Identifier: **SOP-5166** (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	Los Alamos NATIONAL LABORATORY — EST.1943 —

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

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.

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. *[Enter current revision number, beginning with Rev.0]*	Effective Date *[DCC inserts effective date for revision]*	Description of Changes *[List specific changes made since the previous revision]*	Type of Change *[Technical (T) or Editorial (E)]*
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.

ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

5166-1 **Example of a Data Validation Cover Sheet**	Records Use only **Los Alamos** NATIONAL LABORATORY — EST. 1943 —

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: _____

SOP-5166, Revision 0.0	LOS ALAMOS Environmental Restoration Project

ATTACHMENT 2: RAD ANALYTICAL DATA VALIDATION CHECKLIST

5166-2 **Rad Analytical Data Validation Checklist**	Records Use only Los Alamos NATIONAL LABORATORY — EST.1943 —

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 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 3X the 1 sigma TPU.	N/A	U, R11
☐	☐	☐	7. The sample result is ≤5X 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 >5X.	N/A	J+, R4a
☐	☐	☐	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
☐	☐	☐	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

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 < 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 > 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 percent recovery was <10%. Follow the external laboratory limits located within the associated data package.**	**R, R12**	**R, R12**
☐	☐	☐	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**
☐	☐	☐	17. **The LCS percent recovery 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 > the analytical laboratory's acceptance limits.**	**R, R10**	**J, R10**
☐	☐	☐	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**

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				**Non-detected Analyte**	**Detected Analyte**
☐	☐	☐	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 ONLY be used and/or under advisement by 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: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION	
5166-3 **Guidelines for the Qualifier and Reason Code Application**	Records Use only **Los Alamos** NATIONAL LABORATORY — EST.1943 —

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.

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.

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.

This page left blank intentional.