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Original

ENGINEERING CHANGE NOTICE

12-20-93 JB 20

Page 1 of 2

1. ECN 602830

Proj. ECN

2. ECN Category (mark one)	3. Originator's Name, Organization, MSIN, and Telephone No.		4. Date
Supplemental [ ] Direct Revision [x] Change ECN [ ] Temporary [ ] Standby [ ] Supersedure [ ] Cancel/Void [ ]	Karl N. Pool, Technical and Quality Oversite, Hanford Analytical Services Management, H4-23, 372-2557		10-21-93
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11a. Modification Work	11b. Work Package No.	11c. Modification Work Complete	11d. Restored to Original Condition (Temp. or Standby ECN only)
[ ] Yes (fill out Blk. 11b) [X] No (NA Blks. 11b, 11c, 11d)	NA	NA Cog. Engineer Signature & Date	NA Cog. Engineer Signature & Date

12. Description of Change

Validation procedures have been updated to reflect those required to meet current requirements and techiques approved by HASM.

13a. Justification (mark one)							
	Criteria Change	[x]	Design Improvement	[ ]	Environmental	[ ]	
As-Found	[ ]	Facilitate Const.	[ ]	Const. Error/Omission	[ ]	Design Error/Omission	[ ]

13b. Justification Details

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14. Distribution (include name, MSIN, and no. of copies)

See Distribution Sheet for ECN 602830

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ENGINEERING CHANGE NOTICE

1. ECN (use no. from pg. 1): 602830

15. Design Verification Required	16. Cost Impact				17. Schedule Impact (days)	
	ENGINEERING		CONSTRUCTION			
[] Yes	Additional	[] $	Additional	[] $	Improvement	[]
[X] No	Savings	[] $	Savings	[] $	Delay	[]

18. Change Impact Review: Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 12. Enter the affected document number in Block 19.

SDD/DD	[]	Seismic/Stress Analysis	[]	Tank Calibration Manual	[]
Functional Design Criteria	[]	Stress/Design Report	[]	Health Physics Procedure	[]
Operating Specification	[]	Interface Control Drawing	[]	Spares Multiple Unit Listing	[]
Criticality Specification	[]	Calibration Procedure	[]	Test Procedures/Specification	[]
Conceptual Design Report	[]	Installation Procedure	[]	Component Index	[]
Equipment Spec.	[]	Maintenance Procedure	[]	ASME Coded Item	[]
Const. Spec.	[]	Engineering Procedure	[]	Human Factor Consideration	[]
Procurement Spec.	[]	Operating Instruction	[]	Computer Software	[]
Vendor Information	[]	Operating Procedure	[]	Electric Circuit Schedule	[]
OM Manual	[]	Operational Safety Requirement	[]	ICRS Procedure	[]
FSAR/SAR	[]	IEFD Drawing	[]	Process Control Manual/Plan	[]
Safety Equipment List	[]	Cell Arrangement Drawing	[]	Process Flow Chart	[]
Radiation Work Permit	[]	Essential Material Specification	[]	Purchase Requisition	[]
Environmental Impact Statement	[]	Fac. Proc. Samp. Schedule	[]		[]
Environmental Report	[]	Inspection Plan	[]		[]
Environmental Permit	[]	Inventory Adjustment Request	[]		[]

19. Other Affected Documents: (NOTE: Documents listed below will not be revised by this ECN.) Signatures below indicate that the signing organization has been notified of other affected documents listed below.

Document Number/Revision	Document Number/Revision	Document Number Revision

Validation Procurement Documents

20. Approvals

	Signature	Date		Signature	Date
OPERATIONS AND ENGINEERING			ARCHITECT-ENGINEER		
Cog Engineer Karl N. Pool	Karl N. Pool	10/26/93	PE		
Cog. Mgr. Cleve Mooers	C Mooers	10/26/93	QA		
QA Dana Farwick	D Farwick	10/4/93	Safety		
Safety			Design		
Security			Environ.		
Environ. Mel Adams	M A Adams	11/10/93	Other		
Projects/Programs					
Tank Waste Remediation System Al Noonan	AM Noonan	11/12/93			
Facilities Operations			DEPARTMENT OF ENERGY		
Restoration & Remediation			Signature or Letter No.		
Operations & Support Services			DOE-RL Donna Wanek	Donna Wanek	12/15/93
IRM			ADDITIONAL		
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SDLA Jerry Paetel	Jerry Paetel	11/1/93			

Date Received: 12/16/93

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Reference: WHC-CM-3-4

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- [ ] Technical Report
- [ ] Thesis or Dissertation
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Author/Requestor (Printed/Signature): Karl N. Pool — Date: 10/26/93

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Responsible Manager (Printed/Signature): G. C. Mooers — Date: 10/29/93

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7. Abstract

This document provides procedures to Westinghouse Hanford Company (WHC) staff and subcontractors tasked with the validation of radiochemistry analytical data produced as the result of Hanford Site environmental investigations. Data validation procedures for chemical analytical data, though not included in this document, are specified in the WHC document "Data Validation Procedures for Chemical Analyses" (Pool 1993). This document shall be included in all procurement packages for radiochemical data validation services.

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1.0 INTRODUCTION

This document provides procedures for use by Westinghouse Hanford Company (WHC) staff and subcontractors tasked with the validation of radiochemistry analytical data produced as the result of Hanford Site environmental investigations. Data validation procedures for chemical analytical data, though not included in this document, are specified in the WHC document "Data Validation Procedures for Chemical Analyses" (WHC-SD-EN-SPP-002). This procedures document shall be included in all procurement packages for radiochemical data validation services.

Data validation is the process of reviewing a body of analytical data to determine if it meets the criteria defined in this document to assure that the data are adequate for their intended use. The process of data validation consists of:

- Editing and correcting of reported results
- Verifying compliance with quality assurance (QA) requirements
- Checking quality control (QC) values against defined limits
- Applying qualifiers to analytical results for the purpose of defining the limitations in use of the reviewed data

Data validation shall be conducted by trained chemists or other scientists using this document in conjunction with applicable project specific work plans, field sampling plans, QA project plans (QAPjPs), analytical method references, and laboratory statements of work (SOW).

The result of data validation will be accomplished by completion of narrative reports, checklists, summary forms and electronic data deliverables established in this document. The completed narrative reports, checklists, summary forms, and electronic deliverables will document whether the analytical data are acceptable for their intended use.

2.0 GENERAL REQUIREMENTS

WHC staff and subcontractors may be tasked with the responsibility for data validation of radiochemical data packages. WHC Hanford Analytical Services Management (HASM) is responsible for the assignment of data validation responsibilities on a task basis and will assign a project coordinator for each task. The WHC project coordinator will provide the data validation subcontractor current copies of the applicable project specific work plans, field sampling plans, descriptions of work (DOW), QAPjPs, laboratory SOW, laboratory QAPjPs, and laboratory standard operating procedures (SOP), specifying the radionuclides of interest, reference analytical methods, required detection limits (RDL) and goals for analytical precision, accuracy, representativeness, completeness and comparability.

Sections 4.0 through 12.0 provide the necessary procedures for the performance of specific categories of data validation.

Five activity levels of data validation are specified in this document, they are:

- Level A (minimum requirements for all data) - This level of data validation will include the verification of required deliverables, requested versus reported analyses, evaluation and qualification of results based on analytical holding times. No other validation, transcription or calculation checks will be performed.

- Level B - This level of data validation will include level A validation, verification of transcription errors (if not already performed prior to receipt of the data package by the validation subcontractor) and evaluation and qualification of results additionally on method blank results. No calculation checks will be performed.

- Level C - This level of data validation will include levels A and B validation and additionally, the evaluation and qualification of sample results based on matrix spikes, laboratory control samples, laboratory duplicates and chemical and tracer recoveries. No other validation or calculation checks will be performed.

- Level D - This level of data validation will include levels A, B and C validation and the additional evaluation and qualification of results based on initial and continuing instrument calibrations and other QC checks that are performed as required by the particular analytical method such as quench monitoring and counting instrument resolution checks. Calculation checks of both sample and QC results will be performed at a frequency of 20% or at least one sample and one complete QC sample series (standard, blank, LCS, spike, chemical and/or tracer recovery) will be recalculated, whichever is greater. QC samples will be defined as initial and continuing calibration standards, method blanks, spike samples, chemical and tracer recovery, duplicates and laboratory control samples.

- Level E - This level of data validation will include all the requirements of levels A, B, C, and D validation and will be considered the highest level of validation intended for the verification of site clean-up actions. Calculation checks will be conducted on 100% of the sample and QC results.

During data validation, the data validator will be required to complete validation checklists for documentation and reporting purposes. Appendix A provides copies of the data validation checklists.

The data validators shall complete several tasks on a sample delivery group basis during validation of laboratory data packages. A sample delivery group shall be defined as a group of samples (usually 20 or fewer) reported within the same laboratory data package. Figure 2-1 shows the overall flow of data packages during the data validation process, while Figure 2-2 provides a detailed flow chart outlining the technical validation tasks to be performed. These tasks are summarized as follows:

- Receipt of the analytical data package from HASM and performance of records management activities which shall include the making of duplicate copies of the sample concentration report forms.

- Verifying that all requested analyses have been reported as required by the sample analysis request and sample chain of custody documentation.

- Verify reported results against the raw data and validate the quality of the data package according to the procedures described in Sections 4.0 through 12.0 and document the review using the checklists provided.

- Qualify the sample results as directed by the validation requirements on a duplicate copy of the sample concentration reports. All annotations must be made in black ink and must be initialed and dated by the data validator. Data that are rejected at any point during the validation shall be eliminated from further validation.

- Annotated data qualifiers, for all parameters, shall be neatly printed in the right handside column of the report form and shall be clearly visible as to what radiochemical parameter the qualifier is applied to.

- Check result and QC calculations at the frequency based on the activity level specified as described above.

- Following completion of validation of a single data package, prepare a data validation package summarizing the data acceptability and which includes copies of the marked-up photocopy of the original laboratory sample concentration reports and any supporting validation documentation. This validation package shall be returned to HASM along with the original data package.

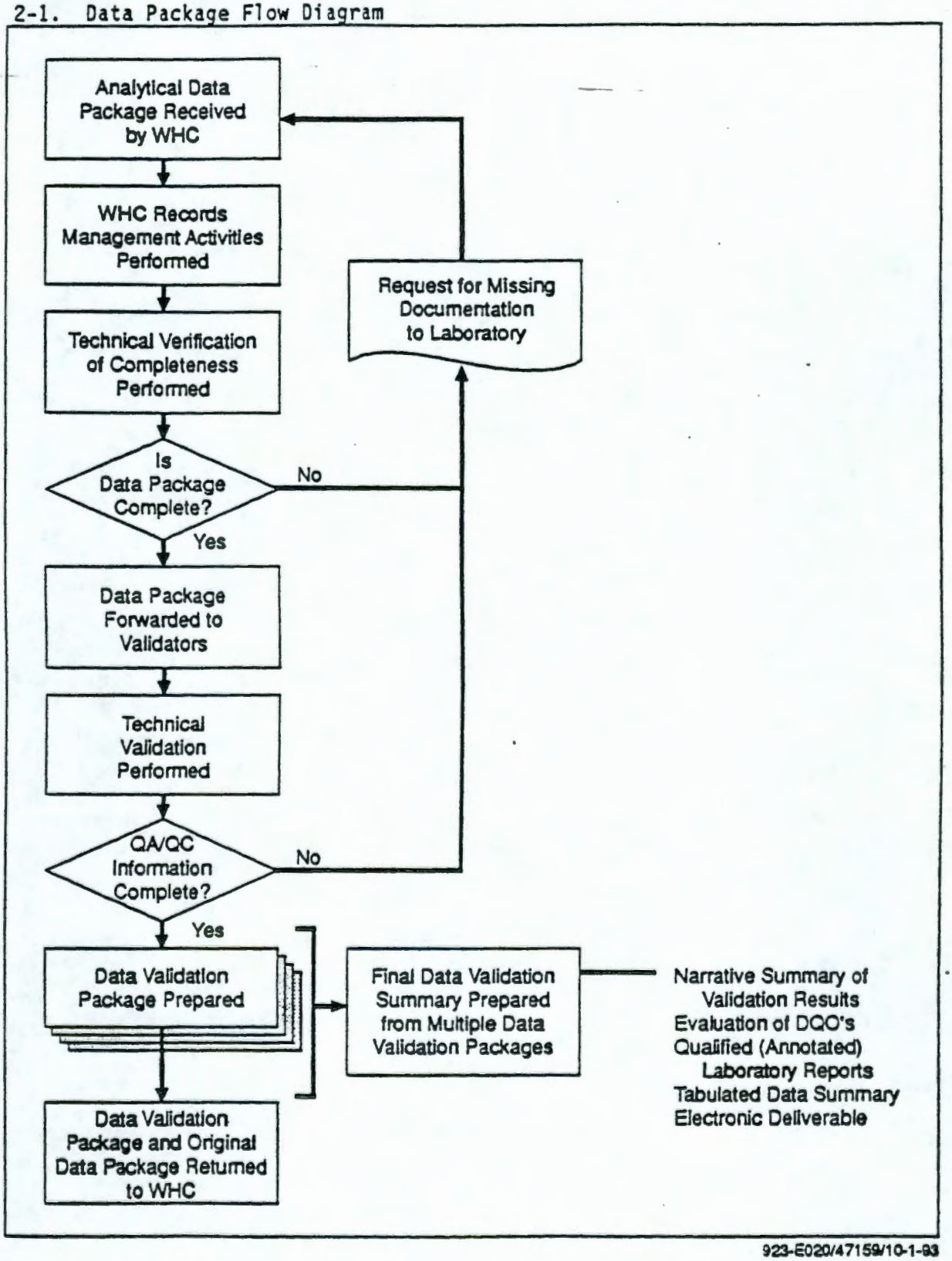
- Data validation packages shall be completed within 21 calendar days after receipt of the complete data package from HASM.

- Following completion of validation on a series of data packages a summary of all data validation performed will be compiled into a final data validation summary report in accordance with the guidelines specified in Section 13.0.

- Transmittal of electronic data deliverables in accordance with the requirements specified in Section 13.0

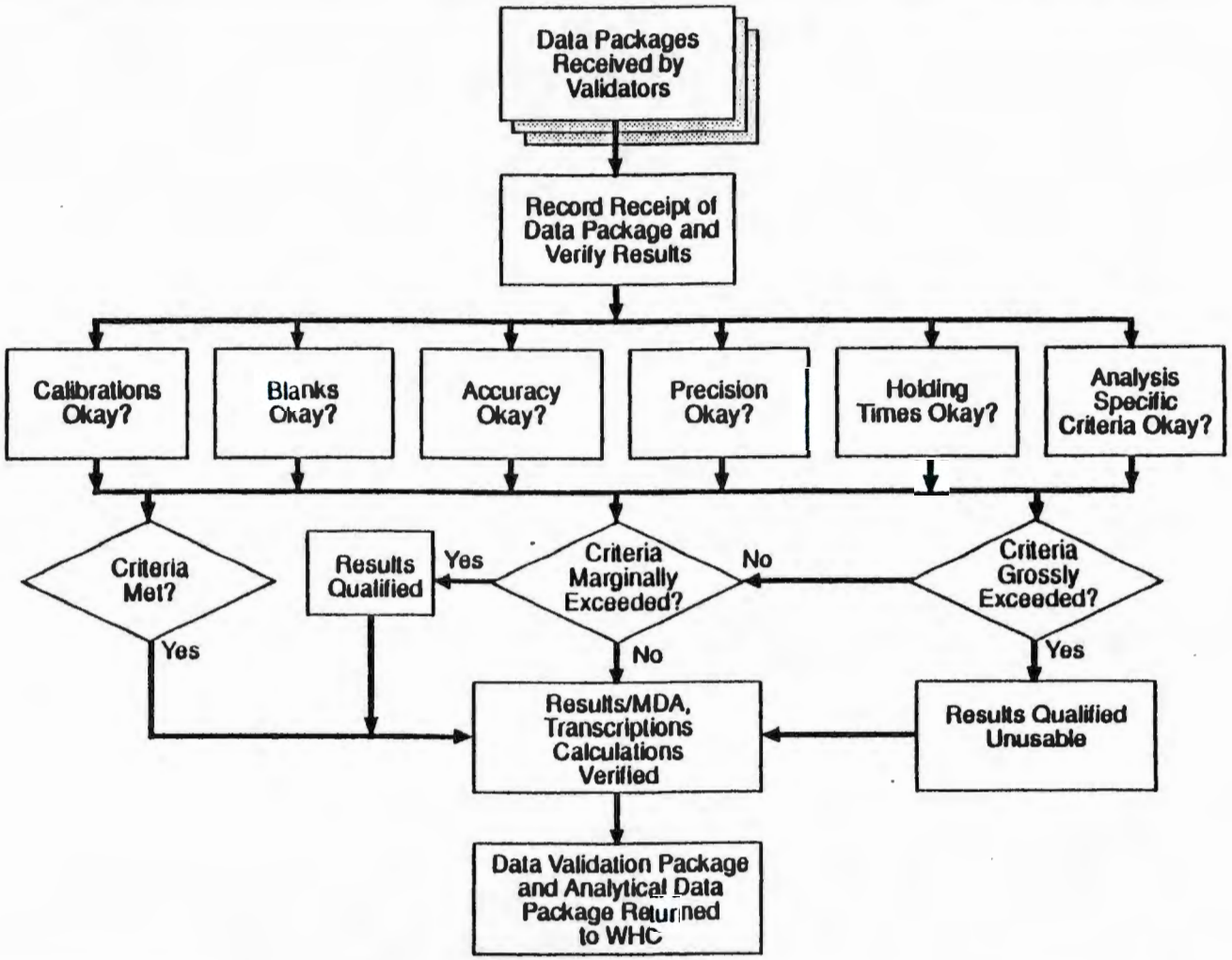
- Final data summary reports shall be submitted within 21 calendar days following receipt by the validation subcontractor of the last complete data package for the task.

2-1. Data Package Flow Diagram



923-E020/47159/10-1-93

2-2. Data Validation Flow Diagram



923-E020047160/10-1-93

3.0 SPECIFIC REQUIREMENTS

This section presents specific requirements that apply to all data validation activities specified in this document.

3.1 RECORDS MANAGEMENT

The subcontractor(s) shall have a records management and document control program established that meets the following requirements. Upon receipt of the data package by the data validator, the date of receipt shall be recorded and a duplicate record of the sample concentration reports shall be made for use during the data validation and for transmittal in data validation packages and final reports. The data package will be maintained in original as-received condition for transmittal at the conclusion of data validation activities.

3.2 DATA PACKAGE COMPLETENESS

Prior to receipt of data packages by the subcontractor, the data package shall have been verified for completeness (missing forms, data sheets, etc.) by the HASM technical verification subcontractor. Therefore, verification of data package completeness by the validation subcontractor is not required. The observation of omitted deliverables or technical data necessary to complete the validation shall prompt the validation subcontractor to contact HASM with a request for the missing information by facsimile. When requesting missing information from HASM the following items must be supplied by the validation subcontractor:

- HASM Data package tracking number of the data package for which information is being requested.
- WHC Hanford Environmental Information System (HEIS) sample number of the sample for which information is being requested. If more than one sample is involved, each sample identification number must be supplied.
- The type of analysis for which the information is being requested. If more than one type of analysis is involved, each analysis must be identified.
- Analysis specific information must also be supplied such as instrument and detector identification, date of analysis, and page number of the data package where the missing information was identified.

3.3 PERSONNEL REQUIREMENTS

Contractors shall have an organization with defined responsibilities and defined technical capabilities for individuals responsible for successful completion of data validation reviews. The contractor shall

designate personnel to conduct the following tasks for all WHC data review contracts or task orders.

- Data Validators — Data validators shall be responsible for conduct of data validation, and reporting activities as assigned by the subcontractor project manager. Data validators shall have a minimum of a bachelor's degree in chemistry or related physical or life science with a minimum of 40 h of training in data validation under the supervision of a senior data validator.

- Senior Validator — Senior validators shall provide oversight and sign-off on all work performed by the data validators. This senior validator shall then submit the qualified data to the project manager. Senior validators shall have a minimum of a bachelor's degree in chemistry, physical, or life science plus 1 yr of radiochemistry data validation experience with the Environmental Protection Agency (EPA) or other contractors in data review and validation.

- Project Manager — Project managers shall be responsible for overall management and direction of the data validation, and reporting activities and assignment of responsibilities to validation personnel. Project managers shall have a minimum of a bachelor's degree in chemistry, physical or life science with a minimum of 3 yr experience data validation or laboratory analysis but preferred experience in both areas and including at least 1 yr of supervisory experience.

- Document Custodian — Document custodians shall be responsible for records management activities associated with data validation as assigned by the project manager. Document custodians shall have a minimum of 1 yr experience in records management.

- Data Manager — Data managers shall be responsible for data entry of validated results into electronic databases for transmittal in accordance with Section 13.0. Data managers shall have a minimum of 40 h of training and 1 yr experience in computer-based data entry and data management.

- Quality Assurance Officer — QA officers shall be responsible for verification of compliance with the data validation procedures embodied in this document. QA officers shall have a minimum of a bachelor's degree in a technical field and 1 yr experience in laboratory analyses or data validation, and shall have sufficient independence from project management, cost and schedule concerns to permit the identification and resolution of quality problems related to the validation process.

3.4 TECHNICAL VALIDATION REQUIREMENTS

Data validation contractors shall conduct the data validation using the procedures and criteria specified in sections 4.0 through 12.0.

3.4.1 Radiochemistry Data Validation Checklists

The data validation checklist for radiochemistry is contained in Appendix A and cover each validation section contained in Sections 4 through 12. All validation activities shall be documented using this checklist.

3.4.2 Data Validation Qualifiers

Qualifiers to be applied as a result of the validation are summarized below. All qualifiers applied to the sample concentration report forms are to be written on the forms by crossing out the original qualifier and writing the validation qualifier in the right hand margin. Each form must be initialed and dated by the responsible data validator.

U - The constituent was analyzed for, but was not detected. The value reported is the minimum detectable activity (MDA) corrected for sample dilution and moisture content by the laboratory. The data should be considered usable for decision making purposes.

UJ - The constituent was analyzed for and was not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the MDA. The data should be considered usable for decision making purposes.

J - Indicates the constituent was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. The data should be considered usable for decision making purposes.

UR - Indicates the constituent was analyzed for and not detected; however, due to an identified quality control deficiency the data should be considered unusable for decision making purposes.

R - Indicates the constituent was analyzed for and detected; however, due to an identified quality control deficiency the data should be considered unusable for decision making purposes.

4.0 GROSS ALPHA/BETA DATA VALIDATION REQUIREMENTS

This section presents specific data validation requirements for gross alpha and gross beta analyzed by gas proportional counters. The analysis consists of the evaporation and drying of a quantity of water sample or an aliquot of a digested solid sample onto a planchet. The sample is then counted by gas proportional counting for both alpha and beta emitting radioactivity.

4.1 CASE NARRATIVE

A case narrative should be included with each data package and should be reviewed for information specific to the reported data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

4.2 INSTRUMENT CALIBRATION

The objective of instrument calibration is to ensure that detectors used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed.

4.2.1 Initial Calibration

Initial calibration data demonstrate that the instrument used for sample analysis was capable of producing acceptable quantitative results prior to sample analyses. The initial calibration data is submitted with the data package or as a separate supplement.

Verify that the counting system used for sample analysis meets the following criteria:

- Each counting system used for sample analysis was efficiency calibrated within one year prior to sample analysis. If not calibrated within one year, then the continuing calibration requirements listed below must be met.

- Calibration standards are National Institute of Standards and Technology (NIST)-traceable and certificates are provided.

- Self-absorption curves were prepared for each counting system from a series of planchets with weights ranging from 0 to approximately 150 mg, and the counting error for net counts is less than 5% for each planchet. For example, standards containing americium-241 or plutonium-239 as alpha emitting radionuclides and cesium-137 or strontium-90 as beta emitting radionuclides may be used for the calibration standards.

- Efficiency of each detector at 0% solids must be at least 20% or greater for either gross alpha or gross beta.

Verify the laboratory has provided the necessary raw data, as described below, or that the data are available in the most recent calibration supplement provided by WHC.

- Detector identification, self absorption curves, and raw data including calibration date, planchet weights, raw and background counts for each counting system used for sample analysis.

- NIST traceability certificates for all calibration standards including a dilution log documenting the preparation including date of preparation, radionuclide, lot numbers, and dpm values.

After evaluation is complete, qualify all associated sample results as follows:

- If initial detector efficiency at 0% solids is <20%, then reject all associated sample results (R for detects, UR for non-detects).

- If the detector specific raw calibration data is unavailable and cannot be provided by the laboratory, reject all associated sample results (R for detects, UR for non-detects).

4.2.2 Continuing Calibration

Verify that the continuing instrument calibration meets the following criteria:

- Acceptable continuing calibration checks have been performed at least once per analytical run, sample batch, or daily, whichever is more frequent.

- Check standards are NIST-traceable and certificates are provided.

Evaluate continuing calibration results by verifying the laboratory has provided the following information:

- Results of continuing calibration checks including detector identification, dates, source and background counts, count duration, and control limits.

- NIST traceability certificates including a dilution log documenting the preparation including date of preparation, radionuclide, lot numbers, and dpm values.

After evaluation is complete, qualify all associated sample results as follows:

- If any calibration check (before or after sample analysis) is out of the control limits, qualify the associated sample results as unusable (R for detects, UR for non-detects).

4.2.3 Background Counts

Background counts are random counts that are detected by the instrument from other sources besides the samples being analyzed and are used to calculate the sample activity value.

Verify that the instrument background counts meets the following criteria:

- Performed within one week prior to sample analysis.
- Performed on each detector used for sample analysis.
- Within the laboratory control limits.

Evaluate the background data and qualify associated sample results as follows:

- If background counts were not performed within one week prior to the associated sample analysis, qualify all associated sample results as unusable (R for detects, UR for non-detects).
- If background counts were not performed on the detector used for the sample analysis, qualify all associated sample results as unusable (R for detects, UR for non-detects).
- If the background counts are not within the laboratory control limits, however, the sample results are significantly greater (> 40%) than the background counts, qualify the associated sample results as estimated (J for detects, UJ for non-detects).
- If the background counts are not within the laboratory control limits, and the sample results are low enough that the change in background will significantly affect the result (by > 10%), qualify the associated sample results as unusable (R for detects, UR for non-detects).

4.3 BLANKS

Blank sample results are reviewed to assess the extent of contamination introduced through sampling, sample preparation and analysis. Summarize all blank results in the validation narrative.

4.3.1 Laboratory Blanks

Verify that the laboratory blank analysis meets the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per sample delivery group (SDG).
- Prepared at the same time and analyzed with the samples using the same procedure.
- Results are less than or equal to the MDA and RDL.

Evaluate the laboratory blank data by verifying the following:

- The laboratory has provided the raw data including detector identification, count duration, gross and background counts.
- Results and MDA values were accurately reported.

After evaluation is complete, qualify all associated sample results as follows:

- If a laboratory blank was not performed with the associated samples, qualify all associated sample results as estimated (J for detects, UJ for non-detects).
- If positive results are present in the laboratory blanks, qualify all associated positive sample results that are less than five times the highest blank concentration as estimated (J). For negative sample results, elevate the result to the MDA and qualify as undetected (U).
- If the sample result is >MDA and >5 times the associated highest blank result, no qualification is necessary.

4.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually referred to as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

4.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the laboratory control samples, blank spikes, or performance audit samples.

4.4.1 Laboratory Control or Blank Spike Samples

Verify that the laboratory control sample (LCS) or blank spike sample (BSS) meets the following criteria and that the laboratory provided the following information:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- LCS or BSS activity is between 5 and 30 times the associated RDL value.
- The actual LCS concentration or the spike concentration and the amount of spike added for the BSS were provided by the laboratory.
- Results are within the limits of 70% to 130% recovery

After evaluation, qualify associated sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

If neither an LCS nor BSS sample was performed with the associated analytical batch, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

4.4.2 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample, and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

4.5 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

4.5.1 Laboratory Duplicates

Verify that the duplicate samples meet the following criteria and that the laboratory provided the following information:

- The laboratory has conducted a duplicate analysis sample at a frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.
- The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.
- The relative percent difference (RPD) must be less than or equal to 20% for water samples (≤ 35% for soils) if the sample concentration is greater than five times the RDL.
- For sample results less than five times the RDL, the range between the primary and duplicate sample results must be less than or equal to the RDL for water samples (≤2x RDL for soils).

Check all calculations, and after evaluation is complete, qualify associated sample results as follows:

Original Sample Result	RPD or Range	Qualification
No duplicate analyzed	Not applicable	J for detects, UJ for non-detects
> 5x RDL	> 20% for waters > 35% for soils	J for detects, UJ for non-detects
< 5x RDL	> RDL for waters >2x RDL for soils	J for detects, UJ for non-detects

4.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils.

When one or both the results are < 5x RDL, the limit should be expressed as the difference between result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

4.5.3 Field Split Sample

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

4.6 HOLDING TIMES

Verify that all samples were preserved properly (water samples should be preserved with nitric acid, HNO_3, preferably in the field or otherwise immediately upon receipt at the laboratory) and analyzed within 180 days. If holding times are exceeded qualify sample results as follows:

- If water samples were not preserved and samples were not analyzed within 180 days, reject all associated results (R for detects, UR for non-detects).
- If holding times are > 180 days but ≤ 360 days qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days reject all associated results (R for detects, UR for non-detects).

4.7 SAMPLE RESULT QUANTITATION AND MINIMUM DETECTABLE ACTIVITIES

Verify that the laboratory has reported the following information for each sample:

- WHC sample identification,
- laboratory sample identification,
- detector identification and efficiency,

- gross alpha and beta sample and background counts,
- count duration,
- planchet weights,
- sample volumes,
- alpha and beta crosstalk factors (if applicable),
- calculated sample activities, uncertainties and MDA values,
- required detection limits.

Check calculations according to the specified data validation level and correct sample results as necessary. Note in the validation report if MDA values do not meet the RDL values. If sample results and MDA values cannot be verified, qualify the results as estimated (J for detects, UJ for non-detects).

4.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

5.0 STRONTIUM-90 DATA VALIDATION REQUIREMENTS

This section presents specific data validation requirements for strontium-90 and other beta-emitting radionuclide analyses such as technetium-99. The analysis is performed by the addition of a chemical carrier followed by separation and purification of the carrier along with the target radiochemical analyte. The chemical preparation is mounted on a planchet and counted by gas proportional counting.

5.1 CASE NARRATIVE

A case narrative will be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

5.2 INSTRUMENT CALIBRATION

The objective of instrument calibration is to ensure that detectors used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed.

5.2.1 Initial Calibration

Initial calibration data demonstrate that the instrument used for sample analysis was capable of producing acceptable quantitative results prior to sample analyses. The initial calibration data is submitted with the data package or as a separate supplement.

Verify that the counting system used for sample analysis meets the following criteria:

- Each counting system used for sample analysis was efficiency calibrated within one year prior to sample analysis. If not calibrated within one year, then the continuing calibration requirements listed below must be met.

- Calibration standards are NIST-traceable and certificates are provided.

- Self-absorption curves were prepared for each detector used with planchet weights ranging from 0 to approximately 150 mg and the counting error for net counts is less than 5% for each planchet. The calibration reference standard should be prepared from a traceable solution of yttrium-90, strontium-90, strontium-89, technetium-99 or cesium-137.

- Efficiency of each detector at 0% solids is at least 20% or greater and the method of determining empirical efficiencies for non-calibrated isotopes is described.

Verify the laboratory has provided the following raw data or that the data are available in the most recent calibration supplement:

- Detector identification, self absorption curves, and raw data including calibration date, planchet weights, raw and background counts for each counting system used for sample analysis.

- NIST traceability certificates for all calibration standards including a dilution log documenting the preparation including date of preparation, radionuclide, lot numbers, and dpm values.

After evaluation is complete, qualify all associated sample results as follows:

- If initial detector efficiency at 0% solids is <20%, then reject all associated sample results (R for detects, UR for non-detects).

- If the detector specific calibration raw data is unavailable and cannot be provided by the laboratory, reject all associated sample results (R for detects, UR for non-detects).

5.2.2 Continuing Calibration

Verify that the continuing instrument calibration met the following criteria:

- Acceptable continuing calibration checks have been performed at least once per analytical run, sample batch, or daily, whichever is more frequent.

- Check standards are NIST-traceable and certificates are provided.

Evaluate continuing calibration results by verifying the laboratory has provided the following information:

- Results of continuing calibration checks including detector identification, dates, source and background counts, count duration and control limits.

- NIST traceability certificates including a dilution log documenting the preparation including date of preparation, radionuclide, lot numbers, and dpm values.

After evaluation is complete, qualify all associated sample results as follows:

- If any calibration check (before or after sample analysis) is out of the control limits qualify the associated sample results as unusable (R for detects, UR for non-detects).

5.2.3 Background Counts

Background counts are random counts that are detected by the instrument from other sources besides the samples being analyzed and are used to calculate the sample activity value.

Verify that the instrument background counts met the following criteria:

- Performed within one week prior to sample analysis.
- Performed on each detector used for sample analysis.
- Within the laboratory control limits

Evaluate the background data and qualify associated sample results as follows:

- If background counts were not performed within one week prior to the associated sample analysis, qualify all associated sample results as unusable (R for detects, UR for non-detects).
- If background counts were not performed on the detector used for the sample analysis, qualify all associated sample results as unusable (R for detects, UR for non-detects).
- If the background counts are not within the laboratory control limits, however, the sample results are significantly greater (> 40%) than the background counts, qualify the associated sample results as estimated (J for detects, UJ for non-detects).
- If the background counts are not within the laboratory control limits, and the sample results are low enough that the change in background will significantly affect the result (by > 10%), qualify the associated sample results as unusable (R for detects, UR for non-detects).

5.3 BLANKS

The blank data results are reviewed to assess the extent of contamination introduced through sampling, sample preparation, and analysis. Summarize all blank results in the validation narrative.

5.3.1 Laboratory Blanks

Verify that the laboratory blank analysis met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed with the samples using the same procedure.
- Results are less than or equal to the MDA and RDL.

Evaluate the laboratory blank data by verifying the laboratory has provided the following information:

- Raw data including detector identification, count duration, and gross and background counts were provided by the laboratory.
- Results and MDA values were accurately reported.

After evaluation is complete, qualify all associated sample results as follows:

- If a laboratory blank was not performed with the associated samples, qualify all associated sample results as estimated (J for detects, UJ for non-detects).
- If positive results are present in the laboratory blanks, qualify all associated positive sample results that are less than five times the highest blank concentration estimated (J). For negative sample results elevate the result to the MDA and qualify as undetected (U).
- If the sample result is >MDA and >5 times the associated highest blank result, no qualification is necessary.

5.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually identified as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

5.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the matrix spike, laboratory control or blank spike, and performance audit sample recovery values.

5.4.1 Laboratory Control or Blank Spike Samples

Verify that laboratory control (LCS) or blank spike (BSS) samples met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- LCS or BSS activity is between 5 and 30 times the associated RDL value and results are within the limits of 70% to 130%.

Qualify associated sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

If neither an LCS nor BSS sample was performed with the associated analytical batch, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

5.4.2 Chemical Recovery Factors

The evaluation of chemical recovery factors provides an assessment of chemical separation process affected by the laboratory procedure, sample matrix, or interference. The chemical recovery factor is used to calculate the sample activity, uncertainty, and the MDA.

Verify the following regarding chemical recoveries:

- Chemical carrier was added to every sample analyzed including blanks and all quality control samples (duplicates, blanks, matrix spike samples, LCS, BSS, etc.).
- The amount and concentration of the chemical carrier added to each sample and recovered from each sample was reported along with a dilution log documenting the traceability.
- The chemical recovery factor is within the limit of 30% to 105%.

Qualify results as follows:

Percent Chemical Recovery	Qualification
30% to 105%	None, acceptable for use
10% to 29%	Estimated (J for detects, UJ for non-detects)
> 105%	Estimated (J for detects, no qualification required for non-detects)
< 10%	Unusable (R for detects, UR for non-detects)

5.4.3 Matrix Spike Samples

The matrix spike sample analysis optionally provides information about the effect of each sample matrix on the preparation and measurement methodology. If laboratory control or blank spike samples are not analyzed, the requirements for matrix spikes must be met.

Verify that matrix spike analyses were conducted as follows:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG if a carrier was not used in the analysis.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- Percent recovery is within the limits of 60% to 140% unless sample concentration exceeds the spike concentration by a factor of 4 or more.

Qualify associated sample results as follows:

MS %R	Sample Activity	Qualification
60% - 140%	≥ MDA < MDA	None Required None Required
≥ 10% and < 60%	≥ MDA < MDA	J UJ
> 140%	≥ MDA < MDA	J None Required
< 10%	≥ MDA < MDA	R UR

If a matrix spike sample was not performed, with the associated analytical batch, but was required, qualify associated sample results as estimated (J for detects, UJ for non-detects).

5.4.4 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample, and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

5.5 PRECISION

Review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

5.5.1 Laboratory Duplicates

Verify that the duplicate samples met the following criteria and that the laboratory provided the following information:

- The laboratory has conducted a duplicate analysis sample at a frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.
- The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.
- The relative percent difference (RPD) must be less than or equal to 20% for water samples (≤ 35% for soils) if the sample concentration is greater than five times the RDL.
- For sample results less than five times the RDL, the range between the primary and duplicate sample results must be less than the RDL for water samples (<2x RDL for soils).

After evaluation is complete, qualify associated sample results as follows:

Original Sample Result	RPD or range	Qualification
No duplicate analyzed	Not applicable	J for detects, UJ for non-detects
>5x RDL	>20% for waters >35% for soils	J for detects, UJ for non-detects
<5x RDL	>RDL for waters >2x RDL for soils	J for detects, UJ for non-detects

5.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils. When one or both the results are < 5x RDL, the limit should be expressed as the difference between result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

5.5.3 Field Split Sample

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

5.6 HOLDING TIMES

Verify that all samples were preserved properly (water samples should be preserved with nitric acid, HNO_3, preferably in the field or otherwise immediately upon receipt at the laboratory) and analyzed within 180 days. If holding times are exceeded, qualify sample results as follows:

- If water samples were not preserved and samples were not analyzed within 180 days, reject all associated results (R for detects, UR for non-detects).
- If holding times are > 180 days but ≤ 360 days (for preserved water samples), qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days reject all associated results (R for detects, UR for non-detects).

5.7 SAMPLE RESULT QUANTITATION AND MINIMUM DETECTABLE ACTIVITIES

Verify that the laboratory has reported the following information for each sample:

- WHC sample identification,
- laboratory sample identification,
- detector identifications and efficiencies,
- start date and time of each analysis step (i.e. ingrowth, separation and counting),
- sample and background counts,
- count duration,
- planchet weights,
- sample volumes,
- chemical recovery factors including amounts added to each sample, duplicate, blank, LCS and matrix spike and amounts recovered,
- ingrowth and decay factors for all analyses,
- calculated sample activities, uncertainties and MDA values,
- required detection limits.

Check calculations according to the specified data validation level and correct sample results as necessary. Note in the validation report if MDA values do not meet the RDL values. If sample results and MDA values cannot be verified qualify the results as estimated (J for detects, UJ for non-detects).

5.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

6.0 ALPHA SPECTROMETRY DATA VALIDATION REQUIREMENTS

This section presents specific data validation requirements for plutonium, uranium and other radioisotopes analyzed by alpha spectrometry. Samples are analyzed by the addition of a suitable tracer followed by chemical precipitation, purification, and electrodeposition on a planchet or mounting of the purified precipitate on a planchet. The sample is then counted in an alpha spectrometer and the target radioisotopes are determined by the comparison to the recovered tracer.

6.1 CASE NARRATIVE

A case narrative will be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

6.2 INSTRUMENT CALIBRATION

The objective of instrument calibration is to ensure that detectors used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed.

6.2.1 Initial Calibration

Initial calibration data demonstrate that the instrument used for sample analysis was capable of producing acceptable quantitative results prior to sample analyses. The initial calibration data is submitted with the data package or as a separate supplement. However, for alpha spectrometry analysis, requirements for initial calibration are not mandatory if the laboratory meets the continuing calibration and LCS performance criteria.

Verify that the initial instrument calibration, if required according to the specifications above, meets the following criteria.

- Each detector used was calibrated within one year prior to the sample analysis.
- Efficiency values are provided for each detector and were measured within one year of the sample analysis.
- Calibration standards are NIST-traceable and certificates are provided.
- Detectors were calibrated in the energy range of approximately 4 to 6 MeV with a maximum range of 2 to 8 MeV, and the standards were counted in order to accumulate a minimum of 2000 counts for each target radioisotope.

Evaluate the initial calibration data by verifying the laboratory has provided the following raw data or that the data are available in the most recent calibration supplement:

- Energy calibration curves and all associated raw data including detector identification, calibration date, count duration, peak counts, and efficiency values.
- NIST traceability certificates for all calibration standards including a dilution log documenting the preparation dates, lot numbers, DPM activities, expiration dates, and amount of standards used.

After evaluation is complete, qualify all associated sample results as follows:

- If the detector was not calibrated across the range of interest, then reject all associated sample results (R for detects, UR for non-detects).
- If the detector specific calibration raw data, including the efficiency values, is unavailable and cannot be provided by the laboratory, reject all associated sample results (R for detects, UR for non-detects).

6.2.2 Continuing Calibration

Verify that the continuing instrument calibration met the following criteria or that the information has been provided in the most recent calibration supplement:

- Energy calibration and detector efficiencies were checked at least weekly prior to sample analysis and for each detector used for sample analysis.
- Detector efficiencies determined from the weekly checks are within the laboratory control limits.
- Tracer preparation, activity, dilution log, and traceability is submitted with each data package.
- The activity, NIST certificates and dilution log, is submitted for each check standard used for weekly checks.

After evaluation is complete, qualify all associated sample results as follows:

- If a particular detector efficiency check is outside the laboratory control limits or less than 20%, qualify associated sample results as unusable (R for detects, UR for non-detects).
- If calibration checks have not been performed weekly, qualify associated sample results as estimated (J for detects, UJ for non-detects).

- If the calibration check information is unavailable and cannot be provided by the laboratory qualify all associated sample results as unusable (R for detects, UR for non-detects).

6.2.3 Background Counts

Background counts are random counts that are detected by the instrument from other sources besides the samples being analyzed and are used to determine the net sample counts in order to calculate the sample specific activity.

Verify that the instrument background counts met the following criteria:

- Performed at least monthly on each detector used for sample analysis for each region of interest (ROI) monitored for the particular analysis.

- Within the laboratory control limits.

Evaluate the background data and qualify associated sample results as follows:

- If background counts were not performed monthly and prior to sample analysis, qualify all associated sample results as estimated (J for detects, UJ for non-detects).

- If background counts were not performed on the detector used for the sample analysis, qualify all associated sample results as unusable (R for detects, UR for non-detects).

- If the background counts are not within the laboratory control limits, however, the sample results are significantly greater (> 40%) than the background counts, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

- If the background counts are not within the laboratory control limits, and the sample results are low enough that the change in background will significantly affect the result (by >10%), qualify the associated sample results as unusable (R for detects, UR for non-detects).

6.3 BLANKS

Blank sample results are reviewed to assess the extent of contamination introduced through sampling, sample preparation, and analysis. Summarize all blank results in the validation narrative.

6.3.1 Laboratory Blanks

Verify that the laboratory blank analysis met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed with the samples using the same procedure, aliquot size, and counting time.
- Results are less than or equal to the MDA and RDL.

Evaluate the laboratory blank data by verifying the following:

- Raw data including detector identification, count duration, gross and background counts were provided by the laboratory.
- Results and MDA values were accurately reported.

After evaluation is complete, qualify all associated sample results as follows:

- If a laboratory blank was not performed with the associated samples, qualify all associated sample results as estimated (J for detects, UJ for non-detects).
- If positive results are present in the laboratory blank, qualify positive sample results which are less than the MDA as undetected (U).
- If positive results are present in the laboratory blanks, qualify all associated positive sample results that are greater than the MDA and less than five times the highest blank concentration as estimated (J). For negative sample results, elevate the result to the MDA and qualify as undetected (U).
- If the sample result is >MDA and >5 times the associated highest blank result, no qualification is necessary.

6.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually identified as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

6.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the evaluation of tracer recovery, laboratory control samples or blank spike samples and performance audit samples.

6.4.1 Laboratory Control or Blank Spike Samples

Verify that LCS or BSS samples met the following criteria and that the laboratory provided the following information:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- LCS or BSS activity is between 5 and 30 times the associated RDL value.
- Provided the actual LCS concentration or the spike concentration and the amount of spike added for the BSS.
- Results are within the limits of 70% to 130% recovery.

After evaluation, qualify associated sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

If neither an LCS nor BSS sample was performed with the associated analytical batch, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

6.4.2 Tracer Recovery

Tracer recovery provides an evaluation as to the effectiveness of the sample preparation process used to isolate the radioisotope of interest. The tracer recovery factor is used to calculate the sample activity, uncertainty and MDA.

Review the calculation sheets and raw data and verify the laboratory has provided the following information and met the following criteria:

- Each sample was spiked with an appropriate tracer as applicable for the analytical method.
- Tracer activity and NIST-traceability and a dilution log was provided.

- Raw data was provided showing the amount of tracer added to each sample and the gross counts per minute of the tracer.
- Tracer recovery is within the limits of 20% to 105%.

After evaluation qualify associated sample results according to the following table:

Tracer Recovery	Qualification
20% to 105%	None, acceptable for use
5% to 19%	Estimated, (J for detects, UJ for non-detects)
105% to 115%	Detects as estimated, (J), no qualification required for non-detects)
<20%	Unusable, (R for detects, UR for non-detects)
> 115%	Detects as unusable, (R), no qualification required for non-detects

6.4.3 Matrix Spike Samples

The matrix spike sample analysis optionally provides information about the effect of each sample matrix on the preparation and measurement methodology. If laboratory control or blank spike samples are not analyzed, the requirements for matrix spikes must be met.

Verify that matrix spike analyses were conducted as follows:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG if a tracer was not used in the sample analysis.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- Percent recovery is within the limits of 60% to 140% unless sample concentration exceeds the spike concentration by a factor of 4 or more.

Qualify associated sample results as follows:

MS %R	Sample Activity	Qualification
60% - 140%	≥ MDA < MDA	None Required None Required
≥ 10% and < 60%	≥ MDA < MDA	J UJ

MS %R	Sample Activity	Qualification
> 140%	≥ MDA < MDA	J None Required
< 10%	≥ MDA < MDA	R UR

If a matrix spike sample was not performed with the associated analytical batch, but was required, qualify associated sample results as estimated (J for detects, UJ for non-detects).

6.4.4 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

6.5 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

6.5.1 Laboratory Duplicates

Verify that the duplicate samples met the following criteria and that the laboratory provided the following information:

- The laboratory has conducted a duplicate analysis sample at a frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.
- The duplicate analysis was prepared and analyzed in the same batch, using the same procedure, as the associated samples.
- The relative percent difference (RPD) must be less than 20% for water samples (<35% for soils) if the sample concentration is greater than five times the RDL.
- For sample results less than five times the RDL, the difference between the primary and duplicate sample results must be less than the RDL for water samples (<2x RDL for soils).

After evaluation is complete, qualify associated sample results as follows:

Original Sample Result	RPD or range	Qualification
No duplicate analyzed	not applicable	J for detects, UJ for non-detects
>5x RDL	>20% for waters or >35% for soils	J for detects, UJ for non-detects
<5x RDL	>RDL for waters or >2x RDL for soils	J for detects, UJ for non-detects

6.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils. When one or both the results are < 5x RDL, the limit should be expressed as the difference between result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

6.5.3 Field Split Sample

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

6.6 HOLDING TIMES

Verify that all samples were preserved properly (water samples should be preserved with nitric acid, HNO_3, preferably in the field or otherwise immediately upon receipt at the laboratory) and analyzed within 180 days. If holding times are exceeded qualify sample results as follows:

- If water samples were not preserved and samples were not analyzed within 180 days, reject all associated results (R for detects, UR for non-detects).
- If holding times are >180 days but ≤360 days qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days reject all associated results (R for detects, UR for non-detects).

6.7 SAMPLE RESULT QUANTITATION AND MINIMUM DETECTABLE ACTIVITIES

Verify that the laboratory has reported the following information for each sample:

- WHC sample identification,
- laboratory sample identification,
- detector identification and efficiency,
- gross sample counts, regions of interest (ROI), and channel by channel counts,
- gross tracer counts, ROI, and channel by channel counts,
- background counts (monthly),
- count duration,
- sample spectra showing peak integration parameters and full width at half maximum (FWHM) values,
- planchet weights (if in the case of precipitated mounts rather than electroplated mounts),
- sample volumes,
- calculated sample activities, uncertainties and MDA values,
- required detection limits.

Check calculations according to the specified data validation level and correct sample results as necessary. Note in the validation report if MDA values do not meet the RDL values. Qualify results as follows:

- If sample results and MDA values cannot be verified qualify the results as estimated (J for detects, UJ for non-detects).
- If peak integration results indicate FWHM values of >100 for either the target radioisotopes or tracer, reject all associated results (R for detects, UR for non-detects) since this indicates inadequate resolution.

6.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

7.0 GAMMA SPECTROSCOPY DATA VALIDATION REQUIREMENTS

This section presents specific data validation requirements for radionuclides analyzed by gamma spectroscopy. Samples are normally analyzed by direct count on a lithium-drifted germanium diode detector since higher resolution and greater sensitivity can be obtained for counting gamma emitting radionuclides. Samples are mounted in a particular geometry such as a Marinelli beaker or low-density polyetythlene bottle, placed in the detector well and counted for a time duration adequate to achieve an acceptable MDA. Since the results are sensitive to the particular sample geometry, calibration of the instrument must be conducted for each geometry used.

7.1 CASE NARRATIVE

A case narrative should be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

7.2 INSTRUMENT CALIBRATION

The objective of instrument calibration is to ensure that detectors used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed.

7.2.1 Initial Calibration

Initial calibration data demonstrate that the instrument used for sample analysis was capable of producing acceptable quantitative results prior to sample analyses. The initial calibration data may be submitted with the data package or as a separate supplement.

Verify that the initial instrument calibration meets the following criteria:

- Each detector and geometry used for sample analysis was initially calibrated within one year prior to sample analysis.
- Each detector was calibrated within the energy range of approximately 0 to 2,000 KeV.
- Detector resolution at the cobalt-60 photopeak of 1332 KeV was at least 3.0 KeV FWHM (5 channels) or less.
- Initial calibration standards are NIST-traceable and certificates and a dilution log are provided.

- Coefficients of the energy calibration and efficiencies for each target radionuclide are provided with each data package.

Qualify sample results as follows:

- If the samples were analyzed on a geometry with no documented initial calibration, qualify the results as unusable (R for detects, UR for non-detects).

- If the detector specific calibration raw data is unavailable and cannot be provided by the laboratory, reject all associated sample results (R for detects, UR for non-detects).

7.2.2 Continuing Calibration

Verify that the continuing instrument calibration met the following criteria:

- Calibration check standards have been counted at least weekly in each detector used for sample analysis, and the results have been submitted with the data package or are at least available in the most recent calibration supplement.

- Calibration check system gain, FWHM, and efficiency is within the laboratory control limits.

- Check standards are NIST-traceable and certificates and a dilution log have been provided with the data package or are available in the most recent calibration supplement.

Qualify results as follows:

- If the calibration check standards have not been counted at least monthly on the same geometries used for sample analysis, qualify sample results as estimated (J for detects, UJ for non-detects).

- If the most recent calibration check on the sample specific geometry exceeds any of the laboratory control limits for system gain, FWHM, or efficiency; qualify associated sample results as unusable (R for detects, UR for non-detects).

7.2.3 Background Counts

Instrument background counts are counts that are detected by the instrument from other radioactive sources besides the associated samples being analyzed. They are subtracted from sample counts in order to calculate the sample specific activities, uncertainties, and MDA values.

Verify the instrument background counts meet the following criteria:

- Instrument backgrounds are counted prior to sample analysis on a monthly basis for a duration similar to the sample counts.

- Background counts, including a spectral summary, are provided for each detector and geometry used for sample analysis in each data package.

After evaluation is complete, qualify all associated sample results as follows:

- If the instrument background counts were not performed monthly, qualify all associated sample results as estimated (J for detects, UJ for non-detects).

- If the sample specific instrument background data is not provided and is unavailable from the laboratory, qualify all associated sample results as unusable (R for detects, UR for non-detects).

- If the background counts are not within the laboratory control limits, however the sample results are significantly greater (> 40%) than the background counts, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

- If the background counts are not within the laboratory control limits, and the sample results are low enough that the change in background will significantly affect the result (by > 10%), qualify the associated sample results as unusable (R for detects, UR for non-detects).

7.3 BLANKS

The blank data results are reviewed to assess the extent of contamination introduced through sampling, sample preparation, and analysis. Summarize all blank results in the validation narrative.

7.3.1 Laboratory Blanks

Verify that the laboratory blank analysis met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.

- Analyzed using a similar aliquot size, counted in the same geometry and count time as the samples.

- Results are less than or equal to the MDA and RDL.

Evaluate the laboratory blank data by verifying the following:

- Raw data including detector identification, count duration, geometry, and gross and background counts were provided by the laboratory.

- Results and MDA values were accurately reported.

After evaluation is complete, qualify all associated sample results as follows:

- If a laboratory blank was not performed with the associated samples, counted in the same geometry and for the same duration as the samples, qualify all associated sample results as estimated (J for detects, UJ for non-detects).
- If positive results are present in the laboratory blank, qualify positive sample results which are less than the MDA as undetected (U).
- If positive results are present in the laboratory blanks, qualify all associated positive sample results that are greater than the MDA and less than five times the highest blank concentration as estimated (J). For negative sample results, elevate the result to the MDA and qualify as undetected (U).
- If the sample result is >MDA and >5 times the associated highest blank result, no qualification is necessary.

7.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually identified as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

7.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the laboratory control or blank spike, and performance audit sample recovery values.

7.4.1 Laboratory Control or Blank Spike Samples

Verify that an LCS or BSS was analyzed and met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- LCS or BSS activity does not exceed 1000 pCi total activity or is not greater than 5 to 50 times the total sample activities.
- LCS or BSS was analyzed in the same geometry, count duration, and aliquot size as the samples.
- The actual LCS concentration or spike concentration including traceability and a dilution log were reported.

- Results are within the limits of 70% to 130% recovery.

After evaluation, qualify associated sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

- If neither an LCS or BSS were performed with the SDG or were performed in a different geometry than the samples, qualify associated results as estimated (J for detects, UJ for non-detects).

- If the LCS or BSS concentration and percent recovery cannot be verified and the information is unavailable from the laboratory, reject all associated sample results (R for detects, UR for non-detects).

7.4.2 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample, and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source, and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

7.5 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

7.5.1 Laboratory Duplicates

Verify that the duplicate samples met the following criteria and that the laboratory provided the following information:

- The laboratory has conducted a duplicate analysis sample at a frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.
- The duplicate analysis was prepared and analyzed at the same time, using the same geometry, aliquot size and count duration as the samples.
- The relative percent difference (RPD) must be less than 20% for water samples (<35% for soils) if the sample concentration is greater than five times the RDL.
- For sample results less than five times the RDL, the difference between the primary and duplicate sample results must be less than the RDL for water samples (<2x RDL for soils).

After evaluation is complete, qualify associated sample results as follows:

Original Sample Result	RPD or range	Qualification
No duplicate analyzed	not applicable	J for detects, UJ for non-detects
>5x RDL	>20% for waters and >35% for soils	J for detects, UJ for non-detects
<5x RDL	>RDL for waters and >2x RDL for soils	J for detects, UJ for non-detects

7.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils. When one or both the results are < 5x RDL, the limit should be expressed as the difference between result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

7.5.3 Field Split Sample

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

7.6 HOLDING TIMES

Verify that all samples were preserved properly (water samples should be preserved with nitric acid, HNO_3, preferably in the field or otherwise immediately upon receipt at the laboratory) and analyzed within 180 days. If holding times are exceeded, qualify sample results as follows:

- If water samples were not preserved and samples were not analyzed within 180 days, reject all associated results (R for detects, UR for non-detects).
- If holding times are >180 days but ≤360 days, qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days, reject all associated results (R for detects, UR for non-detects).

7.7 SAMPLE RESULT QUANTITATION AND MINIMUM DETECTABLE ACTIVITIES

Verify that the laboratory has reported the following information for each sample:

- WHC sample identification,
- laboratory sample identifications, batch numbers, geometry numbers,
- date and time of sample, blank, LCS, BSS, and duplicate analyses,
- detector identification, geometry, energy, efficiency, and FWHM coefficients,
- sample and background net counts,
- printouts of regions of interest (ROI) and channel by channel counts or spectra,
- count duration,

- sample volumes,
- calculated sample activities, uncertainties, and MDA values,
- required detection limits.

Check calculations according to the specified data validation level and correct sample results as necessary. Note in the validation report if MDA values do not meet the RDL values. If sample results and MDA values cannot be verified, qualify the results as estimated (J for detects, UJ for non-detects).

7.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

8.0 LIQUID SCINTILLATION DATA VALIDATION REQUIREMENTS

This section presents specific data validation requirements for the analysis of water samples for tritium or carbon-14 by liquid scintillation counting. Tritium samples are distilled to remove gamma activity interferences and the sample is mixed with a scintillant and placed in a suitable counting vial. The sample is counted in a liquid scintillation spectrometer and is counted on a batch basis along with a standard or group of standards. Tritium background water samples are prepared at the same time as the samples since exposure of samples, blanks, and standards to daylight or fluorescent lighting will cause biased results. Therefore, all tritium samples must be dark-adapted for at least 30 minutes to two hours.

8.1 CASE NARRATIVE

A case narrative should be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

8.2 INSTRUMENT CALIBRATION

The objective of instrument calibration is to ensure that detectors used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed.

8.2.1 Initial Calibration

Initial calibration data demonstrate that the instrument used for sample analysis was capable of producing acceptable quantitative results prior to sample analyses. The initial calibration data may be submitted with the data package or as a separate supplement.

Verify that the initial instrument calibration met the following criteria:

- Each counting system used was factory calibrated at installation and after any maintenance or repair and a certificate of calibration is provided in the data package or the most recent calibration supplement.

- Calibration standards used are NIST-traceable and certificates and a dilution log are provided.

Evaluate the initial calibration data by verifying the laboratory has provided the following raw data or that the data is available in the most recent calibration supplement:

- Factory calibration results and certificates

- NIST traceability certificates for all calibration standards including a dilution log documenting the preparation dates, lot numbers, DPM activities, expiration dates, and amount of standards used.

After evaluation is complete, qualify all associated sample results as follows:

- If the counting system has not been factory calibrated before the analysis of the samples, qualify all associated sample results as unusable (R for detects, UR for non-detects).

- If the calibration data is unavailable and cannot be provided by the laboratory, qualify all associated sample results as unusable (R for detects, UR for non-detects).

8.2.2 Continuing Calibration and Quench Monitoring

Verify that the continuing instrument calibration met the following criteria:

- Calibration checks are performed with each analytical run, sample batch, or daily, whichever is more frequent. The results and control limits shall be reported with each SDG.

- Calibration checks are within the laboratory control limits.

- Calibration checks are performed at the same aliquot size as the samples.

- Efficiency check standards are NIST-traceable and certificates and a dilution log are provided.

- Quench monitoring values are reported with each data package and are within the laboratory control limits.

After evaluation is complete, qualify the sample results as follows:

- If the calibration check was not performed with the sample batch, qualify the sample results as estimated (J for detects, UJ for non-detects).

- If the calibration check was not performed at the same aliquot size as the samples qualify the sample results as estimated (J for detects, UJ for non-detects).

- If a calibration check is out of the control limits qualify the associated sample results as unusable (R for detects, UR for non-detects).

- If the quench monitoring values are out of the laboratory control limits qualify the associated sample results as unusable (R for detects, UR for non-detects).

- If the calibration check was not reported and the data are not available from the laboratory, qualify the sample results as unusable (R for detects, UR for non-detects).

8.2.3 Background Counts

Verify that instrument background checks meet the following criteria:

- Background checks were performed, on each counting system used for sample analysis and were performed with each analytical run, sample batch, or daily, whichever is more frequent. The results and control limits are reported with each SDG.
- The most recent background check was within the laboratory control limits.

Qualify sample results as follows:

- If the background checks have not been performed weekly, qualify sample results as estimated (J for detects, UJ for non-detects).
- If the most recent background count is not within the laboratory control limits, however, the sample results are significantly greater (> 40%) than the background count, qualify the associated sample results as estimated (J for detects, UJ for non-detects).
- If the most recent background count is not within the laboratory control limits, and the sample results are low enough that the change in background will significantly affect the result (by > 10%), qualify the associated sample results as unusable (R for detects, UR for non-detects).

8.3 BLANKS

The blank data results are reviewed to assess the extent of contamination introduced through sampling, sample preparation, and analysis. Summarize all blank results in the validation narrative.

8.3.1 Laboratory Blanks

Verify that the laboratory blank analysis met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Laboratory blanks have been prepared, distilled and analyzed using the same procedure and aliquot size as the samples.
- Results are reported along with the laboratory control limits.

Evaluate the laboratory blank data by verifying the following:

- Raw data including counting system identification, count duration, and gross and background counts were provided by the laboratory.

- Results and MDA values were accurately reported.

After evaluation is complete, qualify all associated sample results as follows:

- If a laboratory blank was not performed with the associated samples, qualify all associated sample results as estimated (J for detects, UJ for non-detects).

- If positive results are present in the laboratory blank, qualify positive sample results which are less than the MDA as undetected (U).

- If positive results are present in the laboratory blanks, qualify all associated positive sample results that are greater than or equal to the MDA and less than five times the highest blank concentration as estimated (J). For negative sample results, elevate the result to the MDA and qualify as undetected (U).

- If the sample result is >MDA and >5 times the associated highest blank result, no qualification is necessary.

8.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually identified as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

8.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the laboratory control or blank spike, and performance audit sample recovery values.

8.4.1 Laboratory Control or Blank Spike Samples

Verify that LCS or BSS samples met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.

- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.

- LCS or BSS activity is less than 100 times the RDL.

- LCS or BSS traceability, concentration and dilution log is provided.
- Results are within the limits of 70% to 130% recovery.

After evaluation qualify associated sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

If neither an LCS nor BSS sample was performed with the associated analytical batch, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

8.4.2 Matrix Spike Samples

The matrix spike sample analysis provides information about the effect of each sample matrix on the preparation and measurement methodology.

Verify that matrix spike analyses were conducted as follows:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- Percent recovery is within the limits of 60% to 140% unless sample concentration exceeds the spike concentration by a factor of 4 or more.

Qualify associated sample results as follows:

MS %R	Sample Activity	Qualification
60% - 140%	≥ MDA < MDA	None Required None Required
≥ 10% and < 60%	≥ MDA < MDA	J UJ
> 140%	≥ MDA < MDA	J None Required

MS %R	Sample Activity	Qualification
< 10%	≥ MDA < MDA	R UR

If a matrix spike sample was not performed with the associated analytical batch, qualify associated sample results as estimated (J for detects, UJ for non-detects).

8.4.3 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample, and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source, and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

8.5 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

8.5.1 Laboratory Duplicates

Verify that the duplicate samples met the following criteria:

- Conducted at a frequency of 10% (two in twenty samples) or at least once per SDG.
- Prepared and analyzed using the same aliquot size as the samples.
- The relative percent difference (RPD) is less than 20% for sample concentrations greater than five times the RDL.
- For sample results less than five times the RDL, the difference between the primary and duplicate sample results must be less than the RDL.

Qualify associated sample results as follows:

Original Sample Result	RPD or range	Qualification
No duplicate analyzed	not applicable	J for detects, UJ for non-detects
>5x RDL	>20%	J for detects, UJ for non-detects
<5x RDL	>RDL	J for detects, UJ for non-detects

If a duplicate was not performed, qualify all sample results as estimated (J for detects, UJ for non-detects).

8.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils. When one or both the results are < 5x RDL, the limit should be expressed as the difference between result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

8.5.3 Field Split Sample

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

8.6 HOLDING TIMES AND SAMPLE PREPARATION

8.6.1 Holding Times

Verify that all samples were analyzed within 180 days. Water samples to be analyzed for tritium should not be prepared with nitric acid. If holding times are exceeded qualify sample results as follows:

- If water samples were not analyzed within 180 days, qualify all associated results as unusable (R for detects, UR for non-detects).
- If holding times are >180 days but ≤360 days, qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days, qualify all associated results as unusable (R for detects, UR for non-detects).

8.6.2 Sample Preparation

Evaluate the preparation data by verifying the laboratory has met the following criteria:

- All tritium field and QC samples were distilled prior to analysis.
- Samples were analyzed within seven days after distillation.

Qualify sample results as follows:

- If a copy of the distillation log was not submitted with the data package and cannot be provided by the laboratory, qualify all associated sample results estimated (J for detects, UJ for non-detects).
- If the samples were not analyzed within seven days of distillation, qualify the results as unusable (R for detects, UR for non-detects)

8.7 SAMPLE RESULT QUANTITATION AND MINIMUM DETECTABLE ACTIVITIES

Review the calculation sheets, raw data, and sample report forms and verify the laboratory has provided the following information:

- WHC sample identification,
- laboratory sample identification,
- detector identification and efficiency,
- sample and background counts and count durations,
- date and time of all sample analyses,
- sample volumes,

- calculated sample activities, uncertainties, and MDA values,
- required detection limits.

Check calculations according to the specified data validation level and correct sample results as necessary. Note in the validation report if MDA values do not meet the RDL values. If sample results and MDA values cannot be verified, qualify the results as estimated (J for detects, UJ for non-detects).

8.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

9.0 RADIUM-226 BY RADON EMANATION VALIDATION REQUIREMENTS

This section presents specific data validation requirements for the analysis of water samples for radium-226 by radon emanation technique. Samples are prepared by co-precipitation of the radium with a barium carrier. The precipitate is dissolved in a basic-EDTA solution, placed in a bubbler and the radon-222 decay product purged out of solution with inert gas. The sample contained within the bubbler is sealed and placed in the dark for 10 to 15 days to allow for the ingrowth of radon gas. After ingrowth, the radon gas is purged into a scintillation cell (Lucas Cell) whose interior surfaces are coated with a suitable phosphor. The radon gas contained within the cell is allowed to equilibrate and the sample is counted on a photomultiplier tube.

9.1 CASE NARRATIVE

A case narrative will be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

9.2 INSTRUMENT CALIBRATION

The objective of instrument calibration is to ensure that detectors used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed.

9.2.1 Initial Calibration

Initial calibration in radium-226 analysis is a twofold process, first the photomultiplier voltage and gain settings must be optimized for the detection of radium-226 then the lucas cell efficiencies must be determined for each cell used. The initial calibration data may be submitted with the data package or as a separate supplement.

9.2.1.1 Detector Plateau Determination. Verify that detector plateaus and instrument settings meet the following criteria:

- Detector plateau settings were determined at least annually and within one year prior to the analysis of samples by the analysis of at least two standards at different concentrations in which at least 10,000 counts for radium-226 were accumulated for the high standard.

- Calibrations were performed using NIST-traceable radium-226 standards and the certificates and dilution log were provided with the data package or in the most recent calibration supplement.

Qualify sample results as follows:

- If the detector calibration was not performed within one year and prior to the analysis of samples, qualify all results as unusable (R for detects, UR for non-detects).

- If calibration standard traceability is unavailable and cannot be provided by the laboratory, qualify sample results as unusable (R for detects, UR for non-detects).

9.2.1.2 Cell Factors. Verify that all cells used for sample and QC analysis meet the following criteria:

- Cell factors have been determined at least annually and prior to the analysis of samples.

- Cell factors have been reported using NIST-traceable standards with certificates and a dilution log.

- All raw data documenting the cell factor determination and calculation is reported with the data package or in the most recent calibration supplement.

- Background counts on cells used for sample and QC analysis have been determined at least weekly and prior to sample analysis.

Qualify sample results as follows:

- If cell factors have not been determined at least annually and prior to the analysis of samples, qualify all sample results as estimated (J for detects, UJ for non-detects).

- If the cell factor raw data and standards traceability information is unavailable and cannot be provided by the laboratory, qualify all sample results as unusable (R for detects, UR for non-detects).

- If background counts have not been determined at least weekly prior to the analysis of samples, qualify all sample results as estimated (J for detects, UJ for non-detects).

9.2.2 Continuing Calibration

Continuing calibration checks are performed periodically in order to demonstrate the instrument reliability and therefore to determine if the instrument is capable of producing acceptable quantitative results at the time the associated samples are analyzed.

Verify that the continuing instrument calibration met the following criteria:

- A calibration check was performed on a daily basis or at the beginning of each analytical run using a low concentration standard (less than 10 times the RDL).
- Calibration check raw data and control limits are reported with the data package or in the most recent calibration supplement.
- Check standards are NIST-traceable and certificates and a dilution log are provided are provided.

Qualify sample results as follows:

- If a calibration check was not performed, qualify associated sample results as estimated (J for detects, UJ for non-detects).
- If the calibration check is outside the laboratory control limits, qualify associated sample results as unusable (R for detects, UR for non-detects).
- If the calibration check raw and traceability date is unavailable and cannot be provided by the laboratory, qualify all sample results as unusable (R for detects, UR for non-detects).

9.3 BLANKS

The blank data results are reviewed to assess the extent of contamination introduced through sampling, sample preparation and analysis. Summarize all blank results in the validation narrative.

9.3.1 Laboratory Blanks

Verify that the laboratory blank analysis met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared in the same analytical batch using similar sample volumes and the same procedure as the samples.
- Reported with all raw data including detector and cell identification and efficiency, gross counts, background counts, count duration, date and time of analysis.
- Results are less than or equal to the MDA and RDL.

After evaluation is complete, qualify all associated sample results as follows:

- If a laboratory blank was not performed with the associated samples, qualify all associated sample results as estimated (J for detects, UJ for non-detects).
- If positive results are present in the laboratory blank, qualify positive sample results which are less than the MDA as undetected (U).
- If positive results are present in the laboratory blanks, qualify all associated positive sample results that are greater than or equal to the MDA and less than five times the highest blank concentration as estimated (J). For negative sample results, elevate the result to the MDA and qualify as undetected (U).
- If the sample result is >MDA and >5 times the associated highest blank result, no qualification is necessary.

9.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually identified as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

9.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the chemical recovery, laboratory control or blank spike, and performance audit sample recovery values.

9.4.1 Laboratory Control or Blank Spike Samples

The laboratory control (LCS) or blank spike (BSS) sample analysis provides information concerning the effectiveness and accuracy of the laboratory method.

Verify that the LCS or BSS samples met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- LCS or BSS activity is less than 100 times the RDL.

- LCS or BSS traceability, concentration, and dilution log are provided.
- Verify that the results are within the limits of 70% to 130% recovery.

After evaluation is complete, qualify all associated sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

If neither an LCS nor BSS sample was performed with the associated analytical batch, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

9.4.2 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample, and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source, and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

9.5 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

9.5.1 Laboratory Duplicates

Verify that the duplicate samples met the following criteria and that the laboratory provided the following information:

- The laboratory has conducted a duplicate analysis sample at a frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.
- The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.
- The relative percent difference (RPD) must be less than 20% for water samples (<35% for soils) if the sample concentration is greater than five times the RDL.
- For sample results less than five times the RDL, the difference between the primary and duplicate sample results must be less than the RDL for water samples (<2x RDL for soils).

After evaluation is complete, qualify associated sample results as follows:

Original Sample Result	RPD or range	Qualification
No duplicate analyzed	not applicable	J for detects, UJ for non-detects
>5x RDL	>20% for waters and >35% for soils	J for detects, UJ for non-detects
<5x RDL	>RDL for waters and >2x RDL for soils	J for detects, UJ for non-detects

If no duplicate was analyzed, qualify all sample results as estimated (J for detects, UJ for non-detects).

9.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils. When one or both the results are < 5x RDL, the limit should be expressed as the difference between result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be

discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

9.5.3 Field Split Sample

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

9.6 HOLDING TIMES

Verify that all samples were preserved properly (water samples should be preserved with nitric acid, HNO_3, preferably in the field or otherwise immediately upon receipt at the laboratory) and analyzed within 180 days. If holding times are exceeded, qualify sample results as follows:

- If water samples were not preserved and samples were not analyzed within 180 days, reject all associated results (R for detects, UR for non-detects).
- If holding times are >180 days but ≤360 days, qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days, qualify all associated results as unusable (R for detects, UR for non-detects).

9.7 SAMPLE RESULT QUANTITATION AND MINIMUM DETECTABLE ACTIVITIES

Verify that the laboratory has reported the following information for each sample:

- WHC sample identification,
- laboratory sample identification,
- detector identification,
- cell identification and efficiency,
- cell background counts and count duration,
- sample gross counts and count duration,

- sample volumes,
- barium recovery values (if applicable),
- calculated sample activities, uncertainties, and MDA values,
- required detection limits.

Check calculations according to the specified data validation level and correct sample results as necessary. Note in the validation report if MDA values do not meet the RDL values. If sample results and MDA values cannot be verified, qualify the results as estimated (J for detects, UJ for non-detects).

9.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

10.0 FLUOROMETRIC URANIUM DATA VALIDATION REQUIREMENTS

This section presents data validation requirements for the analysis of water samples for uranium by fluorometry. Water samples are analyzed by the evaporation of a suitable aliquot into a platinum dish. The residue is then fused into a pellet at high temperature with a fluoride-carbonate flux. The fluorescence of the uranium-fluoride is measured with a fluorometer. Sample concentrations are determined by comparison to an external calibration curve prepared from uranium standard solutions prepared and analyzed identically as the samples.

10.1 CASE NARRATIVE

A case narrative should be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

10.2 INSTRUMENT CALIBRATION

The objective of instrument calibration is to ensure that detectors used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed.

10.2.1 Initial Calibration

Initial calibration data demonstrate that the instrument used for sample analysis was capable of producing acceptable quantitative results prior to sample analyses. The initial calibration data may be submitted with the data package or as a separate supplement.

Verify that the laboratory calibrated the fluorometer on the day of sample analysis using a blank and at least three standards covering the range of the sample concentrations with a calibration coefficient of at least 0.995 or better.

Qualify sample results as follows:

- If the fluorometer was not acceptably calibrated on the same day of and prior to sample analysis, qualify all sample results as unusable (R for detects, UR for non-detects).
- If the calibration coefficient is <0.995 but >0.9 qualify all sample results as estimated (J for detects, UJ for non-detects).

10.3 BLANKS

Blank sample results are reviewed to assess the extent of contamination introduced through sampling, sample preparation and analysis. Summarize all blank results in the validation narrative.

10.3.1 Laboratory Blanks

Verify that the laboratory blank analysis met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed with the samples using the same procedure.
- Results are less than or equal to the MDA and RDL.

Evaluate the laboratory blank data by verifying the following:

- Raw data including detector identification, count duration, and gross and background counts were provided by the laboratory.
- Results and MDA values were accurately reported.

After evaluation is complete, qualify all associated sample results as follows:

- If a laboratory blank was not performed with the associated samples, qualify all associated sample results as estimated (J for detects, UJ for non-detects).
- If positive results are present in the laboratory blanks, qualify all associated positive sample results that are greater than or equal to the MDA and less than five times the highest blank concentration as estimated (J). For negative sample results, elevate the result to the MDA and qualify as undetected (U).
- If the sample result is >MDA and >5 times the associated highest blank result, no qualification is necessary.

10.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually identified as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

10.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the laboratory control or blank spike, and performance audit sample recovery values.

10.4.1 Laboratory Control or Blank Spike Samples

Verify the laboratory has met the following criteria for LCS or BSS analysis:

- At least one LCS or BSS was performed with the SDG.
- The LCS or BSS true concentration, traceability, and dilution log was reported.
- The LCS or BSS concentration is less than 100 times the RDL.
- The LCS or BSS was analyzed using the same procedure and sample volume as the samples.
- The LCS or BSS recovery is within the limits of 70% to 130%.

After evaluation, qualify sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

- If an LCS or BSS was not performed with the samples, qualify all sample results as estimated (J for detects, UJ for non-detects).
- If the LCS or BSS concentrations cannot be verified or the traceability information is unavailable and cannot be provided by the laboratory, qualify all sample results as estimated (J for detects, UJ for non-detects).

10.4.2 Matrix Spike Samples

The matrix spike sample analysis provides information about the effect of each sample matrix on the preparation and measurement methodology.

Verify that matrix spike analyses were conducted as follows:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- Percent recovery is within the limits of 60% to 140% unless sample concentration exceeds the spike concentration by a factor of 4 or more.

Qualify associated sample results as follows:

MS %R	Sample Activity	Qualification
60% - 140%	≥ MDA < MDA	None Required None Required
≥ 10% and < 60%	≥ MDA < MDA	J UJ
> 140%	≥ MDA < MDA	J None Required
< 10%	≥ MDA < MDA	R UR

If a matrix spike sample was not performed with the associated analytical batch, qualify associated sample results as estimated (J for detects, UJ for non-detects)

10.4.3 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample, and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source, and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

10.5 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

10.5.1 Laboratory Duplicates

Verify that the duplicate samples met the following criteria and that the laboratory provided the following information:

- The laboratory has conducted a duplicate analysis sample at a frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.
- The duplicate analysis was prepared and analyzed in the same batch, using the same procedure, as the associated samples.
- The relative percent difference (RPD) must be less than 20% for water samples (<35% for soils) if the sample concentration is greater than five times the RDL.
- For sample results less than five times the RDL, the difference between the primary and duplicate sample results must be less than the RDL for water samples (<2x RDL for soils).

Check all calculations and after evaluation is complete, qualify associated sample results as follows:

Original Sample Result	RPD or range	Qualification
No duplicate analyzed	not applicable	J for detects, UJ for non-detects
>5x RDL	>20% for waters and >35% for soils	J for detects, UJ for non-detects
<5x RDL	>RDL for waters and >2x RDL for soils	J for detects, UJ for non-detects

10.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils. When one or both the results are < 5x RDL, the limit should be expressed as the difference between result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

10.5.3 Field Splits

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

10.6 HOLDING TIMES

Verify that all samples were preserved properly (water samples should be preserved to a pH < 2 with nitric acid) and analyzed within 180 days. If holding times are exceeded, qualify sample results as follows:

- If water samples were not preserved and samples were not analyzed within 180 days, qualify all associated results as unusable (R for detects, UR for non-detects).
- If holding times are >180 days but ≤360 days, qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days, qualify all associated results as unusable (R for detects, UR for non-detects).

10.7 SAMPLE RESULT QUANTITATION AND MINIMUM DETECTABLE ACTIVITIES

Verify that the laboratory has reported the following information for each sample:

- WHC sample identification,
- laboratory sample identification,
- sample and QC results and raw data,
- fluorometer calibration and raw data.

Check calculations as specified in the appropriate validation level and correct sample results as necessary. Note in the validation report if sample quantitation limit values do not meet the RDL values. If the sample results and detection limits cannot be verified, qualify the associated results as estimated (J for detects, UJ for non-detects).

10.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

11.0 PHOSPHORIMETRIC URANIUM DATA VALIDATION REQUIREMENTS

This section presents data validation requirements for the analysis of water samples for uranium by laser phosphorimetry. Samples are analyzed by mixing with a specified phosphate reagent, then analyzed on a laser fluorescence instrument. The uranium ions present in solution fluoresce when excited by a tuned ultraviolet laser, and their intensity is measured by a photomultiplier tube. Sample concentration is determined by an internal standard technique.

11.1 CASE NARRATIVE

A case narrative should be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

11.2 INSTRUMENT CALIBRATION

The objective of instrument calibration is to ensure that systems used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed. The calibration data may be submitted with the data package or as a separate supplement.

Verify that the following requirements were met:

- Check that the laboratory calibrated the instrument on the day of sample analysis using a blank and at least three standards covering the range of the sample concentrations and that the calibration coefficient was at least 0.98 or better.

- Check that standards used for calibration were NIST traceable or equivalent and that certificates and a dilution log are provided.

After evaluation is complete qualify associated sample results as follows:

- If the instrument was not calibrated on the day of sample analysis, qualify associated results as unusable (R for detects, UR for non-detects).

- If the calibration coefficient is less than 0.98, qualify sample results according to the following table:

Correlation Coefficient	Qualifier
0.95 - 0.97	J for detects, UJ for non-detects
<0.95	R for detects, UR for non-detects

- If the NIST traceability certificates are unavailable and cannot be provided by the laboratory, qualify associated sample results as unusable (R for detects, UR for non-detects) providing other calibration indicators are non-compliant; otherwise, qualify sample results as estimated (J) for detected results and unusable (UR) for non-detects.

11.3 BLANKS

Blank sample results are reviewed to assess the extent of contamination introduced through sampling, sample preparation and analysis. Summarize all blank results in the validation narrative.

11.3.1 Laboratory Blanks

Verify that the laboratory blank analysis met the following criteria:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed with the samples using the same procedure.
- Results are less than or equal to the RDL.

Evaluate the laboratory blank data by verifying the following:

- Raw data including instrument printouts were provided by the laboratory.
- Results and detection limits were accurately reported.

After evaluation is complete, qualify all associated sample results as follows:

- If a laboratory blank was not performed with the associated samples, qualify all associated sample results as estimated (J for detects, UJ for non-detects).
- If positive results are present in the laboratory blanks, qualify sample results that are less than the MDA as undetected (U).
- If positive results are present in the laboratory blanks, qualify all associated positive sample results that are greater than or equal to the MDA and less than five times the highest blank concentration as estimated (J). For negative sample results, elevate the result to the RDL and qualify as undetected (U).
- If the sample result is >RDL and >5 times the associated highest blank result, no qualification is necessary.

11.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually identified as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

11.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the laboratory control or blank spike, and performance audit sample recovery values.

11.4.1 Laboratory Control or Blank Spike Samples

Verify that LCS or BSS samples met the following criteria and that the laboratory provided the following information:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed in the same analytical run, using the same procedure, as the associated samples.
- LCS or BSS activity is between 5 and 30 times the associated RDL value.
- Provided the actual LCS concentration or the spike concentration and the amount of spike added for the BSS.
- Results are within the limits of 70% to 130% recovery.

After evaluation qualify associated sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

If neither an LCS nor BSS sample was performed with the associated analytical batch, qualify the associated sample results as estimated (J for detects, UJ for non-detects)

11.4.2 Matrix Spike Samples

The matrix spike sample analysis provides information about the effect of each sample matrix on the preparation and measurement methodology.

Verify that matrix spike analyses were conducted as follows:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- Percent recovery is within the limits of 60% to 140% unless sample concentration exceeds the spike concentration by a factor of 4 or more.

Qualify associated sample results as follows:

MS %R	Sample Activity	Qualification
60% - 140%	≥ MDA < MDA	None Required None Required
≥ 10% and < 60%	≥ MDA < MDA	J UJ
> 140%	≥ MDA < MDA	J None Required
< 10%	≥ MDA < MDA	R UR

If a matrix spike sample was not performed, but is required, with the associated analytical batch, qualify associated sample results as estimated (J for detects, UJ for non-detects)

11.4.3 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample, and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

11.5 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

11.5.1 Laboratory Duplicates

Verify that the duplicate samples met the following criteria and that the laboratory provided the following information:

- The laboratory has conducted a duplicate analysis sample at a frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.
- The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.
- The relative percent difference (RPD) must be less than 20% for water samples (<35% for soils) if the sample concentration is greater than five times the RDL.
- For sample results less than five times the RDL, the difference between the primary and duplicate sample results must be less than the RDL for water samples (<2x RDL for soils).

Check all calculations and after evaluation is complete, qualify associated sample results as follows:

Original Sample Result	RPD or Range	Qualification
No duplicate analyzed	Not applicable	J for detects, UJ for non-detects
>5x RDL	>20% for waters and >35% for soils	J for detects, UJ for non-detects
<5x RDL	>RDL for waters and >2x RDL for soils	J for detects, UJ for non-detects

11.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils.

When one or both the results are < 5x RDL, the limit should be expressed as the difference between results, between the result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

11.5.3 Field Splits

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

11.6 HOLDING TIMES

Verify that all samples were preserved properly (water samples should be preserved with nitric acid, HNO_3, preferably in the field or otherwise immediately upon receipt at the laboratory) and analyzed within 180 days. If holding times are exceeded qualify sample results as follows:

- If water samples were not preserved and samples were not analyzed within 180 days, qualify all associated results as unusable (R for detects, UR for non-detects).
- If holding times are >180 days but ≤360 days qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days, qualify all associated results as unusable (R for detects, UR for non-detects).

11.7 SAMPLE RESULT QUANTITATION AND DETECTION LIMITS

Verify that the laboratory has reported the following information for each sample:

- WHC sample identification,
- laboratory sample identification,
- instrument identification,

- sample raw data including instrument readings, analysis date, and time,
- sample and QC results and required detection limits.

Check calculations according to the specified data validation level and correct sample results as necessary. Note in the validation report if MDA values do not meet the RDL values. If sample results and MDA values cannot be verified, qualify the results as estimated (J for detects, UJ for non-detects).

11.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

12.0 INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY REQUIREMENTS

This section presents specific data validation requirements selected radionuclides analyzed by inductively coupled plasma/mass spectrometry (ICP/MS) instruments.

12.1 CASE NARRATIVE

A case narrative will be included with each data package and should be reviewed for information specific to the associated data such as abnormalities encountered with the samples, matrix problems, reanalyses, and deviations from the referenced analytical method.

12.2 INSTRUMENT CALIBRATION

This section describes the specifications for initial and continuing instrument calibration.

The objective of instrument calibration is to ensure that detectors used for sample analysis were initially capable of producing quantitative results and that the calibration was maintained throughout the time period in which samples were analyzed. The initial calibration data may be submitted with the data package or as a separate supplement.

12.2.1 Tuning and Mass Calibration

Verify that the ICP/MS instrument was tuned prior to sample analysis and that the following criteria were met:

- Instrument was tuned with a mixture of the target radioisotopes at a concentration level not greater than 10 times the RDL and the tuning results are reported along with the raw data.
- The observed versus tune mass response agree within 5%.
- Tuning standards are NIST-traceable and certificates and a dilution log are provided.

If the criteria are not met, qualify the associated sample results as unusable (R for detects, UR for non-detects).

12.2.2 Initial Calibration

Verify that the initial instrument calibration met the following criteria:

- Each ICP/MS instrument used was calibrated at the beginning of each analytical run with a calibration mixture containing all radioisotopes of interest.

- Calibration standards are NIST-traceable and certificates are provided.

- Initial calibration verification (ICV) percent recoveries are within the control limits of 90% to 110% recovery.

Evaluate the initial calibration data by verifying the laboratory has provided the following raw data or that the data is available in the most recent calibration supplement:

- ICV percent recovery values for each radionuclide analyzed by ICP/MS.

- NIST traceability certificates for all calibration standards including a dilution log documenting the preparation dates, lot numbers, DPM activities, expiration dates, amount of standards used.

Check for calculation errors on at least one ICV standard. After evaluation is complete, qualify all associated sample results as follows:

- If the ICV is out of the control limits of 90% to 110%, then qualify the associated sample results for that radionuclide as estimated (J for detects, UJ for non-detects).

- If the ICP/MS instrument initial calibration raw data is unavailable and cannot be provided by the laboratory, reject all associated sample results (R for detects, UR for non-detects).

12.2.3 Continuing Calibration

Continuing calibration checks are performed periodically in order to demonstrate the instrument reliability and therefore to determine if the instrument is capable of producing acceptable quantitative results at the time the associated samples are analyzed.

Verify that the continuing instrument calibration met the following criteria:

- Continuing calibration checks were performed at a 10% frequency (after every 10 samples), or every two hours, whichever is most frequent.

- Check standards are NIST-traceable and certificates and a dilution log are provided.

- Continuing calibration verification (CCV) percent recoveries are within the control limits of 90% to 110% recovery.

After evaluation is complete, qualify all associated sample results as follows:

- If a CCV is out of the control limits of 90% to 110%, qualify the associated sample results for that radionuclide as estimated (J for detects, UJ for non-detects).

- If the associated continuing calibration data is unavailable and cannot be provided by the laboratory, reject all associated sample results (R for detects, UR for non-detects).

12.3 BLANKS

The blank data results are reviewed to assess the extent of contamination introduced through sampling, sample preparation and analysis. Summarize all blank results in the validation narrative.

12.3.1 Laboratory Blanks

The purpose of laboratory blanks is to determine if contamination is introduced in the sample through the laboratory sample preparation and analysis process.

The three different laboratory blanks analyzed for ICP/MS include:

- ICB - Initial calibration blank, analyzed after the initial calibration samples and before the laboratory and QC samples.

- CCB - Continuing calibration blank, analyzed at a 10% frequency, or every two hours, whichever is most frequent. The CCB is usually analyzed immediately after the CCV standard.

- PB - Preparation blank, digested and analyzed with the laboratory and field samples.

Verify that the following criteria were met:

- ICB was performed at the appropriate time as described above.

- CCB samples were analyzed at the specified frequency as described above.

- PB was prepared at the same time and analyzed in the same analytical run, using the same procedure, as the associated samples.

After evaluation, qualify sample results as follows:

- If any of the required blank samples were not performed within the associated sample run, then qualify all associated sample results as estimated (J for detects, UJ for non-detects).

- If positive results are present in the laboratory blanks, qualify all associated positive results that are less than five times the highest blank concentration as estimated (J).

- If the sample result is >RDL and >5 times the associated highest blank result, no qualification is necessary.

12.3.2 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually identified as equipment blanks) and sample types. Verify that the field blanks were handled in the laboratory as actual samples. No qualification is to be done based on field blank results, however the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

12.4 ACCURACY

The degree of accuracy is defined by the laboratory performance and compliance with project specific and analytical requirements as determined by the matrix spike, laboratory control or blank spike, and performance audit sample recovery values.

12.4.1 Laboratory Control or Blank Spike Samples

The laboratory control (LCS) or blank spike (BSS) sample analysis provides information concerning the effectiveness and accuracy of the laboratory method.

Verify that the LCS or BSS samples met the following criteria and that the laboratory provided the following information:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix or at least once per SDG.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- LCS or BSS activity is <100 times the RDL value.
- Provided the actual LCS concentration or the spike concentration and the amount of spike added for the BSS.
- Verify that the results are within the limits of 70% to 130% recovery.

After evaluation is complete, qualify all associated sample results as follows:

LCS or BSS %R	Sample Activity	Qualification
70% - 130%	≥ MDA < MDA	None Required None Required

LCS or BSS %R	Sample Activity	Qualification
≥ 30% and < 70%	≥ MDA < MDA	J UJ
> 130%	≥ MDA < MDA	J None Required
< 30%	≥ MDA < MDA	R UR

If neither an LCS nor BSS sample was performed with the associated analytical batch, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

12.4.2 Matrix Spike Samples

The matrix spike sample analysis provides information about the effect of each sample matrix on the preparation and measurement methodology.

Verify that the matrix spike samples met the following criteria and that the laboratory provided the indicated information:

- Performed at a 5% frequency (1 in 20 samples) all of the same matrix.
- Prepared at the same time and analyzed in the same batch, using the same procedure, as the associated samples.
- Provided the spike concentration and the amount of spike added.
- Verify that the results are within the limits of 60% to 140% recovery unless the sample activity exceeds the spike activity by a factor of four or more.

After evaluation is complete, qualify all associated sample results as follows:

MS %R	Sample Activity	Qualification
60% - 140%	≥ MDA < MDA	None Required None Required
≥ 10% and < 60%	≥ MDA < MDA	J UJ
> 140%	≥ MDA < MDA	J None Required
< 10%	≥ MDA < MDA	R UR

If a matrix spike sample was not performed with the associated analytical batch, qualify the associated sample results as estimated (J for detects, UJ for non-detects).

12.4.3 Performance Audit Samples

Performance audit samples are generated by WHC, introduced to the laboratory as a normal field sample, and used to determine the accuracy of the laboratory analytical procedure.

Contact the WHC project coordinator for the identity, source and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the final data validation report.

12.5 PRECISION

The review of field and laboratory precision provides information on the laboratory reproducibility and whether sampling activities are adequate to acquire consistent samples. Field blanks should not be used for laboratory duplicates.

12.5.1 Laboratory Duplicates

Verify that the duplicate samples met the following criteria and that the laboratory provided the following information:

- The laboratory has conducted a duplicate analysis sample at a frequency of 10% (two in twenty samples) for each matrix in each analytical batch or at least once per SDG.
- The duplicate analysis was prepared and analyzed in the same batch, using the same procedure as the associated samples.
- The relative percent difference (RPD) must be less than 20% for water samples (<35% for soils) if the sample concentration is greater than five times the RDL.
- For sample results less than five times the RDL, the difference between the primary and duplicate sample results must be less than the RDL for water samples (<2x RDL for soils).

Check all calculations and after evaluation is complete, qualify associated sample results as follows:

Original Sample Result	RPD or Range	Qualification
No duplicate analyzed	not applicable	J for detects, UJ for non-detects
>5x RDL	>20% for waters and >35% for soils	J for detects, UJ for non-detects
<5x RDL	>RDL for waters and >2x RDL for soils	J for detects, UJ for non-detects

12.5.2 Field Duplicate Sample

The preparation of field duplicate samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

Calculate the difference between the two results according to the calculations in Appendix D. The RPD limits for the field duplicates (where both results are ≥ 5x RDL) are 20% for water samples and 35% for soils. When one or both the results are < 5x RDL, the limit should be expressed as the difference between result and MDA value or the difference between the MDA values, in which the acceptable limits are the range of ± RDL for water samples and ± 2x RDL for soils. Data qualification is not required for field duplicate RPD however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision making.

12.5.3 Field Splits

A field split sample is primarily used to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. The validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data is used to help formally evaluate the project data quality objectives at the end of the data validation process and is not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

12.6 HOLDING TIMES

Verify that all samples were preserved properly (water samples should be preserved with nitric acid, HNO_3, preferably in the field or otherwise

immediately upon receipt at the laboratory) and analyzed within 180 days. If holding times are exceeded qualify sample results as follows:

- If water samples were not preserved and samples were not analyzed within 180 days, qualify all associated results as unusable (R for detects, UR for non-detects).
- If holding times are >180 days but ≤360 days qualify all results as estimated (J for detects, UJ for non-detects).
- If holding times are >360 days, qualify all associated results as unusable (R for detects, UR for non-detects).

12.7 SAMPLE RESULT QUANTITATION AND MINIMUM DETECTABLE ACTIVITIES

Verify that the laboratory has reported the following information for each sample:

- WHC sample identification,
- laboratory sample identification,
- instrument identification,
- sample analysis date and time,
- sample analysis raw data,
- sample results and detection limits,
- sample preparation data,
- required detection limits.

Check calculations according to the specified data validation level and correct sample results as necessary. Note in the validation report if MDA values do not meet the RDL values. If sample results and MDA values cannot be verified, qualify the results as estimated (J for detects, UJ for non-detects).

12.8 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the qualified results as specified in Section 13.0, Reporting Requirements.

13.0 REPORTING REQUIREMENTS

This section presents reporting requirements for validation reports on both a sample group and overall case basis, where several groups of sample analyses are summarized for inclusion into individual environmental site investigation reports. The three types of deliverables required for data validation activities are summarized below:

- Data validation packages — validation documentation and qualified results prepared and submitted with the original analytical data package for inclusion in the project QA record
- Data validation summary reports — a report prepared which summarizes the validation of multiple data packages on a project basis such as a round of groundwater sampling or a group of samples collected for a project
- Electronic data deliverables — validated data provided in a specific electronic format at the conclusion of validation of multiple data packages on a project basis. The frequency of submittal of electronic data will be determined on a case by case basis

13.1 DATA VALIDATION PACKAGES

After completing the validation of a single data package and analysis type or group, summarize the results of the validation in a technical memorandum that addresses the following items:

- **Introduction**—This section of the memorandum shall provide a short introduction identifying the samples and analyses validated, laboratories involved, and applicable plans and specifications.
- **Data Quality Objectives**—This section of the memorandum shall provide a brief summary of the degree to which project specific data quality objectives were met as related to the sample analysis. Brief summaries of the precision, accuracy, sample result verification, detection limits, and completeness shall be addressed.
- **Major Deficiencies**—This section will address major deficiencies that resulted in the qualification of sample data as unusable.
- **Minor Deficiencies**—This section will address minor deficiencies that resulted in the qualification of sample data as estimated.
- **References**—This section will provide a list of references used for validation of the subject data.

Attached to the memo will be an explanation of the data validation qualifiers applied to the sample results, a copy of the annotated laboratory report forms, and a copy of the data validation supporting documentation.

The memo and attachments will be inserted in the front of the original data package and returned to HASM within 21 calendar days of receipt of the data package. An example of this report format is provided in Appendix B.

13.2 DATA VALIDATION SUMMARY REPORTS

At the completion of a project that involves several analytical data packages, a final narrative summary will be prepared, reviewed, and submitted to the WHC project coordinator. Attached to this report will be a tabulated data summary of all validated data and copies of the annotated laboratory reports. At a minimum, the tabular summary must provide the HEIS number, sample collection date, sample location (if available), sample type, constituent name, constituent result, result qualifier, and constituent reporting limits. In preparation of this tabular data summary, the validator must have a system of performing a 100% check for transcription errors of all data against the written documentation. An outline for this type of report is provided in Appendix C.

13.3 ELECTRONIC DATA TRANSMITTAL REQUIREMENTS

At the conclusion of a validation project, results of the validated data are additionally to be provided in the format described in Table 13-1 on a 3.5-inch disk in MS-DOS[1] high density format compatible with the applicable subject areas specified in the HEIS Users Manual (WHC 1990). This requirement shall apply to analytical data initially provided to the validators in the format specified in Table 13-1 or in CLP-Format A electronic format to facilitate loading, manipulation, and update of analytical results and qualifier flags.

Each record in the transmittal file is designed to contain the analytical results for one chemical analysis parameter. All fields in the record are to be fixed-length, containing no special format codes, delimiters, or separators. Data entry fields marked with an asterisk (*) in Table 13-1 refer to fields in the transmittal file that must contain the specified information, since these fields make up the unique identifier used by HEIS for retrieval of the record. The remainder of the fields are to report data changes. Data shall be supplied for records with changed data fields only such as the value_rptd and qualifier fields. Each line in the transmitted file must contain 76 characters plus one additional character for the end-of-line terminator (typically the carriage return character). Tentatively identified compound results shall be transmitted only if a valid Chemical Abstracts Service (CAS) number is supplied for the result.

The validator must have a procedure in place for verifying the accuracy of the electronic data with the written record if changes are made as a result of the validation effort; this procedure shall be submitted to the WHC project coordinator for approval prior to use. At a minimum, a 100% check of all changed data against the written documentation must be performed.

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[1]MS-DOS is a trademark of Microsoft Corporation, Redmond, Washington.

The WHC project coordinator and HEIS coordinator may specify options for electronic data submittals on a case by case basis since laboratory ectronic data transmittal formats are currently in development.

14.0 REFERENCES

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15.0 ACRONYMS AND ABBREVIATIONS

CCV	continuing calibration verification
cpm	counts per minute
RDL	required detection limit
%D	Percent Difference
dpm	Disintegrations per minute
dps	Disintegrations per second
EPA	U.S. Environmental Protection Agency
eV	electron volt
FWHM	Full Width at Half Maximum
g	gram
GPC	gas proportional counting
HEIS	Hanford Environmental Information System
ICP/MS	inductively-coupled plasma/mass spectrometry
ICP	inductively-coupled plasma
ICV	initial calibration verification
KeV	kilo electron volts
LCS	laboratory control sample
LLD	lower limit of detection
LSC	liquid scintillation counter
MDA	minimum detectable activity
MeV	million electron volts
MS	matrix spike
MSD	matrix spike duplicate
NIST	National Institute of Standards and Technology
QA	quality assurance
QAPjP	quality assurance project plan
QC	quality control
r	correlation coefficient
RF	response factor
RPD	relative percent difference
%R	percent recovery
SAR	sample analysis request
SDG	sample delivery group
sigma	standard deviation
SOW	statement of work
WHC	Westinghouse Hanford Company

16.0 GLOSSARY

Abundance: The number of photons of a specific energy emitted by 100 atom decays.

Accuracy: The degree of agreement of measurement (or an average of several measurements of the same thing) with an accepted reference or true value.

Activity: The rate of decay of a radioactive source.

Aliquot: A measured portion of a sample taken for analysis.

Alpha Particle: A ^{4}He nucleus emitted by nuclei undergoing alpha decay. Most alpha particle energies range between 4 and 6 MeV.

Analysis date/time: The date and military time (24-hour clock) of the start of a count on a prepared sample.

Analysis: The separation and determination of the component parts or a specific property or element contained within a sample. The determination of the concentration or activity of an analyte contained within a sample.

Analyte: For radiochemistry analysis, the specific isotope or radionuclide of interest which an analyst seeks to determine; the radioactive element of interest.

Analytical Batch: A group of samples of the same matrix analyzed together using the same method and containing the required number of method blanks, matrix spike samples, lab control samples, and duplicate samples.

Analytical sample: Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, calibration verification, and calibration blank.

Autozero: Zeroing the instrument at the proper setting. It is equivalent to running a standard blank with the instrument response set at a value of zero.

Average Intensity: The mathematical average of at least two different intensity measurements.

Background: Random counts detected by the instrument which arise from sources other than the sample being analyzed, such as interfering isotopes within the reagents used for sample preparation, detector contamination, electronic noise, and cosmic rays.

Background Water: Tritium-free water used for sample analysis in which the tritium present is near undetectable. Tritium content of samples is measured relative to the background water.

Batch: A group of samples prepared at the same time in the same location using the same method.

Beta Particle: A highly energetic electron emitted by a nucleus undergoing beta decay.

Blank: An artificial sample designed to monitor the introduction of artifacts into the measurement process. For aqueous samples, reagent water is used as a blank matrix. A universal matrix does not exist for solid samples; therefore, no matrix or reagent water is routinely used. There are several types of blanks, that monitor a variety of processes:

- A **Laboratory Blank** is taken through sample preparation and analysis only. It is a test for contamination in sample preparation and analyses.
- A **Trip Blank** is shipped to and from the field with the sample containers. It is not opened in the field, and therefore, provides a test for contamination from sample preservation, site conditions, and transport as well as sample storage, preparation, and analysis.
- A **Field Blank** is opened in the field and tests for contamination from the atmosphere as well as those activities listed under trip blank.
- An **Equipment Blank** is poured appropriately over or through sample collection devices and tests for the cleanliness of sampling equipment as well as those activities listed under field blank.

Trip, field, and equipment blanks are handled by the laboratory as actual samples. However, they should not be used for matrix spike or duplicate samples.

Blank Spike Sample (BSS): A blank spike sample is a known, clean sample matrix spiked with a known composition. Blank spike samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

Calibration Verification: The periodic analysis of one or more standards independent of the calibration standards to verify the accuracy and stability of the initial instrument calibration.

Calibration Blank: A volume of acidified deionized/distilled water, or empty planchet or geometry analyzed to establish the instrument accuracy at the low end of the calibration curve.

Calibration: The establishment of an instrument response curve or mathematical correlation based on the measured response of a known concentration of radiochemical analyte or group of analytes.

Case: A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers may be assigned by the Hanford Environmental Information System (HEIS). A Case may consist of one or more Sample Delivery Groups (SDG).

Chain of Custody: A document designed to trace the custody of a sample(s) from the point of origin to final disposition with the intent of legally

proving that custody remained intact and that tampering or substitutions were precluded.

Checksource: A radioactive source which is used to verify the calibration of the counting systems.

Chemical Carrier: A quantity of non-radioactive or non-labeled material of the same or a chemically similar composition as the corresponding radioactive or labeled constituent being analyzed.

Chemical Yield: The amount of carrier recovered compared to the amount added. The chemical yield is used as a correction factor in the calculation of the final analytical result.

Coefficient of Variation (CV): The standard deviation as a percent of the arithmetic mean.

Comparability: The degree of confidence with which one set of data can be compared to a related set of data.

Completeness: A measure of the amount of valid data obtained from a measurement system relative to the amount that was expected to be obtained under current, normal conditions.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA): A Federal law passed in 1980 and modified in 1986 by the Superfund Amendments and Reauthorization Act. The Acts created a special tax that goes into a Trust Fund, commonly known as Superfund, to investigate and clean up abandoned or uncontrolled hazardous waste sites. Under the program, EPA can either:

- Pay for the site cleanup when parties responsible for the contamination cannot be located, or are unwilling or unable to perform the work.
- Take legal action to force parties responsible for site contamination to clean up the site or repay the federal government for the cost of the cleanup.

Concentration: The relative fraction of one substance in another, normally expressed in weight percent, volume percent, or as a weight per volume ratio.

Continuing Calibration: The analysis of one or more checksource standards analyzed periodically, on a daily to weekly basis, in order to verify that the initial calibration continues to be valid.

Control Limits: A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

Correlation Coefficient (r): A numeric value (r) which indicates the degree of dependence between two variables (concentration vs response). The more

dependent they are, the closer the value to one. Determined on the basis of the least squares function.

Curie: 3.7×10^{10} disintegrations per second

Custody: Immediate charge, control, or possession exercised by a person or competent authority on a sample.

Day (d): Unless otherwise specified, day shall mean calendar day.

Detection: The act of measuring the quantity of a property, compound or element contained in a sample.

Disintegrations per minute (dpm): The number of times a radioactive element undergoes radioactive decay in one minute.

Disintegrations per second (dps): The number of times a radioactive element undergoes radioactive decay in one second.

Dry Weight: The weight of a sample based on percent solids or the weight after drying in an oven for a specified time period at a temperature of 105°C.

Duplicate: A second aliquot of a homogenized sample which is analyzed as an individual sample, using the same procedure. This is used to determine the precision of the method.

Efficiency: The number of counts per minute (cpm) registered on an instrument divided by the disintegrations per minute (dpm) value of the standard being used to check the efficiency.

Energy Resolution: A value representing the peak Full Width at Half Maximum (FWHM) (in KeV) divided by the energy of the peak in the assigned centroid channel; expressed as a percentage.

Field Blank: A blank sample prepared in the field at the sample collection site and returned to the lab with the samples to be analyzed. The blank measures contamination introduced during sample collection. Any sample submitted from the field identified as a blank.

Field Screening: An investigative technique utilizing analytical chemistry at or near a worksite to rapidly determine the presence or absence of environmental contaminants and the approximate concentrations of specific "target" compounds.

Field Sample: A portion of material received at the laboratory to be analyzed and that is contained in single or multiple containers and identified by a unique HEIS Sample Number.

Frequency (10%): A frequency specification during an analytical sequence allowing for no more than 10 analytical samples between required calibration verification measurements, as specified by the contractual SOW.

Full Width at Half Maximum (FWHM): The width of the peak distribution at a level that is just half of the maximum height of the peak.

Half-Life: The time required for one half of the initial number of radioactive nuclei to undergo radioactive decay.

Holding Time: The maximum amount of time allowed for samples to be held from sample collection to laboratory analysis.

Independent Standard: A laboratory-prepared standard solution that is composed of analytes from a different source than those used in the standards for the initial calibration.

Inductively-Coupled Plasma (ICP): A technique for the simultaneous or sequential multi-element determination of analytes in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio-frequency ICP.

Initial Calibration: The analysis of standards containing varying concentration levels of analytes or activities of the radioactive element of interest in order to establish the ratio of concentration vs response across the working range of the analytical technique. The initial calibration is used to define the linearity and dynamic range of response of the detector to the target isotopes or radionuclides.

Internal Standards: Internal standards may be used as the basis for the quantitation. For example, of tritium, in which two identical aliquots are prepared for each sample, blank, matrix spike, and duplicate. One aliquot is spiked with a standard at a known concentration prior to analysis, the other aliquot is not spiked. The recovery is determined by using the difference of the two results and dividing by the amount of internal standard added, then multiplying by 100 for the percentage.

Instrument Detection Limit (IDL): Determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration estimated to be at three to five times the IDL on three nonconsecutive days with seven consecutive measurements performed per day.

Instrument Calibration: The analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to the radionuclides of interest.

Interferents: Substances that affect the analysis for the element of interest.

Isotope: One of a number of specific atoms with identical atomic numbers but with discrete atomic weights, or similarly specific atoms whose nuclei have the same number of protons but different numbers of neutrons.

KeV: kilo electron volt or 10^3 volts.

Laboratory Blank: A known, clean sample matrix carried through all sample preparation and analysis procedures. In some instances there is no sample matrix but all other preparation analysis procedures are performed. A laboratory blank should be analyzed concurrently with each batch of samples analyzed. This blank measures any contamination due to the laboratory environment or materials.

Laboratory Control Sample (LCS): A control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

Linear Range, Linear Dynamic Range: The concentration range over which the calibration curve remains linear.

Log-In: The receipt and initial management of the sample. It generally involves acknowledging complete chain-of-custody, noting report and invoice information, recording the analysis requested (including methodology and/or special instructions), and assigning a discreet internal laboratory identification (usually a number or bar code) for tracking the progress of the sample analysis within the laboratory.

Micro Curie (μCi): 1×10^{-6} Curies.

Matrix: The predominant material of which the sample to be analyzed is composed. For the purpose of this document, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid). This refers to the physical characteristics or state of a sample (e.g., water, soil/sediment, sludge, gas, etc.).

Matrix Interference: The influence of the sample matrix or sample components upon the ability to quantitatively measure compounds in environmental samples.

Matrix Spike/Matrix Spike Duplicate (MS/MSD): A first and second aliquot of a matrix (water or soil) fortified (spiked) with a known quantity of analyte(s) and subjected to the entire analytical procedure in order to determine the appropriateness of the method for the matrix by measuring accuracy (recovery) and precision (relative percent difference).

Method Blank: An analytical control consisting of all reagents, internal standards and chemical carriers or tracers, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination.

Minimum Detectable Activity (MDA): The smallest quantity of a radionuclide that can be detected in a sample with a 95% confidence level. Expressed as a data quality objective (DQO), the MDA should be less than or equal to the RDL.

Narrative (SDG narrative): A portion of the data package that includes laboratory, contract, Case and sample identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. SDG narrative

specifications are typically included in the contractual SOW to the laboratory.

Nuclide: General term applied to all isotopes of all elements including stable and radioactive forms. Nuclides are not considered isotopes. A given nuclide is characterized by the number of neutrons and protons contained in the atomic nuclei of that species.

Parts Per Billion (ppb) / Parts Per Million (ppm): Units commonly used to express low concentrations of contaminants. For example, 1 oz. of uranium in one million ounces of water is 1 ppm; 1 oz. of uranium in one billion ounces of water is 1 ppb.

Percent Recovery (%R): A measure of recovery that is calculated as the measured value relative to the true value, expressed as a percent.

Percent Moisture: An approximation of the amount of water in a soil/sediment sample determined by drying an aliquot of sample at 105°C until constant weight is achieved.

Percent Solids: The proportion of solid in a soil/sediment sample determined by the percent moisture procedure.

Performance Evaluation (PE) Sample: A sample of known composition which may be provided by the U.S. Department of Energy, Westinghouse Hanford Company, EPA, or Washington State Department of Ecology (Ecology) for laboratory analysis and which is used by these organizations to evaluate laboratory performance.

pico Curie (pCi): 1×10^{12} Curies or 2.22 dpm.

Precision: The agreement or repeatability of a set of replicate results among themselves, usually expressed in terms of the deviation of a set of results from the arithmetic mean. Precision may be qualified in terms of possible sources of variability, replicability, repeatability, and reproducibility.

Preparation Blank: An analytical control that contains purified or distilled, deionized water and reagents, which is carried through the entire analytical procedure (digested and analyzed). An aqueous method blank is treated with the same reagents as a sample with a water matrix. A solid method blank is treated with the same reagents as a soil sample.

Preparation Log: An official record of the sample preparation.

Preservative: Either a chemical compound or reagent added to a sample to prevent or slow decomposition or degradation of a target analyte or a physical process (such as cooling) used for the same purpose. Both physical and chemical preservation may be used in tandem to prevent sample deterioration.

Protocol: Describes the exact procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control. Used synonymously with SOW.

Qualitative Analysis: An analysis to determine the presence or absence of a target analyte.

Quality Assurance (QA): All planned and systematic actions necessary to provide adequate confidence in laboratory results.

Quality Control (QC): Quality assurance actions that provide a means to control and measure the characteristics of measurement equipment and processes to meet established quality requirements.

Quantitative Analysis: An analysis to measure or determine the amount of a target compound or analyte within the limits of defined precision and accuracy requirements.

Quenching: The interference with the conversion of decay energy to signal measured in the photomultiplier tube, commonly resulting in a reduction in counting efficiency.

Quench Monitor: The value obtained by the instrument indicating the level or degree of quenching in the sample.

Radionuclide: Any radioactive isotope of an element.

Range: The difference between the maximum and minimum values within a set of values.

RCRA: See Resource Conservation and Recovery Act.

Reagent Blank: A known, clean sample matrix carried through all sample preparation and analysis procedures. In some instances there is no sample matrix but all other preparation analysis procedures are performed. A reagent blank should be analyzed concurrently with each batch of samples analyzed. This blank measures any contamination due to the laboratory environment or materials.

Reagent Water: Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest.

Recovery: A determination of accuracy of the analytical procedure made by comparing measured values for a reference or fortified (spiked) sample against the known true reference or spike values.

Relative Percent Difference (RPD): A measure of precision that is calculated as the absolute value of the difference between two results, relative to their arithmetic mean, expressed as a percent.

Relative Percent Error: The difference between the observed value and the expected value divided by the expected value and multiplied by 100.

Relative Standard Deviation (RSD): A measure of precision that is calculated as the standard deviation(s) of a set of values, relative to their arithmetic mean (x), expressed as a percent.

Replicability: The precision of repeated, independent measurements made on the same sample by the same analyst at essentially the same time and under the same conditions.

Reproducibility: The precision of measurements of the same sample at different laboratories using the same protocols.

Resolution: The degree to which two signal peaks are separated. Resolution is calculated by dividing the height of the valley between the peaks by the peak height of the peak being resolved, multiplied by 100.

Resource Conservation and Recovery Act (RCRA): A 1976 federal law that established a regulatory system to define and track hazardous wastes from the time of generation to disposal. The law requires safe and secure procedures to be used in treating, transporting, storing, and disposing of hazardous substances.

Rounding Rules: The following are instructions for rounding off or reducing the number of significant figures in a numeric result. If the figure following those to be retained is <5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44. If the figure following those to be retained is >5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45. If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the 5, the figure 5 is dropped, and the last-place figure retained is increased by 1 if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42. In general, if a series of multiple operations are to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

- When rounding off a result from a series of arithmetic operations, the result is rounded to the same number of decimal places as the number with the smallest number or places. However, the operation is completed with all decimal places intact and rounding off is done only on the final result to prevent significant round-off error.

Run: A continuous analytical sequence consisting of prepared samples and all associated QA measurements as required by the contract SOW.

Self-Absorption: The internal absorption of radiation emitted by radioactive atoms by material in which the radioactive atoms are located.

Sample Delivery Group (SDG): A unit within a single Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a Case, received over a period of up to 14 calendar days. Usually, data from all samples contained in an SDG are due concurrently. An SDG is defined by one of the following, whichever occurs first:

- Case

- Each 20 field samples within a Case
- Each 14-day calendar period during which field samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory.

Standard Deviation: The measurement of dispersion about a mean value of a series of observations expressed in the same units as the mean value.

Sample: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Sample Matrix: All of the chemical components and physical characteristics of a sample other than parameter of interest.

Sample Number (HEIS Sample Number): A unique identification number designated by HEIS for each sample. The HEIS sample number appears on the sample chain of custody and shipping documentation that documents information on that sample.

Scintillation Cocktail: The solution in which samples are placed for measurement in a Liquid Scintillation Counter (LSC). The solution is made up of solvents and scintillators.

Sensitivity: The ability of a measurement system to detect and accurately quantitate a parameter at a critical level within a specific sample matrix. The critical level may be a regulatory maximum contaminant level (MCL), MDA, or risk-based exposure level.

Significant Figures: The term "significant figure" refers to a judgment process regarding reportable digits in a numerical result. This process must be based on sound judgment such that meaningful digits are retained, meaningless digits are discarded. The following describes the process for retention of significant digits:

- A number is an expression of quantity composed of any of the characters 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, which, alone or in combination, serve to express a number. A significant figure is a digit that denotes that amount of the quantity in the particular decimal place in which it stands. Reported analytical values should contain only significant figures. A value is made up of significant figures when it contains all digits known to be true and one last digit in doubt. For example, if a value is reported as 18.8 mg/L, the 18 must be firm while the 0.8 is some what uncertain, but presumably better than one of the values 0.7 or 0.9 would be
- The number zero may or may not be a significant figure depending on the situation

- Final zeros after a decimal point are always meant to be significant figures. For example, if weighed to the nearest milligram, the value 9.8 grams is reported as 9.800 grams

- Zeros before a decimal point with nonzero digits preceding them are significant. With no preceding nonzero digit, a zero before the decimal point is not significant

- If there are no nonzero digits preceding a decimal point, the zeros after the decimal point but preceding other nonzero digits are not significant. These zeros only indicate the position of the decimal point

- Final zeros in a whole number may or may not be significant. For example, in a conductivity measurements of 1,000 μmho/cm, there is no implication by convention that the conductivity is 1,000 ± 1 μmho. Rather, the zeros only indicate the magnitude of the number

- Zeros are significant if they cannot be dropped from a number when expressing the number in exponential form (i.e., 100.08)

- Zeros are not significant if they can be dropped from a number when expressing the number in exponential form (i.e., 0.0008).

Sludge: Solid, semisolid, or liquid waste generated from a municipal, commercial, or industrial waste treatment facility or wastewater treatment plant, water supply treatment plant, or air pollution control facility exclusive of treated effluent from a wastewater treatment plant.

Soil: Used herein synonymously with soil/sediment and sediment.

Solvent: Liquid that is capable of dissolving another substance. Solvents are used in a number of manufacturing/industrial processes including the manufacture of paints and coating for industrial and household purposes, equipment cleanup, dry cleaning and surface degreasing in metal fabricating industries.

Standard Analysis: An analytical determination made by comparison with known quantities of specific analytes, compounds, or radioactive elements.

Stock Solution: A standard solution that can be diluted to derive other standards.

Technical Holding Time: The storage time allowed between sample collection and sample analysis when designated preservation and storage techniques are employed. This is determined by the elapsed time in days from the date and time of collection to the date and time of sample preparation and analysis.

- Technical holding time = (sample analysis date and time - sample collection date and time).

Time: When required to record time on any deliverable item, time shall be expressed as Military Time, i.e., a 24-h clock.

Tracer: A quantity of a unique radioisotope of the same element added to a sample, chemically prepared or separated and counted. The quantity of tracer measured is compared to the quantity of target radioactive element measured and the target quantity is calculated on the basis of unity with the tracer concentration.

Trip Blank: A blank sample which travels with sample containers to the sampling site and returns to the lab with the samples to be analyzed. The blank measures contamination during sample transport and typically only analyzed for volatile organic compounds.

Uncertainty: The error associated with the measurement of the activity of a radioactive isotope which takes into account the random nature of the decay process and the finite count duration.

Wet Weight: The weight of a sample aliquot including moisture (undried).

APPENDIX A

RADIOCHEMICAL DATA VALIDATION CHECKLIST

RADIOCHEMICAL DATA VALIDATION CHECKLIST

VALIDATION LEVEL:	A	B	C ___	D	E
PROJECT:			DATA PACKAGE:		
VALIDATOR:		LAB:		DATE:	
CASE:			SDG:		
ANALYSES PERFORMED					
☐ Gross Alpha/Beta	☐ Strontium-90	☐ Technetium-99	☐ Alpha Spectroscopy	☐ Gamma Spectroscopy	
☐ Total Uranium	☐ Radium-22	☐ Tritium	☐		
SAMPLES/MATRIX					

1. Completeness . . . . . ☐ N/A

Technical verification forms present? . . . . . Yes No N/A

Comments:_____

2. Initial Calibration . . . . . ☐ N/A

Instruments/detectors calibrated within one year of sample analysis? . . . . . Yes No N/A

Initial calibration acceptable? . . . . . Yes No N/A

Standards NIST traceable? . . . . . Yes No N/A

Standards Expired? . . . . . Yes No N/A

Comments:_____

3. Continuing Calibration . . . . . ☐ N/A

Calibration checked within one week of sample analysis? . . . Yes No N/A
Calibration check acceptable? . . . . . Yes No N/A
Calibration check standards NIST traceable? . . . . . Yes No N/A
Calibration check standards expired? . . . . . Yes No N/A

Comments:_____

4. Blanks . . . . . ☐ N/A

Method blank analyzed? . . . . . Yes No N/A
Method blank results acceptable? . . . . . Yes No N/A
Analytes detected in method blank? . . . . . Yes No N/A
Field blank(s) analyzed? . . . . . Yes No N/A
Field blank results acceptable? . . . . . Yes No N/A
Analytes detected in field blank(s)? . . . . . Yes No N/A
Transcription/Calculation Errors? . . . . . Yes No N/A

Comments:_____

5. Matrix Spikes . . . . . ☐ N/A

Matrix spike analyzed? . . . . . Yes No N/A
Spike recoveries acceptable? . . . . . Yes No N/A
Spike source traceable? . . . . . Yes No N/A
Spike source expired? . . . . . Yes No N/A
Transcription/Calculation Errors? . . . . . Yes No N/A

Comments:_____

6. Laboratory Control Samples . . . . . ☐ N/A

LCS analyzed? . . . . . Yes No N/A
LCS recoveries acceptable? . . . . . Yes No N/A
LCS traceable? . . . . . Yes No N/A
Transcription/Calculation Errors? . . . . . Yes No N/A

Comments:_____

7. Chemical Recovery . . . . . ☐ N/A

Chemical carrier added? . . . . . Yes No N/A
Chemical recovery acceptable? . . . . . Yes No N/A
Chemical carrier traceable? . . . . . Yes No N/A
Chemical carrier expired? . . . . . Yes No N/A
Transcription/Calculation errors? . . . . . Yes No N/A

Comments:_____

8. Duplicates . . . . . ☐ N/A

Duplicates Analyzed? . . . . . Yes No N/A
RPD Values Acceptable? . . . . . Yes No N/A
Transcription/Calculation Errors? . . . . . Yes No N/A

Comments:_____

9. Field QC Samples . . . . . ☐ N/A

Field duplicate sample(s) analyzed? . . . . . Yes No N/A
Field duplicate RPD values acceptable? . . . . . Yes No N/A
Field split sample(s) analyzed? . . . . . Yes No N/A
Field split RPD values acceptable? . . . . . Yes No N/A
Performance audit sample(s) analyzed? . . . . . Yes No N/A
Performance audit sample results acceptable? . . . . . Yes No N/A

Comments:_____

10. Holding Times

Are sample holding times acceptable? . . . . . Yes No N/A

Comments:_____

11. Results and Detection Limits (Levels D & E) . . . . . ☐ N/A

Results reported for all required sample analyses? . . . . . Yes No N/A
Results supported in raw data? . . . . . Yes No N/A
Results Acceptable? . . . . . Yes No N/A
Transcription/Calculation errors? . . . . . Yes No N/A
MDA's meet required detection limits? . . . . . Yes No N/A
Transcription/calculation errors? . . . . . Yes No N/A

Comments:_____

Comments:

APPENDIX B

EXAMPLE DATA VALIDATION PACKAGE FORMAT

MEMORANDUM

TO: (Project Name) QA Record

FROM: (Data Validator and Company Name)

DATE: (Date of Report)

SUBJECT: RADIOCHEMICAL ANALYSIS DATA VALIDATION SUMMARY FOR DATA PACKAGE: (DATA PACKAGE TRACKING NUMBER)

INTRODUCTION

DATA QUALITY OBJECTIVES

Precision:

Accuracy:

Sample Result Verification:

Detection Limits:

Completeness:

MAJOR DEFICIENCIES (Rejected Data)

MINOR DEFICIENCIES (Qualified Data)

REFERENCES

ATTACHMENTS

ATTACHMENT 1 - GLOSSARY OF DATA VALIDATION QUALIFIERS

ATTACHMENT 2 - QUALIFIED (ANNOTATED) LABORATORY SAMPLE REPORTS

ATTACHMENT 3 - DATA VALIDATION SUPPORTING DOCUMENTATION

ATTACHMENT 1

GLOSSARY OF DATA VALIDATION QUALIFIERS

U - The constituent was analyzed for, but was not detected. The value reported is the minimum detectable activity (MDA) corrected for sample dilution and moisture content by the laboratory. The data should be considered usable for decision making purposes.

UJ - The constituent was analyzed for and was not detected. Due to a quality control deficiency identified during data validation the value reported may not accurately reflect the MDA. The data should be considered usable for decision making purposes.

J - Indicates the constituent was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. The data should be considered usable for decision making purposes.

UR - Indicates the constituent was analyzed for and not detected; however, due to an identified quality control deficiency the data should be considered unusable for decision making purposes.

R - Indicates the constituent was analyzed for and detected; however, due to an identified quality control deficiency the data should be considered unusable for decision making purposes.

ATTACHMENT 2

QUALIFIED (ANNOTATED) LABORATORY SAMPLE REPORT FORMS

RADIOCHEMICAL ANALYSIS DATA SHEET

SAMPLE NO.

EXAMPLE

Lab Name: WHC Contract Laboratory Contract: EXAMPLE

Lab Code: WHC001 Case No.: SAMPLE SAS No.: SAMPLE SDG No.: SAMPLE

Matrix: (soil/water) water

Lab Sample ID: EXAMPLE-1

Lab File ID: EXAMPLE-1

Date Received: 10/5/93

CONSTITUENT	CONCENTRATION UNITS (pCi/L)	Error	MDA	Q
Gross Alpha	3.12	1.3	2	
Gross Beta	6.5 ~~4.3~~	5.4	3	
~~Strontium-90~~	~~2.2~~	~~3.3~~	~~1~~	
~~Technetium-99~~	~~5~~	~~2~~	~~4~~	
Uranium-234	0.14	0.08	0.05	
Uranium-235	0.03	0.03	0.01	
Uranium-238	0.15	0.05	0.05	
Potassium-40	3.2	1.1	2	
Chromium-51	1.1	0.3	0.5	
Iron-59	4.5	1.2	0.5	
Cobalt-58	10		10	U
Cobalt-60	20		20	U
Ruthenium-103	100		100	~~U~~
Ruthenium-106	200		200	~~U~~
Cesium-134	3.14	1.4	1	
Cesium-137	5.2	3.4	3	
Europium-152	15		15	U
Europium-153	4		4	U
Europium-154	12		12	U

ATTACHMENT 3

DATA VALIDATION SUPPORTING DOCUMENTATION

RADIOCHEMICAL DATA VALIDATION CHECKLIST

VALIDATION LEVEL:	A	B	C	D	E
PROJECT: WHC			DATA PACKAGE: SAMPLE		
VALIDATOR: [illegible]		LAB: WHC Contract Lab		DATE: 10/11/93	
CASE: SAMPLE			SDG: Sample		
ANALYSES PERFORMED					
☒ Gross Alpha/Beta	☒ Strontium-90	☒ Technetium-99	☐ Alpha Spectroscopy	☒ Gamma Spectroscopy	
☒ Total Uranium	☐ Radium-22	☐ Tritium	☐		
SAMPLES/MATRIX Example, water					

1. Completeness . . . . . ☐ N/A

Technical verification forms present? . . . . . (Yes) No N/A

Comments: _____

2. Initial Calibration . . . . . ☐ N/A

Instruments/detectors calibrated within one year of sample analysis? . . . . . (Yes) No N/A

Initial calibration acceptable? . . . . . (Yes) No N/A

Standards NIST traceable? . . . . . (Yes) No N/A

Standards Expired? . . . . . Yes (No) N/A

Comments: _____

3. Continuing Calibration . . . . . ☐ N/A

Calibration checked within one week of sample analysis? . . (Yes) No N/A
Calibration check acceptable? . . . . . Yes (No) N/A
Calibration check standards NIST traceable? . . . . . (Yes) No N/A
Calibration check standards expired? . . . . . Yes (No) N/A

Comments: Calibration check assoc. w/ Tc-99 was below the lower control limit, qualify as unusable. (R).

4. Blanks . . . . . ☐ N/A

Method blank analyzed? . . . . . (Yes) No N/A
Method blank results acceptable? . . . . . (Yes) No N/A
Analytes detected in method blank? . . . . . (Yes) No N/A
Field blank(s) analyzed? . . . . . Yes (No) N/A
Field blank results acceptable? . . . . . Yes No (N/A)
Analytes detected in field blank(s)? . . . . . Yes No (N/A)
Transcription/Calculation Errors? . . . . . Yes (No) N/A

Comments: Potassium-40 was detected in the lab blank at 2 pCi/L, action Level = 5 x 2 pCi/L = 10 pCi/L, qualify as estimated (J).

5. Matrix Spikes . . . . . ☐ N/A

Matrix spike analyzed? . . . . . (Yes) No N/A
Spike recoveries acceptable? . . . . . (Yes) No N/A
Spike source traceable? . . . . . (Yes) No N/A
Spike source expired? . . . . . Yes (No) N/A
Transcription/Calculation Errors? . . . . . Yes (No) N/A

Comments:

6. Laboratory Control Samples . . . . . ☐ N/A

LCS analyzed? . . . . . (Yes) No N/A
LCS recoveries acceptable? . . . . . Yes (No) N/A
LCS traceable? . . . . . (Yes) No N/A
Transcription/Calculation Errors? . . . . . Yes (No) N/A

Comments: Gross alpha LCS %R = 65%, qualify as estimated (J). LCS%R are low for ruthenium 103 (50%R) and ruthenium 106 (45%R), qualify associated results as estimated (UJ).

7. Chemical Recovery . . . . . ☐ N/A

Chemical carrier added? . . . . . (Yes) No N/A
Chemical recovery acceptable? . . . . . Yes (No) N/A
Chemical carrier traceable? . . . . . (Yes) No N/A
Chemical carrier expired? . . . . . (Yes) No N/A
Transcription/Calculation errors? . . . . . Yes (No) N/A

Comments: Sr-90 chemical carrier recovery = 16%, qualify as unusable (R).

8. Duplicates . . . . . ☐ N/A

Duplicates Analyzed? . . . . . (Yes) No N/A
RPD Values Acceptable? . . . . . (Yes) No N/A
Transcription/Calculation Errors? . . . . . Yes (No) N/A

Comments:

9. Field QC Samples . . . . . ☒ N/A

Field duplicate sample(s) analyzed? . . . . . Yes No N/A
Field duplicate RPD values acceptable? . . . . . Yes No N/A
Field split sample(s) analyzed? . . . . . Yes No N/A
Field split RPD values acceptable? . . . . . Yes No N/A
Performance audit sample(s) analyzed? . . . . . Yes No N/A
Performance audit sample results acceptable? . . . . . Yes No N/A

Comments:

10. Holding Times

Are sample holding times acceptable? . . . . . (Yes) No N/A

Comments:

11. Results and Detection Limits . . . . . ☐ N/A

Results reported for all required sample analyses? . . . . . (Yes) No N/A
Results supported in raw data? . . . . . (Yes) No N/A
Results Acceptable? . . . . . See next page . . Yes (No) N/A
Transcription/Calculation errors? . . . . . (Yes) No N/A
MDA's meet required detection limits? . . . . . (Yes) No N/A
Transcription/calculation errors? . . . . . Yes (No) N/A

Comments:
The gross beta sample result was corrected from 4.3 pCi/L to 6.5 pCi/L as according to the raw data.

Comments:

The Tc-99 and Sr-90 results for sample "Example" have been qualified as unusable (R) due to calibration check being out of control limits and low chemical carrier recovery respectively.

10/11/93

APPENDIX C

DATA VALIDATION SUMMARY REPORT OUTLINE FORMAT

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EXAMPLE TABULAR SUMMARY OF VALIDATED DATA

Example Tabular Summary of Validated Data
Radiochemical Analysis Results

Parameter	HEIS# Date Site Cmts	EXAMPLE1 10-10-93 LOCATION1		EXAMPLE2 10-10-93 LOCATION2		EXAMPLE3 10-10-93 LOCATION3		EXAMPLE4 10-10-93 LOCATION4	
	Units	Result	Q	Result	Q	Result	Q	Result	Q
GROSS ALPHA	pCi/L	5.00	U	4.00	U	6.70	J	5.00	U
GROSS BETA	pCi/L	27.00		30.00		13.00		14.00	
URANIUM-234	pCi/L	0.20	U	0.22	J	0.59		0.45	
URANIUM-235	pCi/L	0.20	UR	0.20	U	0.20	UR	0.30	UR
URANIUM-238	pCi/L	0.20	U	0.20	J	0.44		0.26	J
PLUTONIUM-238	pCi/L	0.04	U	0.05	U	0.06	U	0.06	U
PLUTONIUM-239	pCi/L	0.03	U	0.03	U	0.04	U	0.02	U
AMERICIUM-241	pCi/L	0.09	U	0.03	U	0.03	U	0.10	UR
STRONTIUM-90	pCi/L	0.10	U	0.90	U	0.20	U	0.20	U
POTASSIUM-40	pCi/L	22.00		23.00		12.00		12.00	
IRON-59	pCi/L	0.40	U	0.40	U	0.30	U	0.10	U
CHROMIUM-51	pCi/L	2.00	U	2.00	U	2.00	U	0.80	U
COBALT-60	pCi/L	0.05	U	0.06	U	0.05	U	0.02	U
ZINC-65	pCi/L	0.20	U	0.20	U	0.10	U	0.06	U
RUTHENIUM-106	pCi/L	0.40	U	0.40	U	0.40	U	0.20	U
CESIUM-134	pCi/L	0.05	U	0.06	U	0.05	U	0.04	U
CESIUM-137	pCi/L	0.04	UJ	0.05	U	0.05	UJ	0.03	J
EUROPIUM-152	pCi/L	0.09	UJ	0.07	U	0.09	UJ	0.05	UJ
EUROPIUM-154	pCi/L	0.06	UJ	0.05	U	0.06	UJ	0.03	UJ
RADIUM-226	pCi/L	0.28		0.24		0.51		0.52	
THORIUM-228	pCi/L	0.55		0.46		1.10		0.82	
THORIUM-232	pCi/L	0.51		0.34		0.75		0.79	

EXAMPLE ANNOTATED LABORATORY SAMPLE REPORT FORM

RADIOCHEMICAL ANALYSIS DATA SHEET

SAMPLE NO.

EXAMPLE

Lab Name: WHC Contract Laboratory Contract: EXAMPLE

Lab Code: WHC001 Case No.: SAMPLE SAS No.: SAMPLE SDG No.: SAMPLE

Matrix: (soil/water) water

Lab Sample ID: EXAMPLE-1

Lab File ID: EXAMPLE-1

Date Received: 10/5/93

CONSTITUENT	CONCENTRATION UNITS (pCi/L)	Error	MDA	Q
Gross Alpha	3.12	1.3	2	— J
Gross Beta	6.5 ~~4.3~~	5.4	3	
Strontium-90	2.2	3.3	1	— R
Technetium-99	5	2	4	— R
Uranium-234	0.14	0.08	0.05	
Uranium-235	0.03	0.03	0.01	
Uranium-238	0.15	0.05	0.05	
Potassium-40	3.2	1.1	2	— J
Chromium-51	1.1	0.3	0.5	
Iron-59	4.5	1.2	0.5	
Cobalt-58	10		10	U
Cobalt-60	20		20	U
Ruthenium-103	100		100	~~U~~ UJ
Ruthenium-106	200		200	~~U~~ UJ
Cesium-134	3.14	1.4	1	
Cesium-137	5.2	3.4	3	
Europium-152	15		15	U
Europium-153	4		4	U
Europium-154	12		12	U

12/11/93

12/11/93

APPENDIX D

CALCULATIONS

APPENDIX D

Gross Alpha/Beta and Tritium

$$\frac{(A-B)\times C}{2.22\times E\times V} \qquad \text{D-1}$$

Where:
- A = gross counts per minute
- B = background counts per minute
- C = Activity of alpha fraction in beta channel (if for calculation of gross beta, otherwise substitute 1)
- 2.22 = conversion factor, dpm/pCi
- E = detector efficiency
- V = sample volume, liters or grams

Strontium (total)

$$\frac{A-B}{2.22\times E\times I\times D\times R\times V} \qquad \text{D-2}$$

Where:
- A = gross counts per minute
- B = background counts per minute
- 2.22 = conversion factor, dpm/pCi
- E = detector efficiency
- I = ingrowth correction factor
- R = carrier recovery factor
- D = strontium decay factor
- V = sample volume, liters or grams

Strontium-90 (corr. for Sr-89)

$$\frac{(A-B)}{2.22\times Y\times E\times I\times D\times R\times V} \qquad \text{D-3}$$

Where:
- A = gross counts per minute
- B = background counts per minute
- Y = Yttrium-90 yield factor
- 2.22 = conversion factor, dpm/pCi
- E = detector efficiency
- I = ingrowth correction factor
- R = Strontium-89 yield factor
- D = strontium decay factor
- V = sample volume, liters or grams

Technetium 99

$$\frac{A-B}{2.22 \times E \times R \times V} \qquad \text{D-4}$$

Where:
A = gross counts per minute
B = background counts per minute
2.22 = conversion factor, dpm/pCi
E = detector efficiency
R = carrier recovery factor
V = sample amount, liters or grams

Alpha Spectroscopy Tracer Recovery

$$\frac{A-B}{2.22 \times E \times T} \times 100 \qquad \text{D-5}$$

Where:
A = gross counts per minute of tracer
B = background counts per minute for tracer
2.22 = conversion from dpm/pCi
E = detector efficiency
T = activity (pCi) of tracer added to sample can be determined by taking dpm of tracer added divided by 2.22

Alpha Spectroscopy Isotope Concentration

$$\frac{A-B}{2.22 \times E \times R \times V} \qquad \text{D-6}$$

Where:
A = gross counts per minute for isotope
B = background counts per minute for detector
2.22 = conversion from dpm/pCi
E = detector efficiency
R = tracer recovery factor (calculated above)
V = sample amount, liters or grams

Gamma Spectroscopy Isotope Concentration

$$\frac{A \times D}{2.22 \times B \times E \times V \times T} \qquad \text{D-7}$$

Where:
A = peak area for isotope
D = decay factor for isotope
2.22 = conversion from dpm/pCi
B = abundance factor for isotope
E = efficiency factor for isotope
V = sample amount, liters or grams
T = live time (minutes)

Total Uranium by Laser Fluorometry

$$\frac{(WF-I)\times R\times D}{WU-WF} \qquad \text{D-8}$$

Where:
WF = sample reading with Fluran
I = initial sample reading
R = concentration of uranium standard after dilution with sample (μg/L)
D = dilution factor
WU = sample reading with uranium standard

Radium-226 by Radon Emanation

$$D=\frac{C}{2.22\times E\times V}\times\frac{1}{1-e^{-\lambda t_1}}\times\frac{1}{e^{-\lambda t_2}}\times\frac{t_3}{1-e^{-\lambda t_3}} \qquad \text{D-9}$$

where:

C = net count rate, cpm,
E = calibration constant of the de-emanation system and the scintillation cell in counts per minutes/disintegrations per minute of radon-222,
V = sample aliquot in liters,
t_1 = the elapsed time in days between the first and second de-emanations and λ is the decay constant for radon-222 (0.181 d^{-1}),
t_2 = the time interval in hours between the second de-emanation and counting and λ is the decay constant of radon-222 (0.00755 hr^{-1}),
t_3 = the counting time in minutes and λ is the decay constant of radon-222 (1.26×10^{-4} min^{-1}), and
2.22 = the conversion factor from dpm/pCi.

Minimum Detectable Activity (MDA)

$$\frac{4.66\times\sqrt{B\times T}}{2.22\times E\times I\times R\times D\times V\times Y\times T} \qquad \text{D-10}$$

Where:
B = background counts per minute (cpm) or the reported standard deviation of the background (S) cpm
T = counting time for associated sample
2.22 = conversion from dpm/pCi
E = detector efficiency
I = ingrowth correction factor (if applicable or 1)
R = carrier recovery factor (if applicable or 1)
D = decay factor (if applicable or 1)
Y = chemical yield factor (if applicable or 1)
V = sample volume, liters or grams

Relative Percent Difference (RPD)

$$\frac{|S-D|}{(S+D)/2} \times 100 \qquad \text{D-11}$$

Where: S = sample result
D = duplicate sample result

Attachment 8

Unit Managers Meeting
303-K STORAGE FACILITY
2440 STEVENS CENTER, RM 1200
Richland, Washington

Meeting Held September 23, 1994
From 8:00 am to 9:30 am

TITLE - DATA VALIDATION PROCEDURES FOR RADIOCHEMICAL ANALYSIS
(WHC-SD-EN-SPP-002)