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

Effective Date: 4/24/2017

Next Review Date: 4/24/2020

Hazard Class: ☒ Low ☐ Moderate ☐ High/Complex

Usage Mode: ☒ Reference ☐ UET ☐ Both UET & Reference

The Responsible Manager has determined that the following organizations' review is required for initial procedure release as well as subsequent major revisions. Review documentation is contained in the Document History File.

Technical Leads Quality Assurance

Classification Review: ☒ Unclassified ☐ UCNI ☐ Classified

Diana Hollis	/ 111125	/ /s/ Diana Hollis	/ 4/18/2017
Name (print)	Z#	Signature	Date

Responsible Manager, Division and Title

Nita Patel	/ 153003	/ /s/ Nita Patel	/ 4/20/2017
Name (print)	Z#	Signature	Date

REVISION HISTORY

Document No./Revision No.	Issue Date	Action	Description
OIO-TP-5166, Rev. 0	8/2/2016	New	New Document, changed Doc # from SOP-5166 to OIO-TP-5166.
ER-AP-20314, R0	4/24/2017	Major Revision	Revised to remove NNSA Model Validation

TABLE OF CONTENTS

1. PURPOSE

This procedure establishes guidance for the qualification of radionuclide analytical data. This document is intended to assist in the technical review of analytical data generated by environmental laboratories.

2. SCOPE

Qualification of data is the product of data validation, analytical laboratory analysis, and focused validation that describe validation anomalies and their consequences. Radiochemistry measurements include:

- 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.

3. BACKGROUND

Data qualifiers and reason codes are assigned to analytical results from radiochemical analyses according to the specifications in this method-specific procedure. This procedure conforms to the requirements of U.S. Environmental Protection Agency (EPA) methodologies. Data qualifiers and reason codes are assigned according to the specifications in this method specific procedure.

4. 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 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.

5. PREREQUISITE ACTIONS

Data Validators must:

- 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 or two (2) years of experience in data validation.
- Complete Attachment 1, Data Validation Cover Sheet, and Attachment 2, Radiochemistry Analytical Data Validation Checklist, during data validation.

6. PERFORMANCE

6.1 Validation Process

EIM applies a subset of qualifiers described in this procedure to analytical data using auto-validation subroutines. EIM auto-validation applies qualification to analytical records using tests listed in Attachment 2 that have a Valid Reason Description containing "(AV)". When the project leader requests a focused validation the assigned data validator completes the following steps to assess all potential analytical data qualification:

[1] **REVIEW** the qualifiers assigned during EIM auto-validation to verify that qualifiers were assigned consistently with this procedure. If auto-validation qualification is found to be inconsistent with this procedure then the validator initiates a change request using ER-AP-20304, Change Control for Data in the Environmental Information Management (EIM) Database.

[2] **PRINT** Attachment 1 and **REVIEW** the data package for potential qualification using Attachment 2.

[3] **NOTE** conditions causing recommendation for qualification and options for qualification.

[4] **COMPLETE** Attachment 1 and **FORWARD** to the project leader with conditions and options.

6.1 Validation Process (continued)

The project leader is the responsible party for making the decision of record if validation qualifiers should be assigned and EIM validation records updated. This record of decision is added to comments section of Attachment 1.

Once the decision of record has been made, Attachment 1 is sent to the Sample Management Office (SMO) staff. The SMO staff re-print the data validation record from EIM and add Attachment 1 that includes the record of decision to the final records package.

6.2 Analyte Quantitation

The assignment of the detection status to analytical measurements is the first step of analytical data validation. Most validation qualifiers and validation reason codes are applied based on the measurement's initial detection status. Results that are less than the report minimum detectable activity (MDA) are qualified as nondetect with the U validation qualifier and R5 validation reason code. Results greater than or equal to the MDL are qualified as detected with the NQ validation qualifier.

Criteria	Validation Qualifier	Validation Reason Code
Target analyte result is < MDA; a nondetect	U	R5
Target analyte result is ≥ MDA; a detect	NQ	NQ

Since a result can have only one validation qualifier and one validation reason code the sequencing of validation steps is important. Analyte quantitation occurs first, then analyte identification, because most other validation functions depend on the correct identification and quantitation of the analytical parameter. When two or more qualifiers can be applied to a record, the qualifier representing the more severe consequence to data usability supersedes the qualifier with less severe consequence. The R validation qualifier has the greatest impact on data usability and supersedes other validation qualifiers.

6.2 Analyte Quantitation (continued)

Order Of Severity	Validation Qualifier	Description
1	R	The reported sample result is classified as rejected due to serious non-compliances regarding quality control acceptance criteria. The presence or absence of the analyte cannot be verified.
2	UJ	The analyte is classified as not detected, with an expectation that the reported result is more uncertain than usual.
3	U	The analyte is classified as not detected.
4	J	The analyte is classified as detected but the reported concentration value is expected to be more uncertain than usual.
5	NQ	No validation qualifier flag is associated with this result, and the analyte is classified as detected.

LANL project chemists may identify quality deficiencies in analytical results affecting analyte quanitation. These deficiencies can include analytical results with detection limits elevated above project data-quality objectives, concentrations above the calibration range of the instrument or method, results exhibiting carryover or detector contamination, spectral interference from another radioisotope, and other quality deficiencies. The reason code of R19 is applied to affected records by the project chemist to identify these quality deficiencies when they are identified.

6.3 Analyte Identification

The identification of an analytical parameter is the second step of analytical data validation. Identification of radioisotopes depends upon the energy signature of radioactive decay, isotope luminescence, and/or chemical separation. When counting uncertainty, peak-width, or abundance do not meet criteria the R5a reason code is applied to affected parameters.

6.4 Holding Times and Sample Preservation

Sample handling requirements are specified to ensure integrity and defensibility of analytical measurements. Samples are to be prepared and analyzed within specified time limits. Samples are also preserved chemically with the addition of acids and physically by controlling temperature. When sample handling requirements are not met the R9 series of reason codes are applied to affected samples.

Reference

6.5 Initial and Continuing Calibration

Calibration is performed to ensure that the instrument is performing within specifications. The calibration establishes the energy/channel relationship, counting efficiency, and energy resolution. Instrument performance checks are performed periodically to confirm the instrument remains within operating specifications. When a calibration has expired the R7 series of reason codes are applied to affected analytes in all samples analyzed since the expiration of the calibration for that instrument. When an instrument performance check is not performed at the analytical laboratory's established frequency or is not within limits the R7 series of reason codes is applied to affected analytes in all samples analyzed after the unacceptable instrument performance check to the next acceptable instrument performance check for that instrument.

6.6 Carrier

Carriers are compounds not normally found in the environment, but which have quantitation limits and energies similar to the radioisotopes of interest in a sample. Carriers are added to samples, standards, and QC samples to determine the effectiveness of analyte quantitation and chemical separation. Sample results are not adjusted based on carrier recoveries. When carrier recovery criteria are not met the R1 series of reason codes are applied to affected samples.

6.7 Tracer

Tracers are compounds not normally found in the environment, but which are easily measurable. They are added to samples, standards, and QC samples to compensate for fluctuations in the analytical system. Sample results are quantitated or adjusted by the relative response of associated tracers. When tracer criteria are not met the R3 series of reason codes are applied to the affected sample.

6.8 Blanks

The Method Blank is an analyte-free matrix that is prepared and analyzed in the laboratory with the samples. The method blank determines contamination from the analytical processes. Method blanks are prepared with every preparation batch. If more than one method blank is associated with a given sample, qualification is based upon a comparison with the associated blank having the highest concentration of the parameter. When method blank criteria are not met the R4 series of reason codes are applied affected samples.

The Field Blank is an analyte-free matrix opened to the atmosphere at the time of sample collection. Field blanks are used to determine if atmospheric conditions resulted in contamination of samples during sample collection. Samples collected the same day as the field blank that does not meet blank criteria are qualified with the R4 series of reason codes.

The Equipment Blank is an analyte-free matrix poured over or through sample collection equipment. Equipment blanks are used to determine the cleaning effectiveness of sampling equipment between samples. Samples collected using the same tools as the equipment blank that does not meet blank criteria are qualified with the R4 series of reason codes.

6.9 Laboratory Control and Matrix Spike Samples

The laboratory control sample is created by adding known amounts of parameters of interest to an aliquot of a blank matrix. The laboratory control sample is used to evaluate the effect of the analytical process of the recovery of analytes. When laboratory control sample criteria are not met the R12 series of reason codes are applied to all associated samples.

The matrix spike is created by adding known amounts of parameters of interest to an aliquot of a sample matrix. The matrix spike is used to evaluate the effect of the sample matrix on the recovery of analytes. When matrix spike criteria are not met the R6 series of reason codes are applied to all associated samples.

6.10 Sample Duplicate

Field duplicate samples are collected from the same material at the same time as the primary sample. The relative percent difference between the results of the parent sample and the field duplicate sample is used to determine the field and laboratory precision of the analytical measurement. When field duplicate precision criteria are not met the appropriate R10 reason code is applied to the parent sample.

7. RECORDS

Records generated by this procedure will be submitted to the Environmental Protection Records Management Office for document management in accordance with Institutional Records Management Procedure, P1020-1 and EP-AP-10003, Records Management.

- Completed Data Validation Cover Sheets
- Completed Radiochemistry Analytical Data Validation Checklists

8. REFERENCES

EP-AP-10003, Records Management

ER-AP-20304, Change Control for Data in the Environmental Information Management (EIM) Database

P1020-1, Laboratory Records Management

9. ATTACHMENTS

Attachment 1: Data Validation Cover Sheet
Attachment 2: Radiochemistry Analytical Data Validation Checklist

ATTACHMENT 1

Page 1 of 1

Data Validation Cover Sheet

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 & Cyanide** ☐ **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: _____

ER-AP-20314, R0	Los Alamos Environmental Safety & Health
	(Attach additional comment sheets as necessary)

ATTACHMENT 2

Page 1 of 4

Radiochemistry Analytical Data Validation Checklist

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
Holding Times and Sample Preservation					
□	□	□	1. The affected radionuclide is regarded as rejected because the analytical holding time was exceeded, 28 days C-14, I-129, I-131 in water, 180 days all others. (AV)	R, R9b	J-, R9b
□	□	□	2. C-14, I-129, I-131 temperature > 10°C upon receipt at the laboratory.	UJ, R9c	J-, R9c
Instrument Calibration					
□	□	□	3. Instrument calibration or instrument performance check is expired or deficient.	UJ, R7	J, R7
□	□	□	4. Instrument calibration not performed.	R, R7a	R, R7a
Blanks					
□	□	□	5. The sample result is ≤5 times the concentration of the related analyte in the method blank. (AV)	N/A	U, R4
□	□	□	6. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5 times. (AV)	N/A	J+, R4a
□	□	□	7. 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
□	□	□	8. 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

Page 2 of 4

Yes No N/A (Check One)				Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	**Detected Analyte**
Tracer/Carrier					
□	□	□	9 . The tracer/carrier 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
□	□	□	10. The tracer/carrier is less than the lower acceptance level (LAL) but ≥10%R. Follow the external laboratory limits located within the associated data package. tracer/carrier %R is not applicable for gamma spectroscopy.	UJ, R3a	J-, R3a
□	□	□	11. The tracer/carrier %R value is greater than the upper acceptance limit (UAL). Follow the external laboratory limits located within the associated data package. tracer/carrier %R is not applicable for gamma spectroscopy.	N/A	J+, R3b
□	□	□	12. Required tracer/carrier information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. tracer/carrier %R is not applicable for gamma spectroscopy.	R, R3d	R, R3d
Laboratory Control Samples					
□	□	□	13. The LCS %R was <10%. Follow the external laboratory limits located within the associated data package. (AV)	R, R12	R, R12
□	□	□	14. The LCS %R was < the LAL but >10%. Follow the external laboratory limits located within the associated data package. (AV)	UJ, R12a	J-, R12a
□	□	□	15. The LCS %R was > the UAL. Follow the external laboratory limits located within the associated data package. (AV)	N/A	J+, R12b
□	□	□	16. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, R12c	R, R12c

ATTACHMENT 2

Page 3 of 4

Yes No N/A (Check One)				Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	**Detected Analyte**
Matrix Spike					
□	□	□	17. The associated matrix spike recovery was <10%. Follow the external laboratory limits. MS/MSD is not applicable to gamma spectroscopy.	UJ, R6a	R, R6a
□	□	□	18. 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
□	□	□	19. 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
Duplicate					
□	□	□	20. Associated duplicate sample has DER or RER greater than the analytical laboratory's acceptance limits. (AV)	R, R10	J, R10
□	□	□	21. 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, R10d	R, R10d

ATTACHMENT 2

Page 4 of 4

Analyte Quantitation					
			22. The non-detected analytes have elevated detection limits and may not meet project data-quality objectives because the sample was diluted without any target analytes identified as a result of matrix interference. Reject non-detected results if the analytical laboratory cannot provide proof for matrix interference.	UJ, R, R15	NA
□	□	□	23. The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to the MDC. (AV)	N/A	U, R5
□	□	□	24. The analytical laboratory qualified the result with UI. The result is greater than MDA but is a non-identified nuclide.	U, R5a	U, R5a
□	□	□	25. Interferences prevent positive identification of the detected analyte or misidentification of the non-detected analyte.	R, R5a	R, R5a
□	□	□	26. 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
□	□	□	27. 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
□	□	□	28. Qualification of data via data validation did occur, however no data quality control requirements in this procedure were applicable. Adhere to the external laboratory qualifiers found within the Form 1 analytical data summary sheets generated by the external laboratory. (AV)	U, U_LAB	J, J_LAB NQ, NQ