP-5166, Rev. 0	8/2/2016	New Document, changed Doc # from SOP-5166 to OIO-TP-5166.

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1.0 PURPOSE AND SCOPE

This procedure presents the minimum standards used at Los Alamos National Laboratory (LANL) to evaluate 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 U.S. Environmental Protection Agency (EPA) methodologies. LANL data validation is performed according to procedures based upon the National Nuclear Security Administration (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 herein. If additional directions are required, the data validator shall reference NNSA Model Data Validation Procedure and 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 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, Attachment 2, Rad Analytical Data Validation Checklist, during data validation.
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
   - 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 OR APPENDICES

Attachment 1: *5166-1 Data Validation Cover Sheet*
Attachment 2: *5166-2 Rad Analytical Data Validation Checklist*
Attachment 3: *5166-3 Guidance for the Qualifier and Reason Code Application*

ATTACHMENT 1 – 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):

☐ TPH-GRO ☐ HIGH EXPLOSIVES ☐ DIOXIN FURANS ☐ LCMSMS PERCHLORATES

☐ TPH-DRO ☐ METALS ☐ PCB CONGENERS ☐ ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS

☐ GENERAL CHEMISTRY ☐ RADIOCHEMISTRY ☐ LCMSMS HIGH EXPLOSIVES

☐ OTHER (DESCRIBE): _____

Section II. Completeness Check

YES	NO	N/A	(CHECK ONE)	YES	NO	N/A	(CHECK ONE)
☐	☐	☐	1. CHAIN-OF-CUSTODY FORM(S)	☐	☐	☐	6. RAW/BSS DATA
☐	☐	☐	2. CASE NARRATIVE	☐	☐	☐	7. QUALITY CONTROL FORMS
☐	☐	☐	3. SAMPLE RESULT FORMS	☐	☐	☐	8. QUANTITATION REPORTS
☐	☐	☐	4. SAMPLE CHROMATOGRAMS	☐	☐	☐	9. TICS FORMS
☐	☐	☐	5. STANDARD CHROMATOGRAMS	☐	☐	☐	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: _____

ATTACHMENT 2 – RAD ANALYTICAL DATA VALIDATION CHECKLIST

Page 1 of 3

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	1. The holding time was >1 and ≤2 times the applicable holding time requirement.	UJ, R9	J-, R9
☐	☐	☐	2. The holding time was >2 times the applicable holding time requirement.	R, R9a	J-, R9a
☐	☐	☐	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
☐	☐	☐	4. The analyte should be regarded as rejected (R) because spectral interferences prevent positive identification of the analytes.	R, R5a	R, R5a
☐	☐	☐	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
☐	☐	☐	6. The results for the affected analytes should be regarded as not detected (U) because the associated sample concentration was less than 3 times the 1 sigma TPU.	N/A	U, R11
☐	☐	☐	7. The sample result is ≤5 times the concentration of the related analyte in the method blank.	N/A	U, R4
☐	☐	☐	8. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5 times.	N/A	J+, R4a
☐	☐	☐	9. The sample result is ≤5 times the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank.	N/A	U, R4d
☐	☐	☐	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

ATTACHMENT 2 – RAD ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)

Page 2 of 3

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	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
☐	☐	☐	12. The tracer is less than 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
☐	☐	☐	13. The tracer%R value is greater than 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
☐	☐	☐	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
☐	☐	☐	15. The LCS %R was <10%. Follow the external laboratory limits located within the associated data package.	R, R12	R, R12
☐	☐	☐	16. The LCS %R was < the LAL but >10%. Follow the external laboratory limits located within the associated data package.	UJ, R12a	J-, R12a
☐	☐	☐	17. The LCS %R was > the UAL. Follow the external laboratory limits located within the associated data package.	N/A	J+, R12b
☐	☐	☐	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
☐	☐	☐	19. Associated duplicate sample has DER or RER greater than the analytical laboratory's acceptance limits.	R, R10	J, R10

ATTACHMENT 2 – RAD ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)

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Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	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
☐	☐	☐	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
☐	☐	☐	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
☐	☐	☐	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
☐	☐	☐	24. Duplicate, dilution, or reanalysis.	UJ, R88	J, R88
☐	☐	☐	25. The LANL project chemist identified quality deficiencies in the reported data that require further qualification. This code can be used ONLY under advisement of the LANL project chemist.	UJ, R, R19	J, R, R19
☐	☐	☐	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 – GUIDANCE FOR THE QUALIFIER AND REASON CODE APPLICATION

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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 3 times the 1-sigma TPU.
7	N/A	U	R4	The sample result is ≤5 times 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 >5 times the concentration.
9	N/A	U	R4d	The sample result is ≤5 times the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank.
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.

ATTACHMENT 3 – GUIDANCE FOR THE QUALIFIER AND REASON CODE APPLICATION (CONT.)

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No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
12	UJ	J-	R3a	The tracer is less than the 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 greater than the 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 %R was <10%. Follow the external laboratory limits located within the associated data package.
16	UJ	J-	R12a	The LCS %R was < the LAL but >10%. Follow the external laboratory limits located within the associated data package.
17	N/A	J+	R12b	The LCS %R 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 greater than 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.

ATTACHMENT 3 – GUIDANCE FOR THE QUALIFIER AND REASON CODE APPLICATION (CONT.)

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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 be used ONLY under advisement of 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.

From: Hollis, Diana J
Sent: Tuesday, August 23, 2016 11:08 AM
To: Maestas, Pamela Therese
Subject: RE: DC review of ADESH procedures

Hi Pamela,

All of the procedures are covered by at least one DUSA, so they're approved for posting.

Diana

From: Maestas, Pamela Therese
Sent: Tuesday, August 23, 2016 11:01 AM
To: Hollis, Diana J <dhollis@lanl.gov>
Subject: DC review of ADESH procedures

Hi Diana,
At your leisure, can you please DC review the attached ADESH procedures (EPC-CP-TPP-MetM, EPC-ES-TP-013, OIO-TP-5166, OIO-TP-5171, and OIO-TP-5191). These need to be posted to the EPRR because they are referenced in ADEM/ADESH deliverables.
Thank you.

Pamela T. Maestas
Associate Directorate for Environmental Management
Los Alamos National Laboratory
(505) 665-9042 or (505) 927-7882 cell