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CP2-ES-5102/FR2

Radiochemical Analysis Data Verification and Validation for the Paducah Gaseous Diffusion Plant, Paducah, Kentucky

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Radiochemical Analysis Data Verification and Validation for the Paducah Gaseous Diffusion Plant, Paducah, Kentucky

Date Issued—December 2025

U.S. DEPARTMENT OF ENERGY
Office of Environmental Management

Prepared by
FOUR RIVERS NUCLEAR PARTNERSHIP, LLC,
managing the
Deactivation and Remediation Project at the
Paducah Gaseous Diffusion Plant
under Contract DE-EM0004895

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APPROVALS

Radiochemical Analysis
Data Verification and Validation for the
Paducah Gaseous Diffusion Plant,
Paducah, Kentucky

CP2-ES-5102/FR2

December 2025

Approved by:

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(Affiliate)

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Director, Technical Services

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REVISION/CHANGE LOG				
Revision/ Change Letter	**Description of Changes**	**Pages Affected**	**Date of Revision/ Change**	**Approved By (signature on file)**
FR0	Bluesheet	ALL	11/14/2017	Signature on file
FR1	Non-Intent Changes for Bluesheet Incorporation	ALL	12/13/2017	Signature on file
FR1	In accordance with the Corrective Action Plan for CAPA CA-00003116, Action Item AI-0004709, the periodic review date for this procedure has been extended to December 13, 2022.	1	7/6/2021	Signature on file
FR1A	Periodic Review has been completed with no changes identified in procedure technical content. Non-intent changes have been incorporated per CP3-NS-2001. Date for review cycle has been reset.	ALL	12/13/2022	Signature on file
FR2	General revision to update plan according to DoD/DOE QSM 6.0 requirements and other applicable professional guidance to address CA-005368, AI-0008630.	ALL	12/16/2025	Signature on file

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CONTENTS

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TABLES

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ACRONYMS

CCV	continuing calibration verification
COC	chain-of-custody
DER	duplicate error ratio
DoD	U.S. Department of Defense
DOE	U.S. Department of Energy
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
FWHM	full width half maximum
IAEA	International Atomic Energy Agency
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MB	method blank
MD	mean difference
MDA	minimum detectable activity
MDC	minimum detectable concentration
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable
NIST	National Institute of Standards and Technology
QAPP	quality assurance project plan
QC	quality control
QSM	quality systems manual
RT	retention time
RPD	relative percent difference
SAEP	sampling analysis and event plan
SAP	sampling and analysis plan
SDG	sample delivery group
SMO	sample management office
SOW	statement of work
TPU	total propagated uncertainty
%R	percent recovery

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DEFINITIONS

NOTE 1: Data validation code definitions are listed in Appendix A.

NOTE 2: In this plan, the words "shall" and "must" are used to denote a requirement; the word "should" is used to denote a recommendation; and the word "may" is used to denote permission (neither a requirement nor a recommendation). In conformance to this plan, all steps shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification must be documented for deviations from recommendations.

Activity—Activity is defined as the number of spontaneous nuclear transformations that occur in a quantity of a radioactive nuclide per unit time.

Affected Sample Result—A sample result is affected when it is significantly influenced by a quality deficiency and is qualified accordingly through analytical data validation.

Batch—A batch is a group of samples prepared at the same time in the same location using the same method, not to exceed 20 samples of similar matrix.

Carrier—A carrier is a stable element/compound introduced into the sample preparation/analysis process that will behave chemically like the analyte isotope(s). It is by virtue of this chemical similarity that the carrier will "carry" the analyte isotope(s) through the sample preparation/analysis process.

Case—A finite, usually predetermined number of samples that has been collected over a given time period from a particular site. A case consists of one or more sample delivery groups.

Certified Radioactivity Standard Source—A certified radioactivity standard source is a calibrated radioactivity source, with stated accuracy, whose calibration is certified by the source supplier as traceable to a known originator.

Chain-of-Custody—The history of the transfer of samples from the time of sample acquisition through archival and disposal of samples. Chain-of-custody documentation is required as evidence of sample integrity.

Continuing Calibration Verification—A standard solution analyzed at a specified frequency during an analytical run to assure the continued validity of the calibration curve.

Correctable Problem—Correctable problems are deficiencies within data packages that may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation. Correctable problems that are revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory. Correctable problems that are revealed during validation are those deficiencies with analyses that can be solved either by a second preparation and/or by analysis of a sample.

Counting Batch—A counting batch is a group of sample aliquots analyzed together on the same instrument detector system.

Counting Uncertainty—Counting uncertainty is the component of measurement uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of

observed counts). Units for counting uncertainty are the same as for the reported result, minimum detectable activity, and total propagated uncertainty. Counting uncertainty may also be referenced as counting error.

Data Quality Objective—Data quality objectives are qualitative and quantitative statements derived from the outputs of each step of the data quality objective process that specify the study objectives, domain, limitations, the most appropriate type of data to collect, and specify the levels of decision error that will be acceptable for the decision.

Data Quality Objectives Process—The data quality objective process is a quality management tool based on the scientific method and developed by the U.S. Environmental Protection Agency to facilitate the planning of environmental data collection activities. The data quality objective process enables planners to focus their planning efforts by specifying the use of the data (the decision), the decision criteria (action level), and the decision maker's acceptable decision error rates.

Data Validation—Data validation is a systematic process, performed independently from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

Data Verification—Data verification is a systematic process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract that is performed either by the data generator or by an entity independent to the data generator.

Decision Level—The minimum measured value (e.g., of the instrument signal or the analyte concentration) required to give confidence that a positive (nonzero) amount of analyte is present in the material analyzed. The decision level is sometimes called the critical level or critical value. It is the quantity of analyte at or above which an a posteriori decision is made that a positive quantity of the analyte is present.

Holding Time—Holding time, as described in this plan, is defined as the period of time between sample collection and sample activity determination.

Initial Calibration—Initial calibration, as described in this plan, is defined as the standardization of a gas chromatography/mass spectrometry instrument against a traceable standard of known identity and quantity. This standardization prevails until such a time that analytical conditions are deemed out of acceptable control limits.

Laboratory Control Sample—The laboratory control sample is a control sample of a known composition. Aqueous and solid laboratory control samples are analyzed using the same preparation, reagents, and method employed for field samples.

Laboratory Duplicate—The laboratory duplicate is a randomly chosen split of an analytical sample into two aliquots prior to sample preparation. The purpose of a laboratory duplicate is to monitor the precision of the analytical method.

Matrix Spike—The matrix spike is a split of a field-originating analytical sample in which one half of the split is spiked with a known amount of radionuclide of interest prior to sample preparation. The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation.

Method Blank—The method blank is a laboratory-generated sample of the same matrix as the analytical samples, but in absence of the analyte of interest. The purpose of a method blank is to monitor the presence of contamination of the analyte of interest in the sample preparation and analysis processes.

Minimum Detectable Activity—The minimum detectable activity (MDA) is an estimate of the smallest true activity that ensures a specified high confidence, (1–β), of detection above the decision level, and a low probability β of false negatives below the decision level. For radiometric methods, β is often set to 0.05. MDA is the measure of the detection capability of a measurement process, and as such, it is an *a priori* concept. It may be used in the selection of methods to meet specified measurement quality objectives. Laboratories may also calculate a "sample-specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability; however, the MDA shall never be used instead of the decision level as a detection threshold. For this plan, the terms MDA and minimum detectable concentration (MDC) are equivalent.

Noncorrectable Problem—Noncorrectable problems are deficiencies within a data package that preclude the evaluation of data quality by predefined criteria. Noncorrectable problems may be revealed during both data verification and data validation.

Practical Quantitation Limit—The practical quantitation limit is defined as the lowest concentration of a contaminant that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The practical quantitation limit is typically several times higher than the method detection limit.

Preparation Batch—A preparation batch is a group of sample aliquots prepared together at the same time using the same method and related to the same quality control samples.

Relative Percent Difference—Relative percent difference is the measure of precision between two values, defined as the absolute value of the difference between two values divided by the mean of the two values.

Relative Standard Deviation—Relative standard deviation is the measure of precision between multiple values, defined as the standard deviation of multiple values divided by the mean of the values.

Required Detection Limit—The required detection limit is a contractually specified detection limit that, under typical analytical circumstances, should be achievable.

Reporting Limit—The reporting limit is a contractually specified detection limit that, under typical analytical circumstances, should be achievable.

Sample Delivery Group—A sample delivery group is defined by one of the following, whichever occurs first: (1) case of field samples; (2) each 20 field samples within a case; (3) each 14-day calendar period during which field samples in a case are received, beginning with receipt of the first sample in the sample delivery group.

Sample Quantitation Limit—Sample quantitation limits are detection limits based on the required detection limit, which have been modified due to deviations from analytical method specifications, such as sample weight and extract volume or due to dilution or percent moisture.

Sample Result—A sample result, as described in this plan, is a numeric denotation of the concentration, amount, or activity of a specific analytical parameter uniquely associated with an aliquot of environmental media.

Statement of Work—The validation statement of work is a document prepared to function as the mechanism by which validation requirements are communicated from the project to the validation organization.

Total Propagated Uncertainty—Total propagated uncertainty (TPU) is an estimate of the measurement uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as combined standard uncertainty or, in some older references, as the total propagated error. The Paducah Gaseous Diffusion Plant requests laboratories to report TPU at 2 sigma (2σ).

Tracer—Tracers chemically mimic but do not interfere with the target analyte through radiochemical separations. Isotopic tracers are typically radioactive materials (e.g., plutonium-242, strontium-85). They are added to samples to determine the overall chemical yield for the analytical preparation steps.

Turnaround Time—Turnaround time is contractually specified as the amount of time that elapses between laboratory receipt of the raw samples and subsequent data receipt by the client.

Validation Code—A validation code is an alphabetic character that is physically or electronically associated with a discrete sample result during validation due to a data quality deficiency, which provides guidance in data usability.

Validation Statement of Work—The validation statement of work is a document prepared to function as the mechanism by which data validation implementation requirements are communicated from the sample management office to the validation organization.

1. INTRODUCTION

1.1 PURPOSE AND SCOPE

This plan provides guidance for the verification and validation of radiochemical analysis laboratory data performed by an external party. For the purpose of this guidance, external parties are defined as organizations (including governmental entities, contractors, or vendors) that conduct analytical data review, verification, and validation activities, and that are not part of the immediate laboratory that generates the subject analytical data (but are part of the overall project-specific data review process).

This document focuses on data generated through methods and instrumentation that detect radioactivity (i.e., alpha spectrometry, gamma spectrometry, gas flow proportional counter, scintillation counter). This plan is not applicable to mass spectrometric or fluorometric methodologies. When applicable, this plan incorporates requirements that are defined in the *U.S. Department of Defense (DoD) and U.S. Department of Energy (DOE) Quality Systems Manual (QSM) for Environmental Laboratories Version 6.0*; however, data validators should reference the most current version of the DoD/DOE QSM when validating data (DoD and DOE 2023). In the absence of specific guidance, data validators are advised to seek guidance in the specific method employed and/or from other industry standards. Examples include U.S. Environmental Protection Agency (EPA) Regional Data Validation Guidance and subject matter experts within the industry.

1.2 APPLICABILITY

Data verification and validation is a systematic process, which is performed externally from the data generator that applies a defined set of performance-based criteria to a body of data that can result in the application of validation codes to the data. The project team, with input as needed from a quality assurance specialist and/or representative of the sample management office (SMO), shall develop a data validation strategy based on inputs identified through the data quality objective (DQO) process. The project-specific sampling and analysis plan (SAP), sampling analysis and event plan (SAEP), or quality assurance project plan (QAPP) will define the DQOs and framework for performing data validation.

Data verification is the process of checking data for completeness, correctness, consistency, and contract compliance. These requirements are contained in the analytical laboratory statement of work (SOW) and/or project-specific planning documents (e.g., SAP, SAEP, QAPP). The data verification process compares the laboratory data package to requirements associated with the project. The data verification process can identify deficiencies in the laboratory data package that can be addressed by obtaining additional information from the laboratory.

Data validation is the process of examining a laboratory data package to provide a level of confidence in the reported analyte's identification, concentration (including detectability), and associated measurement uncertainty. The data validation process begins with a review of the laboratory data package to screen the areas of strength and weakness of the data. The data validation process continues with assessing the data against standardized procedures and criteria to confirm the presence or absence of an analyte and to evaluate the uncertainty of the quantification for the analyte. Each data point is then qualified as to its integrity and dependability in the context of the project requirements based on all available laboratory data.

2. RESPONSIBILITIES

Table 1 summarizes the responsibilities of data validator and the SMO.

Table 1. Responsibilities for Data Validator and SMO

Performer	Responsibilities
Data Validator	• Determines if all required information is presented in the laboratory data package. • Makes objective judgments and decisions about the data quality and defensibility. • Assigns data validation codes to the results. The data validation codes indicate the validity and usability of the data and the limitations on its end use. • Produces a data validation report.
SMO	• Reviews each data validation report. • Adds data validation codes to data in the project environmental measurements system. • Distributes the data validation report to the appropriate personnel.

3. GENERAL INFORMATION

3.1 LEVELS OF LABORATORY DATA DELIVERABLES

Laboratory data deliverables consist of a combination of forms and raw data. Depending upon the required laboratory report elements included, the deliverable can range from a Level I to a Level IV laboratory data package. Level IV laboratory data packages are typically used for data validation purposes. The elements included in a laboratory data package for each level are provided in Table 2.

Table 2. Required Laboratory Report Elements

Laboratory Report Elements*	Level I	Level II	Level III	Level IV
Cover/Signature Page/Executive Summary	✓	✓	✓	✓
Table of Contents	✓	✓	✓	✓
Laboratory Report Narrative	✓	✓	✓	✓
Method Summary		✓	✓	✓
Sample Summary/Sample Data Sheets	✓	✓	✓	✓
Shipping and Receiving Documents	✓	✓	✓	✓

Table 2. Required Laboratory Report Elements (Continued)

Laboratory Report Elements*	Level I	Level II	Level III	Level IV
Client Chain-of-Custody (COC)	✓	✓	✓	✓
Sample Receipt Checklist	✓	✓	✓	✓
Interlab COC (where applicable)	✓	✓	✓	✓
Subcontract Laboratory COC (if required)	✓	✓	✓	✓
Glossary of Abbreviations and Laboratory Definitions	✓	✓	✓	✓
Quality Control (QC) Association Summary/Sample Traceability	✓	✓	✓	✓
Analysis Run Log			✓	✓
Surrogate and/or Tracer and Carrier Recovery Report	✓	✓	✓	✓
Method Blank (MB) Reports		✓	✓	✓
Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) Summary		✓	✓	✓
Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Summary		✓	✓	✓
Duplicate Sample Summary		✓	✓	✓
Instrument Performance Check Summary			✓	✓
Calibration Data			✓	✓
Internal Standard Area and Retention Time (RT) Summary			✓	✓
Continuing Calibration [Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV)] Summary Report			✓	✓
Instrument Blank Report			✓	✓
Detection Limits Summary			✓	✓
GC Dual Column Identification Summary			✓	✓
Linear Ranges			✓	✓
Preparation Batch Log			✓	✓
Interference Check Standard Summary			✓	✓
Serial Dilution Summary			✓	✓
Cleanup Log			✓	✓
Standard/Reagent Traceability Log			✓	✓
Accreditation/Certification Summary			✓	✓
Raw Sample Data			✓	✓
Raw QC Data			✓	✓
Manual Integration Summary			✓	✓

*Report elements listed represent common elements. The laboratory may provide more or less information as required by the method being analyzed. For example, those wet chemistry methods with no true calibration information will not have calibration forms included in the laboratory data package.

3.2 STAGES OF VALIDATION

For the purposes of this plan, the following terminology is recommended for use to describe the stages (extent) and processes that are used to validate laboratory analytical data packages, whether the validation is performed by a manual process, electronic process, or combination of both.

NOTE: The following list of required activities per each stage of validation is not considered an "all-inclusive" list or applicable to every method that is validated.

- **Stage 1 Validation**: A verification and validation based only on completeness and compliance of sample receipt condition checks. Client sample IDs and target analytes are verified against the COCs for completeness; sample conditions upon arrival at laboratory are noted; sample preservation was

appropriate and verified by the laboratory; holding times were met; concentrations and units were appropriate; and trip blanks, field blanks, equipment blanks, and field duplicates met the project requirements for frequency and field QC.

- **Stage 2A Validation**: A verification and validation based on completeness and compliance checks of sample receipt conditions and **ONLY** sample-related QC results. MBs, LCSs, MSs, laboratory duplicates (including LCSD and MSD), surrogates (organics), serial dilutions, post-digestion spikes (as appropriate to the method), and any preparatory batch cleanup QC to assure that project requirements for analyte spike list, frequency, and QC limits are met.

- **Stage 2B Validation**: A verification and validation based on completeness and compliance checks of sample receipt conditions and **BOTH** sample-related and instrument-related QC results.

- **Stage 3 Validation**: A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, **AND** recalculation checks.

- **Stage 4 Validation**: A verification and validation based on completeness and compliance of sample receipt conditions, both sample-related and instrument-related QC, recalculation checks, **AND** the review of actual instrument outputs.

The stage of validation required is generally defined at the program or project level. Validation parameters to be reviewed depending on the stage of validation can include instrument calibrations, calibration verification checks, QC sample results, analytical yields, holding times, and sample preservation. It is not the role of data validation to determine if project goals are met or to provide the decisions to be made. Data validation provides the overall appraisal of a data set, and the project team should use this appraisal along with their own judgment to make their own decisions.

3.3 DATA ASSESSMENT REVIEW

The data assessment review includes the following.

- Data verification/contractual screen
- Data validation (if requested)
- Data assessment
- Data usability assessment

The data assessment review is comparable to a Stage 1 and Stage 2A validation (depending on analyte and method). As required by project-specific requirements, a Stage 2B, Stage 3, or Stage 4 validation of the data package **MAY** be requested. See CP3-ES-5003, *Quality Assured Data*, for more information about the data assessment review process.

3.3.1 Data Verification/Contractual Screen

Data verification is the first step of the data assessment review process. The preferred method for performing verification is electronic. Verification criteria are documented using CP3-ES-5003-F01, "Data Assessment Review Checklist and Comment Form," and CP3-ES-5003-F05, "Data Verification/Validation Checklist" (if Stage 2B, Stage 3, or Stage 4 data validation is required). Data verification is performed on 100% of data.

3.3.2 Data Validation

Data validation (if requested) follows data verification in the data assessment review process when requested by the project team. Stage 3 and Stage 4 validations must be performed by a third party. Third-party data validation is defined as validation that is performed by persons independent from the sampling, laboratory, and decision making for the project (i.e., not the project reviewer). Data validation is documented in a formal deliverable from the data validator. The stage and frequency that are chosen for validation are based on project requirements and the following considerations.

- Regulatory drivers/requirements
- End-user of data
- Future applicability of the data (other users such as regulatory agencies, risk assessment personnel, internal users, etc.)
- Legal ramifications and defensibility of data
- Confidence in laboratory (DOE Consolidated Audit Program-approved laboratory)

The project team determines if the data set requires validation. The project team also determines the stage and frequency of data validation.

When data validation is requested by the project, a validation SOW is prepared by the SMO to communicate data verification and validation requirements to the external party performing the data validation. Along with the validation SOW, full copies of the laboratory data packages, as well as an electronic data deliverable in the form of a Microsoft Excel file, are sent to the data validators performing the validation. CP3-ES-5003-F05 is provided to the validator from the SMO and must be completed for every laboratory sample delivery group (SDG) being validated.

3.3.3 Data Assessment

Data assessment follows data verification and data validation (if requested) in the data assessment review process. Data assessment is performed by data reviewers who have been trained to evaluate laboratory quality assurance/QC requirements. Data assessment is performed on 100% of data.

3.3.4 Data Usability Assessment

Data usability assessment is the last review step of the data assessment review process prior to release of the data from the project team. Data usability assessment is an integration of all information collected about a result. Data verification and validation can ensure that analyses are correct; however, data usability assessment must be performed to evaluate the data usability. This includes a review of the data itself, the results of all previous reviews of the data, checking data for trends, and an evaluation against the intended purpose for data collected. Data usability assessment must be performed for all data collection activities and documented using CP3-ES-5003-F01. Data usability assessment is required prior to use of the data or data release into the final data repository (i.e., Oak Ridge Environmental Information System). Data usability assessment is performed on 100% of data.

4. DATA VERIFICATION AND VALIDATION INSTRUCTIONS

NOTE 1: The data verifier and data validator may be the same individual. CP3-ES-5003-F05 is only completed for Stage 2B, Stage 3, and Stage 4 validations. Appendix B has qualification tables for multiple quality deficiencies.

NOTE 2: **If** data reviewers use this plan as a guide for qualifying data during data assessment, **then** they should apply equivalent data assessment codes in place of the data validation codes referenced in this plan.

NOTE 3: For the purpose of this plan, the terms minimum detectable activity (MDA) and minimum detectable concentration (MDC) are equivalent.

4.1 SAMPLE RECEIPT CONDITIONS

4.1.1 Chain-of-Custody

The COC form provides the basis for the traceability of project samples, by documenting the sample from its origin through all steps of the sampling, sample handling, and analysis process. The COC serves as documentation of the sample possession from collection through disposal to ensure that sample representativeness is maintained prior to analysis. By documenting personal accountability for samples, the COC is used to ensure that proper custody has been maintained from the time a sample is generated through its final disposition (cradle to grave). Any break in custody, as demonstrated by the series of signatures denoting sample holders, could jeopardize the legal and/or technical defensibility of associated sample data.

While data verification/validation cannot replicate the custody history of a sample (i.e., fully assure that the sample truly has been in custody from the field to the final result), an evaluation of field notes from sample data forms, laboratory records, and the COCs provide the best available indicator of sample traceability. A sample is defined as being under proper custody if any of the following conditions are met:

- The sample is within the possession of an authorized person (e.g., field personnel, laboratory personnel);
- The sample is within view of an authorized person;
- The sample was in an authorized person's possession and then was secured to prevent tampering; or
- The sample is placed in a designated secure area.

NOTE: Verification of sample documentation includes result report header checks for accuracy from the COC. **If** sample identity is in question, **then** every attempt should be made to verify the identity of each sample. When custody problems cannot be resolved, they will affect the defensibility of the sample.

4.1.1.1 Data verification

Trace custody of all samples in the reporting batch from field sampling through receipt at the laboratory by reviewing the COC forms. **If** the information is missing, **then** the data verifier will seek to obtain field documentation from the sampler or laboratory to determine if the omission affects sample integrity. **If** there is a break in the signature chain on the COC, or other omissions in the custody record (e.g., date of sample

collection, date of transfer to the laboratory), **then** indicate the problem on the data verification/validation checklist.

4.1.1.2 Data validation

If sample data are **NOT** traceable through signature records on COCs **or** other sample record information demonstrating custody (e.g., laboratory logbooks and/or sample data forms) **CANNOT** establish custody history, **then** the data validator **shall** apply an "R" validation code to associated results.

4.1.2 Holding Time, Temperature, and Sample Preservation

Holding times have been established by EPA to define the maximum period of time during which a sample remains representative of its sampling location. Holding times begin when a sample is collected in the field and are measured by determining the elapsed time from collection through extraction (when applicable) and/or analysis. **If** the reported data is the result of a dilution, reinjection, or re-extraction and analysis, **then** the result **must** have been generated within the prescribed holding time in order for the result to be considered definitive.

4.1.2.1 Deliverables

The following are deliverables.

- Sample data forms
- COCs
- Laboratory sample receipt checklist
- Laboratory reports and/or raw data containing the following: dates of collection, preparation, and analysis for all samples, dilutions, and re-extractions

4.1.2.2 Criteria

Table 3 provides current industry-accepted standards for sample preservation and hold times for radionuclide parameters. In all cases, the data verifier or validator shall always follow the most current methodology guidance for sample holding time, temperature, and preservation requirements.

Table 3. Holding Times and Sample Preservation Criteria

Parameters	Matrix	Preservatives	Holding Times[a]
Alpha Isotopes (Americium, Neptunium, Plutonium, Thorium, Uranium)	Liquid	pH < 2 with nitric acid or hydrochloric acid	6 months
Alpha Isotopes (Americium, Neptunium, Plutonium, Thorium, Uranium)	Solid	None	6 months
Carbon-14	Liquid & Solid	None	6 months

Table 3. Holding Times and Sample Preservation Criteria (Continued)

Parameters	Matrix	Preservatives	Holding Times[a]
Gamma Isotopes (Cesium-137, Cobalt-60, Potassium-40, etc.)	Liquid	pH < 2 with nitric acid or hydrochloric acid	6 months
Gamma Isotopes (Cesium-137, Cobalt-60, Potassium-40, etc.)	Solid	None	6 months
Gross Alpha & Beta	Liquid	pH < 2 with nitric acid or hydrochloric acid	6 months
Gross Alpha & Beta	Solid	None	6 months
Iodine-129	Liquid & Solid	None	6 months
Iodine-131[b]	Liquid	None	8 days
Radium-226	Liquid	pH < 2 with nitric acid or hydrochloric acid	6 months
Radium-226	Solid	None	6 months
Radium-228	Liquid	pH < 2 with nitric acid or hydrochloric acid	6 months
Radium-228	Solid	None	6 months
Radon-222	Liquid	None, zero headspace	4 days
Strontium-89/90[c]	Liquid	pH < 2 with nitric acid or hydrochloric acid	6 months
Strontium-89/90[c]	Solid	None	6 months
Technetium-99	Liquid	pH < 2 with nitric acid or hydrochloric acid	6 months
Technetium-99	Solid	None	6 months
Tritium	Liquid & Solid	None	6 months

[a] Holding times of short-lived radionuclides may need to be adjusted to be significantly less than 6 months on a case-by-case basis, depending on the half-life of the isotope, its detectability, and its initial concentration.
[b] Half-life of Iodine-131 is 8.04 days.
[c] Half-life of Strontium-90 is 50.5 days.

4.1.2.3 Data verification

Verify the presence of the pertinent COC forms in laboratory data packages. **If** COC forms are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** missing information cannot be obtained or reconstructed from field notes, COC forms, etc., **then** the data verifier will note the omitted information on the data verification/validation checklist as a noncorrectable problem.

4.1.2.4 Data validation

Holding times

Review the data verification/validation checklist for holding times to confirm that all holding times have been met. Holding times that are listed in hours from collection to analysis always will be calculated using the time collected to ensure that the holding time in hours has not lapsed. Holding times that are listed in days will be calculated using dates only. The data validator shall review COC forms, field notes, laboratory report forms, and laboratory raw data, as necessary, to determine the elapsed time from sample collection to sample analysis for deviations identified on the data verification/validation checklist.

If the elapsed time falls within the prescribed holding time, **then NO** actions will be taken and no validation code applied.

If the holding time is exceeded, apply validation codes to data as follows.

- **If** the holding time is exceeded by a factor of < 2, **then** apply a "J" validation code to detected results and apply a "UJ" validation code to nondetected results.

- **If** the holding time is grossly exceeded by a factor ≥ 2, **then** apply a "J" validation code to detected results and apply an "R" validation code to nondetected results.

Preservation

Review the laboratory receiving records to determine **if** samples were received with proper preservation. **If** records demonstrate that samples were received by the laboratory with the proper preservation, **then NO** action will be taken and **NO** validation code will be applied.

If preservation is incorrect, **then** apply validation codes to data as follows.

- **If** samples are received without the proper preservation and **if** the holding time is exceeded by a factor of < 2, **then** apply a "J" validation code to detected results and apply a "UJ" validation code to nondetected results.

- **If** samples are received without the proper preservation and **if** the holding time is exceeded by a factor of ≥ 2, **then** apply a "J" validation code to detected results and apply an "R" validation code to nondetected results.

- **If** samples are received without the proper preservation, **then** apply a "J" validation code to detected results and apply a "UJ" validation code to nondetected results.

Table 4 summarizes data validation qualification guidance for samples with holding time exceedances and/or preservation issues.

Table 4. Holding Times and Preservation Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	Nondetects
1. Samples extracted and/or analyzed outside the appropriate holding time and not grossly exceeded (i.e., exceedance < 2× holding time)	J	UJ
2. Samples extracted and/or analyzed outside grossly exceed holding time. (i.e., exceedance ≥ 2× holding time)	J	R
3. Samples preserved improperly.	J	UJ

4.2 SAMPLE-RELATED QUALITY CONTROL RESULTS

4.2.1 Blanks

Blank analyses serve to determine the existence and magnitude of contamination resulting from laboratory or field activities. It has been the EPA Region 4 data validation policy to evaluate trip blanks, field blanks, and equipment rinsate blanks as part of the validation process, but not to apply validation codes to the data based on field sample results.

Method Blank

An MB is used to assess the level of contamination that is introduced to the analytical samples throughout the sample preparation and analysis process. **If** contamination is found in any blank, **then** all associated data must be carefully evaluated to determine whether there is a systemic problem affecting greater than one sample or if the contamination is an isolated occurrence.

Field Blank

The project team may elect to collect and analyze a field blank to evaluate the existence and magnitude of contamination that may arise as a result of field-level activities. The field blank provides an indication of ambient conditions during the sampling activities, as well as an indication that the source of decontamination water is free of targeted analytes.

Equipment Rinsate Blank

The equipment rinsate blank provides an indication as to whether nondedicated sampling equipment has been properly decontaminated, and what, if any, carryover may arise between sampled locations.

4.2.1.1 Deliverables

The following are deliverables.

- MB report for each MB
- Raw data (required for confirmation)

4.2.1.2 Frequency

The MB **must** be analyzed at a frequency of one per preparatory batch.

For alpha spectrometry, MB matrices **shall** be consistent with the associated samples (e.g., radon-free distilled or deionized water, representative solid material, or physically and chemically identical filter media).

For gamma spectrometry, MB matrices and geometry **shall** be consistent with the associated samples.

4.2.1.3 Criteria

Blank count time **shall** be equal to or longer than associated sample count time.

NO detectable target radionuclide activity should be found in the blank [i.e., the activity should be less than the total propagated uncertainty (TPU) and its MDA].

4.2.1.4 Data verification

Verify the presence of required reporting forms. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem. Apply a "B07" validation reason code to the affected data if a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

4.2.1.5 Data validation

Verify that the measured MB activity is less than its MDA.

If the MB is a nondetect (MB < MDA), **or** the MB is a detect (MB ≥ MDA) but the associated sample results are nondetects, **then** no qualification of the data is necessary.

If both the MB and sample results are greater than their respective MDAs, **then** perform the following mathematical test to calculate mean difference (MD) to determine the significance of the contamination on the samples. This test is the standard statistical method of assessing differences between radioactivity measurements and determining the significance of those differences. MD calculation **shall NOT** be performed if the MB result has been subtracted from the sample result.

$$MD = \frac{|S - B|}{\sqrt{\left(\frac{1}{2}\sigma_S{}^2 + \frac{1}{2}\sigma_B{}^2\right)}}$$

MD = mean difference
S = the sample result (as pCi/g or pCi/L)
B = the blank sample result (as pCi/g or pCi/L)
σ_S = the associated TPU (2σ) of the sample result
σ_B = the associated TPU (2σ) of the blank sample result

Determine the magnitude of the contamination interference by performing the following mathematical test to determine the difference factor:

$$Difference \quad Factor = \frac{Sample \quad Activity}{Blank \quad Activity}$$

A difference factor that is > 10 indicates that the blank activity is insignificant in relation to the sample results. A difference factor that is < 10 indicates that the blank activity could affect the sample results.

NOTE: An MD > 3 demonstrates that the contaminant had no significant effect on the sample results [i.e., the difference is great enough that there is no statistical overlap of results at the 3σ (99.7%) confidence interval]. The typical statistical values for MD are 1.96 (at the 95% confidence interval) and 2.58 (for the 99% confidence interval); however, this plan has set the upper confidence interval at 99.7% (MD =3) to provide additional assurance that the difference between blank and sample results is well above any possible statistical concern. There are cases where the MD > 3 and the contaminant contribution can be significant. Such cases occur when there are conspicuous amounts of contamination and/or the measured sample and blank uncertainties are small (low). To prevent the assignment of an erroneous data validation code for such cases, the difference factor is evaluated.

Determine if the sample result is a false positive due to either instrument background fluctuations or interferences from other radionuclides or radionuclide quanta (gamma rays and alpha particles).

NOTE: For example, in gamma spectrometry, one of the common interferences is with radium-226 and uranium-235. In alpha spectrometry, one of the common interferences is the thorium-229 tracer peak tailing into the thorium-230 energy region of interest.

For Level IV validation only, verify that the aliquot size, volume/mass of reagents, dilution, and counting times were the same as that of the samples.

If an MB was **NOT** analyzed with reported samples or analyzed of a different matrix than the reported samples, **then** apply an "R" validation code to detected results. No qualification is necessary for nondetected results.

If both the MB **and** sample results are detected **and** the MD is > 3 **and** the difference factor is > 10, **then** no qualification is necessary for detected and nondetected results.

If the MB **and** the sample results are detected **and** the MD is > 2 **and** the difference factor is < 10, **then** apply a "J" validation code to detected results. No qualification is necessary for nondetected results.

NOTE: MD > 3 may demonstrate that the contaminant had an effect on the sample results. MDs between 2 and 3 demonstrate that the contaminant had an effect on the sample results (i.e., the blank and sample result can statistically overlap at the 2σ to 3σ confidence interval).

If the MB **and** the sample results are detected **and** the MD ≥ 0 **and** MD is ≤ 2 **and** the difference factor is < 10, **then** apply a "UJ" validation code to detected results. No qualification is necessary for nondetected results.

NOTE: MDs between 0 and 2 demonstrate that the contaminant had a significant effect on the sample results (i.e., the blank and sample result can statistically overlap at the 1σ to 2σ confidence interval).

If the MB result is negative **and** the absolute value of the MB result is greater than the MDA, **then** all associated samples must be carefully examined as an indication of improper background subtraction.

- **If** sample results are greater than or equal to the MDA **but** are < 5× the absolute value of the negative MB result, **then** apply a "J" validation code to detected results.

- **If** sample results are less than the MDA **and** < 5× the absolute value of the negative MB result, **then** apply a "UJ" validation code to nondetected results.

Table 5 summarizes data validation qualification guidance for issues with blanks.

Table 5. Blanks Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	**Nondetects**
1. MB **NOT** analyzed.	R	Not applicable (N/A)
2. MB **NOT** the same matrix as the samples.	R	N/A
3. MB **and** sample result ≥ MDA, MD > 3, **and** difference factor > 10.	N/A	N/A
4. MB **and** sample result ≥ MDA, MD > 2, **and** difference factor < 10.	J	N/A
5. MB **and** sample result ≥ MDA, MD ≥ 0 **but** ≤ 2, **and** difference factor < 10.	UJ	N/A
6. Sample results ≥ MDA **but** < 5× the absolute value of the negative MB result.	J	N/A
7. Sample results < MDA **and** < 5× the absolute value of the negative MB result.	N/A	UJ

4.2.2 Laboratory Control Sample/Laboratory Control Sample Duplicate

An LCS is analyzed to provide accuracy of the analytical method. An LCSD may be analyzed to demonstrate precision.

4.2.2.1 Deliverable

The following are deliverables.

- LCS/LCSD recovery summary
- Raw data (required for confirmation)
- Standard certificates

4.2.2.2 Frequency

The LCS **shall** be analyzed at a frequency of at least one per preparatory batch.

4.2.2.3 Criteria

The LCS contains the radionuclide of interest (targeted) and/or a radionuclide that has similar quanta-emission energies, and/or contains a radionuclide(s) that adequately indicates the performance of the analytical process/measurement.

Standards used in the preparation of the LCS **shall** be traceable to a reliable source [e.g., National Institute of Standards and Technology (NIST), International Atomic Energy Agency (IAEA)].

The LCS percent recovery (%R) for analytes typically has acceptance criteria established by the laboratory based on an average of LCS data generated by the laboratory for a specific method. In the absence of laboratory-established limits, the following LCS %R limits are recommended.

- The LCS %R acceptance range is established at 100 ± 25% (%R = 75–125%).

- For gross alpha/gross beta analysis, the LCS %R acceptance range of 75–125% is applicable when the analyte in the LCS is the same analyte used for the calibration curve. The LCS %R acceptance criteria for gross alpha and gross beta measurements is 100 ± 30% (70–130%) when the analyte in the LCS is **NOT** the same analyte used for the calibration curve.

4.2.2.4 Data verification

Verify the presence of required LCS/LCSD recovery summary forms. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem. Apply a "L05" validation reason code to the affected data if a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

Verify the presence of required standard certificates. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem. Apply an "E02" validation reason code to the affected data **if** a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

4.2.2.5 Data validation

Verify the traceability of the standards used. **If** the standards used in preparation of the LCS are **NOT** traceable to a reliable source (e.g., NIST, IAEA), **then** apply an "R" validation code to all associated detected and nondetected results.

If the LCS criteria are **NOT** met, **then** laboratory performance and method accuracy are in question. The data validator **shall** verify that the LCS and/or LCSD were prepared and analyzed in the same fashion as the sample they accompany. Qualification should be applied only if the LCS and other QC data within the batch indicate that the accuracy of reported compounds has been affected. Professional judgment should be used to determine **if** the data should be qualified. The following guidance is suggested for qualifying sample data for which the associated LCS and/or LCSD does **NOT** meet the required criteria.

- **If** an LCS was **NOT** analyzed with the analytical batch, **then** apply an "R" validation code to detected and nondetected results.
- **If** the LCS was **NOT** analyzed at the proper frequency, **then** apply a "J" validation code to detected results and a "UJ" validation code to nondetected results.
- **If** the LCS %R for an analyte is less than the lower acceptance limit, **then** apply a "J" validation code to detected results and apply an "R" validation code to nondetected results.
- **If** the LCS %R for an analyte is greater than the upper acceptance limit, **then** apply a "J" validation code to detected results. No qualification is necessary for nondetected results.

Table 6 summarizes data validation qualification guidance for issues with the LCS.

Table 6. LCS Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	Nondetects
1. LCS standards **NOT** traceable.	R	R
2. LCS **NOT** prepared and analyzed.	R	R
3. LCS **NOT** analyzed at the proper frequency.	J	UJ
4. LCS %R < lower acceptance limit.	J	R
5. LCS %R > upper acceptance limit.	J	N/A

4.2.3 Matrix Spike/Matrix Spike Duplicate

The purpose of the MS/MSD is to determine whether the sample matrix contributes bias to the analytical results. **If** the MS/MSD %R criteria are **NOT** satisfied, **then** there is difficulty in assessing whether the cause was due to method performance or matrix. To address this issue, LCS and/or LCSD are analyzed to verify method accuracy. **If** only the MS/MSD are affected, **then** a matrix effect is likely.

The data validator may determine that only some of the samples in the data package are similar to the MS sample, **and** that only these samples should be qualified. The data validator may determine that **NO** samples are sufficiently similar to the sample used for the MS, **and** that only the field sample used to prepare the MS sample should be qualified.

MS/MSD may **NOT** be required for methods where a carrier or tracer is used, provided that the tracer chosen is chemically similar to the radionuclide of interest. Matrix effects will be detected through tracer

recovery; however, difficulty may be experienced in ascertaining that poor recovery is due to matrix effect or through losses in separation.

NOTE: Various filter materials may be submitted for analysis; however, MS/MSD analysis requirements **shall NOT** apply to filter materials because representative splits of these samples are generally **NOT** obtainable.

4.2.3.1 Deliverables

- MS/MSD recovery summary
- Raw data (required for confirmation)
- Standard certificates

4.2.3.2 Frequency

MS/MSDs are **NOT** required to be analyzed when chemical yield tracers or carriers are employed (e.g., alpha spectrometry, gammy spectrometry, liquid scintillation). When applicable, the MS **must** be analyzed at a frequency of at least one per preparatory batch.

4.2.3.3 Criteria

The acceptance criteria for the MS %R **shall** fall between 60%–140% **if** customer or reference method requirements are **NOT** specified. For samples where the native sample activity is > 5× the spike value, the MS %R criteria does **NOT** apply.

Standards used in the preparation of the MS **shall** be traceable to a reliable source (e.g., NIST, IAEA).

4.2.3.4 Data verification

Verify the presence of required MS/MSD recovery summary forms. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem. Apply an "M05" validation reason code to the affected data **if** a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

Verify the presence of required standard certificates. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem. Apply an "E02" validation reason code to the affected data **if** a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

4.2.3.5 Data validation

If MS/MSD analysis was required but **NOT** performed, **then** qualify only **if** the deviation indicates an adverse effect on data quality. Occasionally limited sample volumes prevent the preparation and analysis of MS/MSDs. In these cases, it is common practice to utilize the LCS as a substitute to provide an evaluation of precision in the measurable range of the method.

The laboratory may also include an MS/MSD analysis performed on a parent sample that is **NOT** from the sample set being reviewed in the laboratory data package. This is commonly called a "batch QC sample."

The data validator should consult with the SMO to determine whether the batch QC data is applicable to the sample set being validated.

Consideration should be given to the similarity in matrix type among samples in the preparation batches. **If** the matrices differ notably (particularly in soil particle size), **then** qualification **may** be placed only on the sample associated with the MS. If matrices do **NOT** differ notably, qualification **may** be placed on all samples in the preparation batch. **If** multiple quality deficiencies are encountered, **then** qualify using guidance provided in Appendix B.

Verify that an MS was analyzed for each applicable analysis type within the analytical batch **and** that the MS is traceable to an original sample in the SDG. Verify the MS %R is within the acceptance limits of 60–140%. Verify the traceability of the standards used.

- **If** an MS was **NOT** analyzed within the analytical batch **and** is required, **then** apply a "J" validation code to detected results and apply a "UJ" validation code to nondetected results.
- **If** the MS standards are **NOT** traceable to a reliable source (e.g., NIST, IAEA), **then** apply an "R" validation code to associated detected and nondetected results.
- **If** the MS %R is < 60%, **then** apply a "J" validation code to detected results **and** apply a "UJ" validation code to nondetected results.
- **If** the MS %R is > 140%, **then** apply a "J" validation code to detected results. No qualification is necessary for nondetected results.

Table 7 summarizes data validation qualification guidance for issues with the MS/MSD.

Table 7. MS/MSD Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	Nondetects
1. MS/MSD required **and NOT** analyzed.	J*	UJ*
2. MS standards **NOT** traceable.	R	R
3. MS %R < 60%.	J	UJ
4. MS %R > 140%.	J	N/A

*In cases of insufficient sample volume, alternative QC may be used to evaluate precision and accuracy (e.g., LCS/LCSD and laboratory duplicates).

4.2.4 Duplicates

A laboratory duplicate sample **may** be analyzed for each matrix to evaluate the precision of the laboratory at the time of analysis. DoD/DOE QSM refers to this as a matrix duplicate. A field duplicate sample is collected and analyzed to evaluate the precision of both the sampling techniques and the laboratory methodology. A field duplicate also **may** provide information on the homogeneity of the sample. Nonhomogeneous samples can impact the apparent method precision; however, aqueous/water samples are generally homogenous, and most soil/sediment samples are homogenous within a factor of two or three.

4.2.4.1 Deliverables

The following are deliverables.

- Laboratory duplicate sample summary
- Raw data (required for confirmation)

4.2.4.2. Frequency

If analyzed, laboratory duplicates **shall** be analyzed at a frequency of at least one per preparatory batch. Field duplicates are collected at a frequency identified in associated project planning documents (QAPPs, etc.).

4.2.4.3. Criteria

The following are criteria for laboratory and field blanks.

- Samples identified as field blanks or equipment rinsate blanks **must NOT** be analyzed as the laboratory duplicate.

- The relative percent difference (RPD) precision criteria for aqueous and solid laboratory duplicate samples **must** be within ± 25%.

- The RPD precision criteria for aqueous field duplicate samples **must** be within ± 25%. The RPD precision criteria for solid field duplicate samples **must** be within ± 40%.

- The duplicate error ratio (DER) acceptance criteria is ≤ 3.

4.2.4.4. Data verification

Verify that field blanks and/or equipment rinsate blanks were **NOT** analyzed as laboratory duplicates. **If** a field blank or equipment rinsate blank has been used, **then** contact the SMO to have the laboratory address the issue. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem **and** shall be identified as such on the data verification/validation checklist.

Verify the presence of laboratory and field duplicate results. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem **and** shall be identified as such on the data verification/validation checklist.

4.2.4.5. Data validation

The following are data validation steps to evaluate laboratory and/or field duplicates.

- Examine the raw data (if provided) for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility).

- Verify that appropriate methods and amounts were used in preparing the samples for analysis.

- Verify that there are **NO** transcriptions or reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.

- Verify that results fall within the linear range(s) of the instrument, if applicable.
- Calculate the RPD between sample and duplicate using the following equation:

$$RPD = \frac{High\ \ Result\ \ \ - Low\ \ Result}{(Average\ \ Result\ \ )} * 100$$

NOTE: If the sample matrices are something other than water and soil, the RPD criteria may not apply.

Calculate the DER between the sample result and duplicate sample result.

$$DER = \frac{|S - D|}{\sqrt{\left(\frac{1}{2}{\sigma_s}^2 - \frac{1}{2}{\sigma_D}^2\right)}}$$

Where:

DER = the duplicate error ratio of the duplicate results
S = the original sample result (as pCi/g or pCi/L)
D = the duplicate sample result (as pCi/g or pCi/L)
σ_s = the associated TPU (2σ) of the sample result
σ_d = the associated TPU (2σ) of the duplicate result

NOTE: When evaluating RPD and DER criteria, the DER takes precedence over the RPD.

The following summarizes data qualification guidance for evaluating laboratory duplicates and field duplicates.

- **If** the RPD **and** DER criteria have been met, **then** no qualification is necessary for detected and nondetected results.
- **If** the RPD criteria has **NOT** been met **and** the DER ≤ 3, **then** no qualification is necessary for detected and nondetected results.
- **If** the RPD criteria has **NOT** been met **and** the DER is > 3, **then** apply a "J" validation code to detected results and apply a "UJ" validation code to nondetected results.

Table 8 summarizes data validation qualification guidance for issues with the laboratory and/or field duplicate.

Table 8. Laboratory and Field Duplicate Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	Nondetects
1. RPD criteria met **and** DER ≤ 3.	N/A	N/A
2. RPD criteria **NOT** met **and** DER ≤ 3.	N/A	N/A
3. RPD criteria **NOT** met **and** DER > 3.	J	UJ

NOTE: The control limits above are method requirements for matrix-specific duplicate samples. It should be noted that laboratory variability arising from the subsampling of nonhomogeneous matrices is a common occurrence; therefore, for technical review purposes only, regional policy or project DQOs may allow the use of less restrictive criteria to be used in assessing nonhomogeneous matrices. When project-specific DQOs mandate broader precision requirements, this information will be provided to the data validators as part of the validation SOW.

4.2.5. Chemical Yield—Tracers and Carriers

Tracers and carriers are used in some radiochemical methods, depending on the instrument that is being used, to provide evaluation of chemical separation. Tracers and carriers are analytical method-specific and are added to both field samples and batch QC samples prior to sample preparation to determine the overall chemical yield for the analytical process. Carriers typically are nonradioactive elements with similar chemical characteristics as the analyte being analyzed. Tracers are radionuclides that mimic, but do not interfere with, the target radio analyte through preparation and analysis. The chemical yield provides an indication of any method anomalies such as sample losses (e.g., absorption, reactivity, spillage) or artifacts specific to the measurement step. Thus, the chemical yield of the tracer or carrier is used to normalize the measured activity of the isotope of interest. Because the tracer and carriers will vary from laboratory to laboratory, as well as instrument to instrument, the data validator should use the laboratory's analytical method in conjunction with the guidance in this plan in the validation of radiochemical data.

Chemical yield is evaluated through the recovery of chemical species spiked into samples. It is evaluated radiometrically with a tracer and gravimetrically with a carrier. Generally, a low yield is indicative of losses of tracer and radionuclide of interest through sample separation, and recoveries greater than expected (> 100%) are indicative of instrumental problems, contamination, or presence in sample; tracers and carriers are not expected to be recovered at levels greater than spiked.

4.2.5.1. Deliverables

- Chemical yield data
- Raw data (required for confirmation)
- Standard certificates

4.2.5.2 Frequency

When tracers or carriers are used, each sample (including any batch-associated QC samples) **shall** also be spiked with the same materials and individual sample yields **shall** be determined.

4.2.5.3. Criteria

The acceptance criteria for the isotopic or chemical yield **shall** fall between 30%–110% **if** customer or reference method requirements are **NOT** specified.

For alpha spectrometry tracers, the full width half maximum (FWHM) for the tracer peak should be < 100 keV and the peak energy within ± 50 keV of the known energy.

Standards used for tracers/carriers **shall** be traceable to a reliable source (e.g., NIST, IAEA).

4.2.5.4 Data verification

Verify the presence of required chemical yield reporting forms. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem. Apply an "Y04" validation reason code to the affected data **if** a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

Verify the presence of required standard certificates. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical

laboratory, **then** it is considered a noncorrectable problem. Apply an "E02" validation reason code to the affected data **if** a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

4.2.5.5. Data validation

Verify that a percent yield is reported for each sample result for analyses that require a carrier or tracer **and** compare calculated/reported yield with the acceptance criteria. Verify the traceability of the standards used.

The following guidance is suggested for qualifying sample data for which the tracer/carrier yield does **NOT** meet the required criteria.

- **If** the required carrier or tracer was **NOT** added to the sample, **then** apply an "R" validation code to the associated target analyte results.
- **If** the sample-specific tracer/carrier yield is > 110% **and** ≤ 120%, **then** apply a "J" validation code to detected results. No qualification is necessary for nondetected results.
- **If** the sample-specific tracer/carrier yield is > 120%, **then** apply an "R" validation code to detected results. No qualification is necessary for nondetected results.
- **If** the sample-specific tracer/carrier yield is ≥ 10% **and** < 30%, **then** apply a "J" validation code to detected results and apply a "UJ" validation code to nondetected results.
- **If** the sample-specific tracer/carrier yield is < 10%, **then** apply an "R" validation code to detected and nondetected results.

For alpha spectrometry data, verify the FWHM for the tracer peak is < 100 keV **and** the peak energy falls within ± 50 keV of the known peak energy. **If** either of these criteria is **NOT** met, **then** apply a "J" validation code to detected results and apply a "UJ" validation code to nondetected results.

If the standards used for tracers/carriers **are NOT** traceable to a reliable source (e.g., NIST, IAEA), **then** apply an "R" validation code to detected and nondetected results.

Table 9 summarizes data validation qualification guidance for chemical yield evaluation.

Table 9. Chemical Yield Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	Nondetects
1. Tracer/carrier **NOT** added to sample.	R	R
2. Tracer/carrier yield > 110% **and** ≤ 120%.	J	N/A
3. Tracer/carrier yield > 120%.	R	N/A
4. Tracer/carrier yield ≥ 10% **and** < 30%.	J	UJ
5. Tracer/carrier yield < 10%.	R	R
6. Alpha spectrometry only: FWHM < 100 keV **or** peak energy **NOT** within ± 50 keV of the known peak energy.	J	UJ
7. Tracer/carrier standards **NOT** traceable.	R	R

4.3 INSTRUMENT-RELATED QUALITY CONTROL RESULTS

4.3.1 Initial Calibration

Compliance requirements for satisfactory instrument calibration ensure that the instrument can produce acceptable qualitative and quantitative data for radiochemical analysis. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run.

NOTE 1: For gamma spectrometry analysis, the calibration counting geometry used **must** be the same as that used with the analytical samples.

NOTE 2: Calibration data **must** be background subtracted, regardless of whether data is used in generation of efficiencies, cross-talk, or resolution evaluation.

4.3.1.1 Deliverables

The deliverable for evaluating initial calibration is instrument control charts.

4.3.1.2 Frequency

Initial calibration **must** be performed before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following an instrument repair; after modification of operating parameters that affect the instrument responses; or when instrument performance checks exceed predetermined limits. Initial calibration **shall** use a traceable calibration source that matches sample test source configuration.

NOTE: Calibration sources are typically NIST- or IAEA-traceable.

4.3.1.3 Criteria

The following sections present the DoD/DOE QSM 6.0 requirements for initial calibration per instrument type that the laboratory is to achieve.

Alpha Spectrometry

- Verify manufacturer's specification for point source efficiency.
- At least three isotopes within the energy range of 3-6 MeV.
- Energy versus channel slope equation < 15 keV per channel.
- FWHM < 100 keV for each peak used for calibration.
- Minimum of 3,000 net counts in each peak.
- Peak energy positions of all calibration isotopes are within 40 keV of reference peak energies.

Gamma Spectrometry

- Minimum of 10,000 net counts in each reference peak for at least six calibration peaks that bracket the range of use.

- For standard broad-spectrum detectors, follow one of these two options.

  — Option 1

    – Peak energy difference is within 0.1 keV of reference energy for all points.
    – Peak FWHM < 2.5 keV at 1,332 keV.
    – Energy vs. channel slope equation shall be linear and accurate to 0.5 keV.

  — Option 2

    – Verify manufacturer's specifications for gamma peak resolution.
    – Efficiency vs. energy for each geometry/matrix. 95% confidence limit of the fitted function: ≤ 8% over energy range.
    – Low-energy or thin-window gamma systems shall be calibrated to an energy range in accordance with manufacturer's specifications or data sheets.

Gas Flow Proportional Counting

- Voltage Plateau
    – Perform a series of counts in ≤ 50 V steps from approximately 300–1,500 V. Determine a usage range where slope of the plateau is < 5% over 100 V change.

- Efficiency
    – Detector's counting efficiency, using traceable calibration sources, shall be determined for each radionuclide used to analyze test sources.
    – Verify manufacturer's specifications for detector efficiency for both alpha and beta counting modes using known sources.
    – A 1σ counting uncertainty of ≤ 1% shall be achieved for all detector efficiency determinations.

- Cross-talk Factors
    – Determine cross-talk factors for each nuclide using a traceable calibration source per matrix and method.
    – Verify manufacturer's specifications for cross-talk in alpha and beta channels.

- Self-Absorption Curve
    – Using traceable calibration sources, establish a mass attenuation curve with a minimum of seven mass-attenuated standards.
    – For each radionuclide of interest, establish a mathematical function (curve) of detector efficiency versus source mass loading. 95% confidence limit of the fitted function (curve) over the calibration range to ≤ 10% and ≤ 5% uncertainty for alpha and beta, respectively.

Liquid Scintillation Counter

- Analyze a minimum of five different quench factor sources.
- Obtain a minimum of 10,000 counts for each data point.
- The quench curve shall meet one of the following:
    – Option 1: Correlation coefficient for quench curve ≥ 0.995.
    – Option 2: Region of interest error < 1%, and 95% confidence limit for established curve is ≤ 5% over expected quench range.

Radon Scintillation (Ra-226 by Lucas Cell)

- Verify manufacturer's specifications.
- Confirm each selected operation voltage is within the slope range of < 2%/100 V.

4.3.1.4 Data verification

Verify the presence of required control charts. **If** they are not provided, contact the SMO to have the laboratory provide the missing information. **If** this issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem. Apply a "C07" validation reason code to affected data **if** a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

4.3.1.5 Data validation

Evaluate the control charts for out-of-control conditions. **If** out-of-control conditions exist, **then** verify the detector was **NOT** used for analysis **or** the detector was within acceptance criteria at the time of analysis.

If control chart limits are exceeded **and** the detector was used for sample analysis, **then** apply a "J" validation code to detected results and a "UJ" validation code to nondetected results.

Evaluate standard source traceability and standard source age.

If the standard source **CANNOT** be traced properly, **then** apply an "R" validation code to affected sample results **and** narrate the issue in the data validation report.

If the standard source has expired (i.e., analysis date greater than the expiration date on the certificate), **then** apply an "R" validation code to affected sample results **and** narrate the issue in the data validation report.

Table 10 summarizes data validation qualification guidance for evaluating issues with initial calibration.

Table 10. Initial Calibration Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	Nondetects
1. Control chart limits exceeded **and** detector used for sample analysis.	J	UJ
2. Standards were **NOT** traceable.	R	R
3. Standard source expired.	R	R

4.3.2 Continuing Calibration Verification

CCVs ensure that the instrument is capable of consistently producing acceptable qualitative and quantitative data. The instrument is checked over specific time periods during the sample analysis.

4.3.2.1 Deliverables

The deliverable for evaluating CCVs is the CCV summary report **or** daily calibration checks.

4.3.2.2. Frequency

The following sections present the DoD/DOE QSM 6.0 frequency requirements for CCV per instrument type.

Alpha Spectrometry

Weekly source check or pulser check verification before analysis of samples is required. Pulser check **may** be used to verify energy calibration when using radiotracers during analysis. Source check **may** be used to verify energy, FWHM, and efficiency.

Gamma Spectrometry

CCV is required daily or before use. When working with long count times or batch sequences that run more than a day, the CCV **shall** be performed at the beginning and end of each analytical batch. The maximum elapsed time between CCVs **shall NOT** exceed seven days.

Gas Flow Proportional Counting

CCV is required daily or before use **and** after a counting gas change. When working with long count times **or** batch sequences that run more than a day, the CCV **shall** be performed at the beginning **and** end of each analytical batch **if** the elapsed time between CCVs is **NOT** longer than seven days.

Liquid Scintillation Counter

CCV is required daily before samples analysis for short counting intervals. When working with long count times **or** batch sequences that run more than a day, the CCV **shall** be performed at the beginning **and** end of each analytical batch. The maximum elapsed time between CCVs **shall NOT** exceed seven days.

Radon Scintillation (Ra-226 by Lucas Cell)

Detector response check is required before use using an appropriate sample test source. Each cell/detector pair efficiency **shall** be checked at least annually.

4.3.2.3. Criteria

Each reporting batch submitted from the laboratory to the project should contain data related to calibration verification for all detectors used in the analyses of the analytical samples. This **may** be in the form of control charts **or** continuing calibration report forms, depending on the analytical method.

Calibration verification is performed and monitored with tolerance or QC charts for instrumental parameters specific to each type of detector. **If** the daily check source count result exceeds the tolerance limits **or** ± 3σ control limit, **then** the laboratory should recount the check source to verify the out-of-control condition. **If** the recount again exceeds the control limit, **then** the system is considered out of control, and **NO** samples **shall** be run on that system until it is brought back into control. **If** the recount is in control, **then** a third count **shall** be done, **and if** in control, analytical sample counting **may** continue. Otherwise, **NO** samples **shall** be analyzed on that system until it is brought back into control. Any samples counted after the last in-control check standard **must** be recounted, except for those where decay has eliminated that radionuclide.

If calibration verification data exceed the tolerance limits or the ± 3σ control limits, **then** reference **must** be made to QC sample data in the data package to determine the extent of calibration nonconformance on

the counting batch. Exceeding the control limits **may NOT** constitute qualification of data; conversely, excessive control limit exceedance **may** affect all data in a counting batch, justifying qualification.

The following sections present the DoD/DOE QSM 6.0 requirements for CCVs per instrument type that laboratory is to achieve.

Alpha Spectrometry

CCV energy response **shall** be monitored by one of the following.

- Option 1: Energy response checks **shall** have a tolerance limit **or** control chart set at ± 3% or 3σ.
- Option 2: Pulser check-observed peak centroid falls ≤ 20 keV from reference energy.

Check source FWHM ≤ 100 keV **and** within 40 keV of corresponding calibration peaks in initial energy calibration.

Gamma Spectrometry

Response checks **shall** meet one of the following:

- Option 1: Response **shall** meet acceptance criteria of ± 3% **or** 3σ of the mean.
- Option 2: Peak energy/efficiency: low, mid, and high energies **shall** be within 10% of the initial calibration value.

FWHM: Low, mid, and high energies **shall** be within 10% of initial FWHM value.

Gas Flow Proportional Counting

CCV contains a minimum of 2,000 net counts for each energy type (alpha, beta). Response check **shall** be within a tolerance limit or control chart limits ≤ 3% **or** 3σ of the mean.

Liquid Scintillation Counter

Response acceptance criteria **shall** be ± 3% **or** 3σ of the mean.

Radon Scintillation (Ra-226 by Lucas Cell)

The results of this detector response check **shall** fall within established laboratory-developed control limits. Continuing efficiency for each cell/detector pair **shall** be within < ± 25% of the initial average.

4.3.2.4. Data verification

Verify the presence of required reporting forms. **If** they are not provided, **then** contact the SMO to have the laboratory provide the missing information. **If** the issue cannot be resolved with the analytical laboratory, **then** it is considered a noncorrectable problem. Apply a "C07" validation reason code to the affected data **if** a noncorrectable problem has occurred **and** the problem has an adverse effect on data quality.

4.3.2.5. Data validation

Evaluate the daily calibration checks for out-of-control conditions. **If** daily calibration exceedances exist, **then** verify the detector was **NOT** used for analysis **or** the detector was within acceptance criteria at the time of analysis. When applicable, verify the CCV %R criteria was met.

If the detectors used for sample analysis were outside daily calibration acceptance criteria, **then** apply a "J" validation code to detected results and a "UJ" validation code to nondetected results.

If the CCV %R is outside acceptance criteria, **then** apply a "J" validation code to detected results and a "UJ" validation code to nondetected results.

Evaluate standard source traceability and standard source age.

If the standard source **CANNOT** be traced properly, **then** apply an "R" validation code to affected sample results **and** narrate the issue in the data validation report.

If the standard source has expired (i.e., analysis date greater than the expiration date on the certificate), **then** apply an "R" validation code to affected sample results **and** narrate the issue in the data validation report.

Table 11 summarizes the validation qualification guidance for CCV issues.

Table 11. CCV Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	Nondetects
1. Daily calibration check exceeded **and** detector used for sample analysis.	J	UJ
2. CCV %R exceeds acceptance criteria.	J	UJ
3. Standards were **NOT** traceable.	R	R
4. Standard source expired.	R	R

4.4 RECALCULATION CHECKS

The accuracy and consistency of sample result calculation by the laboratory can be addressed using two different techniques. The application of each strategy depends on the laboratory's ability to minimize transcription during reporting and how familiar the project is with the performance of the laboratory.

If the laboratory has a high rate of manual transcription in generation of sample results, **then** the project **may** choose to manually recalculate sample results at a determined frequency. **If** sample results **CANNOT** be reproduced through manual calculation, **then** contacting the laboratory may be necessary to resolve the problem. "R" validation codes may be applied to data as a last resort, **if NO** actions can reproduce reported values. For Stage 3 and Stage 4 validations only, **if** recalculations are performed, **then** recalculate one sample result from raw data for confirmation.

NOTE: Calculations for compound quantitation and rounding rules can be found in Appendix C.

Activity and TPU equations provided in Appendix C are useful for providing the basic structure for calculating radiochemical results. Modifications to the equations may be needed in method-specific cases. Additional calculations **may** be required and should be included with the specific analytical method being followed. **If** calculations are **NOT** in the method, **then** the laboratory can provide sample calculations such that the data validator will be able to check any necessary calculated results as needed.

4.4.1 Nuclide Identification and Quantitation

Verify that sample results that are less than the MDA are reported correctly. **If** the sample result is less than the MDA **and NOT** qualified with a "U" qualifier by the laboratory, **then** notify the SMO to have the data reviewed by the laboratory.

Evaluate negative sample results to ensure proper background subtraction has been implemented. **When** the absolute value of the negative sample result is greater than the TPU, **then** there is a potential for low bias in reported sample results.

4.4.1.1 Deliverables

The deliverable for evaluating nuclide identification and quantitation is sample summary/sample data sheets.

4.4.1.2 Frequency

MDAs and TPUs **must** be reported for all radionuclides.

4.4.1.3 Criteria

Sample results that are less than the MDA must have a "U" qualifier applied by the laboratory.

4.4.2 Total Propagated Uncertainty

Quality-indicator samples (e.g., MB, LCS, MS/MSD, chemical yield) are evaluated during analytical data validation to determine the control of the analytical method and matrix-related effects on sample data. For analytical data validation, quality-indicator samples help determine what radioanalytical conditions affect the usability of the data. The strategy by which quality-indicator samples are evaluated involves an evaluation of whether the difference between expected and measured results is statistically significant when compared to their TPU. The mathematical relationships presented in the following sections are compared to a factor corresponding to a statistical level of confidence. When the relationship exceeds the factor, the two results differ at that statistical level of confidence when compared to their TPU.

The statistical assumption inherent in these tests is that sample results are drawn from normally distributed populations with estimated means and known variances. Factors in the TPU relationship may originate from populations that are not necessarily normally distributed (e.g., counting uncertainty); however, use of sample results and TPU, assuming approximation to the normal distribution, provides a reasonable and appropriate approach to evaluating control of analytical conditions. Presented in this document are statistical decision-making levels at 95% and 99% levels of confidence [decision-making factors are 1.96 (2σ) and 2.58 (3σ), respectively]. Projects may choose other levels of confidence and decision-making factors based on project DQOs, with the realization that qualification decisions made through data validation will be at differing levels of confidence and conservatism.

The tests presented in this section rely heavily upon evaluation of uncertainty associated with radioanalytical results. The random factor in the TPU relationship is the counting uncertainty; the remaining terms comprise the systematic factors. Many laboratories choose to report uncertainties separately as total random and total systematic. These factors are acceptable to use in the tests in this section provided that the components of the uncertainties are recognized. A laboratory should include in their TPU equation counting uncertainty, efficiency uncertainty, and recovery uncertainty. Other terms can be propagated individually, or a collective term may be used (the collective term being a combination of the systematic uncertainty summed with any other relatively minor, random individual uncertainties). The laboratory should explicitly

show which terms were individually propagated and which were bundled together. In all cases, significant uncertainties should be propagated for the laboratory TPU calculation.

If NOT all the requested uncertainties are available, **then** the magnitude of TPU **must** be evaluated considering which components are the dominant factors in the relationship. At relatively low count rates, the random components will likely be the dominant factors, and at high rates, systematic components may be dominant.

To enable performance of the evaluations in this plan, all samples **must** be reported with a background-subtracted sample result, a ± 2σ counting uncertainty, a ± 2σ TPU, and an MDA.

4.4.3 Elevated Total Propagated Uncertainty

At some elevated level of uncertainty, sample result qualification **may** be needed to alert the data user that the sample result **may** be too uncertain to use for an intended purpose.

4.4.3.1 Deliverables

The deliverable for evaluating elevated TPUs is sample summary/sample data sheets.

4.4.3.2 Frequency

MDAs and TPUs **must** be reported for all radionuclides.

4.4.3.3 Criteria

- TPU should be reported at 2σ.
- **If** the sample result is > 10× the MDA, **then** the TPU should be < 25% of the sample result.
- **If** the sample result is negative, **then** the absolute value of the sample result should be less than the TPU.

4.4.3.4 Data verification

Verify the presence of required reporting forms. **If** they are not provided, **then** contact the SMO to request they be provided by the laboratory. **If** the issue **CANNOT** be resolved, **then** a noncorrectable problem exists.

4.4.3.5 Data validation

Compare sample results and TPUs for each result.

If the TPU is $\geq$ 25% of the sample result **and** the sample result is > 10× the MDA, **then** apply a "J" validation code to detected results. No qualification is necessary for nondetected results.

If the sample result is negative **and** the absolute value of the sample result is greater than the TPU, **then** apply a "UJ" validation code to nondetected results. No qualification is necessary for detected results.

Table 12 summarizes data validation qualification guidance for elevated TPU.

Table 12. Elevated TPU Validation Qualification Guidance

Validation Step	Validation Qualification Guidance	
	Detects	Nondetects
1. TPU ≥ 25% of the sample result **and** sample result > 10× the MDA.	J	N/A
2. Absolute value of negative sample result > TPU.	N/A	UJ

5. RECORDS

Generate and maintain all records in accordance with CP3-RD-0010, *Records Management Process*, which include the following.

- Data verification/validation checklist (Stage 2B, Stage 3, and Stage 4 validation)
- Data validation report (Stage 2B, Stage 3, and Stage 4 validation)

6. REFERENCES

NOTE: Use the most current version of the references that are listed below for data review, verification, and validation processes.

CP3-ES-5003, *Quality Assured Data*

DoD and DOE (U.S. Department of Defense and U.S. Department of Energy) 2023. *Department of Defense and Department of Energy Quality Systems Manual for Environmental Laboratories Version 6.0*, U.S. Department of Defense Environmental Data Quality Workgroup and U.S. Department of Energy Consolidated Audit Program Data Quality Workgroup, Washington, DC, December.

EPA (U.S. Environmental Protection Agency) 2018. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods Compendium*, SW-846, Revisions through Update VI, U.S. Environmental Protection Agency, Washington, DC, December.

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APPENDIX A

DATA VALIDATION CODES AND DATA VALIDATION REASON CODES

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A.1. DATA VALIDATION CODES AND DATA VALIDATION REASON CODES

Data Validation Codes

U The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

NJ Presumptively present at an estimated quantity [use with tentatively identified compounds (TICs) only].

UJ Analyte, compound, or nuclide not detected above the reported detection limit, and the reported detection limit is approximated due to quality deficiency.

R Result rejected by validator.

= Validated result, no additional qualifier necessary.

X Not validated; Refer to the RSLTQUAL field for more information.

Data Validation Reason Codes

Blanks

B01 Sample concentration was less than the reporting limit (RL), and ≤ 5× the blank concentration (10× for common contaminants).
B02 Sample concentration was greater than the RL, and ≤ 5× the blank concentration (10× for common contaminants).
B03 Gross contamination exists; blank result impacted associated analyte data quality.
B04 Negative blank result impacted associated analyte data quality.
B05 Blanks were not analyzed at appropriate frequency.
B06 Sample not significantly different than radiochemical method blank.
B07 Blank data not reported.
B08 Instrument blank not analyzed after high-level sample.
B09 Other (describe in comments)
B10 Method blanks not extracted at appropriate frequency.
B11 Sample results were corrected for blank contamination.
B12 Blank was not the same matrix as the analytical samples.
B13 Concentration of target compound detected in sample affected by carryover.

Calibration

C01 Initial calibration average relative response factor (RRF) was < 0.05 or < 0.01 for poor response compounds
C02 Initial calibration percent relative standard deviation was exceeded
C03 Initial calibration sequence was not followed as appropriate
C04 Continuing calibration RRF was < 0.05 or < 0.01 for poor response compounds
C05 Continuing calibration percent difference (%D) was exceeded
C06 Calibration or performance check was not performed at the appropriate frequency
C07 Calibration data not reported

Calibration (continued)

C08 Calibration not performed
C09 Chemical resolution criteria were not satisfied
C10 Calibration standard matrix not the same as sample matrix
C11 Compounds quantitated against inappropriate standard or standard concentration level
C12 Compound quantitated against inappropriate ion
C13 Calibration factor relative standard deviation criteria were not satisfied
C14 Retention time of compound outside window
C15 Initial calibration percent recovery (%R) was below lower acceptance limit
C16 Initial calibration %R was above upper acceptance limit
C17 Initial calibration curve fit was < 0.995
C18 Inappropriate standard concentrations
C19 Continuing calibration %R was below the lower acceptance limit
C20 Continuing calibration %R was above the upper acceptance limit
C21 Contract-required detection limit %R was below the lower acceptance limit
C22 Contract-required detection limit %R was above the upper acceptance limit
C24 Standard curve was established with fewer than the appropriate number of standards
C27 Calibration verification efficiency outside control criteria
C28 Calibration verification background outside control criteria
C29 Calibration verification energy outside control criteria
C30 Calibration verification peak resolution outside control criteria
C31 Chromatogram does not show adequate gain setting
C32 Other (describe in comments)

Laboratory Duplicate/Dual Column Sample Confirmation

D01 Significant difference between sample and duplicate
D02 Laboratory duplicate was not analyzed at the appropriate frequency
D03 Laboratory duplicate exceeds relative percent difference (RPD) criteria
D04 Laboratory duplicate data not reported
D05 Other (describe in comments)
D06 %D between primary and secondary column confirmation exceeds acceptance criteria

Evidentiary Concerns

E01 Custody of sample in question
E02 Standard not traceable
E03 Other (describe in comments)

Interference Check Samples (ICS)

F01 ICS recovery below lower control limit or advisory limit
F02 ICS recovery above upper control limit or advisory limit

General

G01 Professional judgment was used to qualify the data
G02 Other (describe in comments)

Holding Times/Preservation

H01 Extraction holding times were exceeded
H02 Extraction holding times were grossly exceeded
H03 Analysis holding times were exceeded
H04 Analysis holding times were grossly exceeded
H05 Samples were not preserved properly

Holding Times/Preservation (continued)
H06 Sample preservation cannot be confirmed
H07 Sample temperature exceeded criteria prior to preparation
H08 Other (describe in comments)

Internal Standards
I01 Area count was above upper control limits
I02 Area count was below lower control limits
I03 Extremely low area counts or performance was exhibited by a major drop-off
I04 Internal standard retention time varied by more than 30 seconds
I05 Inappropriate internal standard used
I06 Inappropriate internal standard concentration(s) used
I07 Internal standard data not reported
I08 Other (describe in comments)

Laboratory Control Sample (LCS)
L01 LCS recovery above upper control limit
L02 LCS recovery below lower control limit
L03 LCS was not analyzed at appropriate frequency
L04 LCS not the same matrix as the analytical samples
L05 LCS data not reported
L06 Other (describe in comments)

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)
M01 MS and/or MSD recovery above upper control limit
M02 MS and/or MSD recovery below lower control limit
M03 MS/MSD pair exceeds the RPD limit
M04 MS and/or MS/MSD not analyzed at the appropriate frequency
M05 MS and/or MS/MSD data not reported
M06 Other (describe in comments)

Instrument Performance
P01 High background levels or a shift in the energy calibration were observed
P02 Extraneous peaks were observed
P03 Loss of resolution was observed
P04 Peak tailing or peak splitting that may result in inaccurate quantitation were observed
P05 Instrument performance data not reported
P06 Instrument performance not analyzed at the appropriate frequency
P07 Other (describe in comments)
P08 Resolution check mixture (RCM) not analyzed at the beginning of the initial calibration sequence
P09 RCM criteria were not met
P10 RPD criteria in performance evaluation mixture was not met

Quantitation
Q01 Peak misidentified
Q02 Target analyte affected by interfering peak
Q03 Qualitative criteria were not satisfied
Q04 Cross contamination occurred
Q07 Analysis occurred outside 12-hour gas chromatography/mass spectrometry window
Q09 Tentatively identified compound (TIC) result was not above 10 × the level found in the blank
Q10 TIC reported as detect in another fraction

Quantitation (continued)

Q11 Common artifact reported as a TIC
Q12 No raw data were provided to confirm quantitation
Q13 Minimum detectable activity (MDA) greater than RL
Q14 Inappropriate aliquot sizes were used
Q15 Sample result less than MDA
Q16 Sample result less than 2σ uncertainty
Q17 Negative result
Q18 Compounds were not adequately resolved
Q19 Sample geometry different from calibration geometry
Q20 Sample weight greater than greatest weight on mass attenuation curve
Q21 Isotopes of same radionuclide do not show equilibrium
Q22 Peak not within appropriate energy range
Q23 Counting uncertainty ≥ 80% of sample result
Q24 Raw data anomaly
Q25 Other (describe in comments)
Q26 Retention Time (RT) outside calculated RT window
Q28 Neither RL or the sample quantitation limit (SQL) are reported for a nondetect result
Q29 SQL greater than RL
Q30 Compound detected at less than SQL and not qualified "J"
Q31 Presence of high molecular weight contaminants impacted sample quantitation

Surrogates

S01 Surrogate recovery was above the upper control limit
S02 Surrogate recovery was below the lower control limit
S03 Surrogate recovery was < 10%
S04 Inappropriate surrogate standard used
S05 Inappropriate surrogate standard concentration(s) used
S06 Surrogate data not reported
S07 Surrogate outside retention window
S08 Other (describe in comments)

Instrument Tuning

T01 Mass calibration ion misassignment
T02 Mass calibration was not performed every 12 hours
T03 Mass calibration did not meet ion abundance criteria
T04 Mass calibration data was not reported
T05 Scans were not properly averaged
T06 Other (describe in comments)

Pesticide Sample Cleanup

U01 Florisil performance requirements not met
U02 Gel permeation chromatography (GPC) calibration not checked at required frequency
U03 GPC calibration criteria not met
U04 GPC blank not analyzed after GPC calibration
U05 GPC blank greater than half the RL for target compound

Cleanup

V01 10% recovery or less was obtained during either check
V02 Recoveries during either check were > 120%
V04 Cleanup data not reported

Cleanup (continued)

V05 Cleanup check not performed at the appropriated frequency
V06 Other (describe in comments)

Dilutions

X01 Serial dilution not analyzed at the appropriate frequency
X02 %D between the original sample and the diluted result (or serial dilution) exceeded acceptance criteria
X03 Reported results not corrected for dilution factor
X04 Other (describe in comments)

Radiochemical Yield

Y01 Radiochemical tracer yield was above the upper control limit
Y02 Radiochemical tracer yield was below the lower control limit
Y03 Radiochemical tracer yield was zero
Y04 Radiochemical yield data was not present
Y05 Other (describe in comments)

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APPENDIX B

QUALIFICATION TABLES FOR MULTIPLE QUALITY DEFICIENCIES

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B.1. QUALIFICATION TABLES FOR MULTIPLE QUALITY DEFICIENCIES

This appendix provides guidance in the application of validation codes to data due to instances of multiple quality deficiencies. Quality deficiencies can be categorized based on the potential effect on sample data. The effect of quality deficiencies may be applicable to only a single sample or to all samples within the reporting batch. A validation code should not be placed on sample data until all quality deficiencies have been identified within the reporting batch.

Table B.1 provides a listing of data quality indicators and the probable effects on sample data.

Table B.1. Data Quality Indicators and Effects on Sample Data

Data Quality Indicator	Effect on Sample Data
Initial calibration	Identification and quantitation
ICV/CCV	Quantitation
MB	Positive bias
LCS/LCSD	Method bias and precision
MS/MSD	Positive or negative bias and precision
Duplicates	Precision
Tracer/carrier chemical yield	Positive or negative bias

In the instance of multiple quality deficiencies, the validation code should be placed consistent with the acceptable level of uncertainty associated with the intended use of the data. The validation statement of work should provide a summary of the intended use(s) of the data. (e.g., risk assessment, fate and transport modeling, waste management) to facilitate appropriate placement of validation codes.

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APPENDIX C

RULES, CALCULATIONS, AND EQUATIONS

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C.1. RULES, CALCULATIONS, AND EQUATIONS

Rounding Rules

1. In a series of calculations, carry the extra digits through to the final result, and then round off.
2. If the digit to be removed is < 5, the preceding digit stays the same.
3. If the digit to be removed is ≥ 5, the preceding digit is increased by 1.

Calculations/Equations

C.1 Sample Activity Concentration—Method Blank Corrected Sample Concentrations

ACT_S =sample activity concentration (pCi/g or pCi/L)

$$ACT_S = \frac{NCR_S}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

ACT_S = sample activity concentration (pCi/g or pCi/L)
NCR_S = net sample count rate in cpm
2.22 = factor for converting dpm to pCi
EFF = detector efficiency (fraction)
ALI = sample aliquot volume or mass (g or L)
ABN_S = abundance fraction of the emissions used for analyte identification/quantification
R = sample tracer/carrier (chemical) recovery
λ = analyte decay constant - ln 2/(half-life) (same units as the half-life used to compute λ)
t = time from sample collection to radionuclide separation or mid-point of count time (same units as half-life)
CF = other correction factors as appropriate (i.e., ingrowth factor, self-absorption factor, etc.)

C.2 Net Sample Count Rate (NCR) and σNCR—Method Blank Corrected Sample Concentrations

$$NCR = \left(\frac{C_{GS}}{T_{GS}} - \frac{C_{SB}}{T_{SB}}\right) - \left[\left(\frac{C_{GB}}{T_{GB}} - \frac{C_{BB}}{T_{BB}}\right) * \left(\frac{EFF_{SD}}{EFF_{BD}}\right) * \left(\frac{R_S}{R_B}\right)\right]$$

NCR = net sample count rate
C_{GS} = sample counts
T_{GS} = sample count time (minutes)
C_{SB} = background counts
T_{SB} = background count time (minutes)
C_{GB} = gross method blank counts
T_{GB} = gross method blank count time (minutes)
C_{BB} = method blank background counts
T_{BB} = method blank background count time (minutes)
EFF_{SD} = efficiency of the sample detector
EFF_{BD} = efficiency of the method blank detector
R_S = sample tracer/carrier recovery fraction
R_B = method blank tracer/carrier recovery fraction

C.3 Uncertainty of Net Count Rate

$$\sigma_{NCR}^2 = \left[\frac{C_{GS}}{T_{GS}^2} + \frac{C_{SB}}{T_{SB}^2}\right] + \left\{\left[\frac{C_{GB}}{T_{GB}^2} + \frac{C_{BB}}{T_{BB}^2}\right] * \left(\frac{EFF_{SD}}{EFF_{BD}}\right)^2 * \left(\frac{R_S}{R_B}\right)^2\right\}$$

$$+ \left\{\left[\left(\frac{C_{GB}}{T_{GB}} - \frac{C_{BB}}{T_{BB}}\right) * \left(\frac{EFF_{SD}}{EFF_{BD}}\right) * \left(\frac{R_S}{R_B}\right)\right]^2 * \left[\left(\frac{\sigma_{EFF_{SD}}}{EFF_{SD}}\right)^2 + \left(\frac{\sigma_{EFF_{BD}}}{EFF_{BD}}\right)^2 + \left(\frac{\sigma_{R_S}}{R_S}\right)^2 + \left(\frac{\sigma_{R_B}}{R_B}\right)^2\right]\right\}$$

$$\sigma_{NCR} = \sqrt{\sigma_{NCR}^2}$$

C.4 Calculation of Recovery – Radiometric—Method Blank Corrected Sample Concentrations

$$R = \frac{\left(\frac{C_{GT}}{T_{GT}} - \frac{C_{TB}}{T_{TB}}\right)}{EFF * ABN_T * AMT_T} = \frac{NCR_T}{EFF * ABN_T * AMT_T}$$

R	=	tracer recovery
C_{GT}	=	gross count of tracer
T_{GT}	=	tracer count time (minutes)
C_{TB}	=	background count of tracer [region of interest (ROI)]
T_{TB}	=	background count time (minutes)
EFF	=	detector efficiency fraction
AMT_T	=	amount of tracer activity added (dpm)
NCR_T	=	net count rate of tracer (cpm)
ABN_T	=	abundance fraction of the tracer emissions used for quantification of the tracer

NOTE: It is assumed that the tracer half-life is long enough to be an insignificant uncertainty contributor. If the tracer has a relatively short half-life, then it must be considered and these equations modified. Likewise, uncertainty in the time is also considered to be an insignificant contributor.

C.5 Calculation of RER—Radiometric—Method Blank Corrected Sample Concentrations

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 + \left(\frac{\sigma_{EFF}}{EFF}\right)^2 + \left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2$$

$$\left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 = \frac{\left(\frac{C_{GT}}{T_{GT}^2} + \frac{C_{TB}}{T_{TB}^2}\right)}{NCR_T^2}$$

$$\left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2 = \left(\frac{\sigma_{STS_T}}{STS_T}\right)^2 + \left(\frac{\sigma_{MASS_T}}{MASS_T}\right)^2 + \left(\frac{\sigma_{VOL_T}}{VOL_T}\right)^2 + \Sigma\left(\frac{\sigma_{DIL_T}}{DIL_T}\right)^2 + \Sigma\left(\frac{\sigma_{ALI_T}}{ALI_T}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

RE_R	=	relative error of the tracer recovery
σR	=	standard deviation of the tracer recovery
σNCR_T	=	standard deviation of the tracer's net count rate
σEFF	=	standard deviation of the detector efficiency
σAMT_T	=	standard deviation of the amount of tracer activity added
σSTS_T	=	standard deviation of the amount of tracer activity taken for stock tracer solution (provided with certificates received with standards)
$\sigma MASS_T$	=	standard deviation of the mass of standard solution used to prepare stock tracer solution
σDIL_T	=	standard deviation of the volume(s) of the dilution(s) made to prepare the working tracer solution
σVOL_T	=	standard deviation of the volume of the stock tracer solution
σALI_T	=	standard deviation(s) of the aliquot(s) of tracer solution(s) diluted to prepare working tracer solution
C_{GT}	=	gross count of tracer
T^2_{GT}	=	square of tracer count time (minutes)
C_{TB}	=	background count of tracer (area or ROI)
T^2_{TB}	=	square of background count time (minutes)
R	=	tracer recovery
NCR_T	=	net count rate of the tracer
EFF	=	detector efficiency
AMT_T	=	amount of tracer activity added (dpm)
STS_T	=	amount of tracer activity (dpm) in stock tracer solution
$MASS_T$	=	mass (grams) of standard solution used to prepare stock tracer solution
VOL_T	=	volume of tracer solution added
DIL_T	=	volume(s) of dilution(s) made to prepare the working tracer solution
ALI_T	=	aliquot(s) of tracer solution(s) taken to prepare serial tracer solution dilution(s)

Note: Certificates, such as those from NIST, may give two or even three sigma uncertainty. Only one sigma should be used for σSTS_T.

C.6 Calculation of Recovery and RER—Gravimetric—Method Blank Corrected Sample Concentrations

$$R = \frac{WT_c}{CONC_{CS} * VOL_{CS}}$$

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{PPT.WT.}}{PPT.WT.}\right)^2 + \left(\frac{\sigma_{CONC_{CS}}}{CONC_{CS}}\right)^2 + \left(\frac{\sigma_{VOL_{CS}}}{VOL_{CS}}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

R	=	carrier recovery
RE_R	=	relative error in recovery
WT_c	=	weight of carrier present in final precipitate
$CONC_{CS}$	=	concentration of carrier solution
VOL_{CS}	=	volume of carrier solution added
$\sigma_{PPT.WT.}$	=	standard deviation in weight of precipitate
$\sigma CONC_{CS}$	=	standard deviation in carrier concentration
σVOL_{CS}	=	standard deviation in carrier volume
PPT.WT.	=	weight of final carrier precipitate

Counting Uncertainty (CU) at the 95% Confidence Level

$$CU_{1.96\sigma} = \frac{1.96 * (\sigma_{NCR_S})}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

σNCR_S	=	standard deviation of the net sample count rate
EFF	=	detector efficiency
ALI	=	sample aliquot volume or mass
R	=	sample tracer/carrier recovery
ABN_S	=	abundance fraction of the emissions used for analyte identification/quantification
λ	=	analyte decay constant - ln 2/(half-life) (same units as the half-life used to compute λ)
t	=	time from sample collection to radionuclide separation or mid-point of count time (same units as half-life)
CF	=	other correction factors as appropriate (i.e., ingrowth factor, self-absorption factor, etc.)

C.7 Sample Activity Concentration Total Propagated Uncertainty (TPU)—Method Blank Corrected Sample Concentrations

$$TPU_{1\sigma} = \sigma_{ACT} = \frac{\sqrt{\sigma_{NCR_S}{}^2 + (NCR)^2 * (RE_{EFF}^2 + RE_{ALI}^2 + RE_R^2 + \Sigma\, RE_{CF}^2)}}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

EFF = detector efficiency
ALI = sample aliquot volume or mass
R = sample tracer/carrier recovery
ABN_S = abundance fraction of the emissions used for identification/quantification
σNCR_S = variance of the net sample count rate
NCR = net sample count rate
RE^2_{EFF} = square of the relative error of the efficiency term
RE^2_{ALI} = square of the relative error of the aliquot
RE^2_R = square of the relative error of the sample recovery
RE^2_{CF} = square of the relative error of other correction factors
λ = analyte decay constant - ln 2/(half-life) (same units as the half-life used to compute λ)
t = time from sample collection to radionuclide separation or mid-point of count time (same units as half-life)
CF = other correction factors as appropriate (i.e., ingrowth factor, self-absorption factor, etc.)

C.8 Sample Activity Concentration—Sample Concentrations without Blank Subtraction

$$ACT_B = \frac{NBCR_S}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

ACT_B = sample activity concentration without method blank subtraction
$NBCR_S$ = net sample background-corrected count rate
2.22 = factor for converting dpm to pCi
EFF = detector efficiency
ALI = sample aliquot volume or mass
ABN_S = abundance fraction of the emissions used for identification/quantification
R = sample tracer/carrier recovery
λ = analyte decay constant—in 2/(half-life) (same units as the half-life used to compute λ)
t = time from sample collection to radionuclide separation or mid-point of count time (same units as half-life)
CF = other correction factors as appropriate (i.e., ingrowth factor, self-absorption factor)

C.9 Net Sample Count Rate ($NBCR_S$) and σNBCRS—Sample Concentrations with Blank Subtraction

$$NBCR_S = \left(\frac{C_{GS}}{T_{GS}} \right)$$

$$\sigma_{NBCR_S} = \left[\frac{C_{GS}}{T_{GS}^{\ 2}} \right]^{\frac{1}{2}}$$

$NBCR_S$ = net background corrected count rate
C_{GS} = sample counts
T_{GS} = sample count time (minutes)
C_{SB} = background counts
T_{SB} = background count time (minutes)

C.10 Calculation of Minimum Detectable Concentration (MDC)- general formula

$$MDC = \frac{4.65 * \sqrt{b/T}}{K} + \frac{2.71}{K * T}$$

b = background count rate
T = sample count time (minutes)
K = instrument-specific and sample-specific correction factors (e.g., ALI * e-λt * R * EFFS * ABN_S)

NOTE: In using the above equation, the background and sample count times are either equivalent, or the background count time is greater than sample count time.

C.11 Calculation of Recovery and RER—Radiometric—Sample Concentrations without Blank Subtraction

$$R = \frac{\left(\frac{C_{GT}}{T_{GT}} - \frac{C_{TB}}{T_{TB}}\right)}{EFF * ABN_T * AMT_T} = \frac{NCR_T}{EFF * ABN_T * AMT_T}$$

R = tracer recovery
C_{GT} = gross count of tracer
T_{GT} = tracer count time (minutes)
C_{TB} = background count of tracer ROI
T_{TB} = background count time (minutes)
EFF = detector efficiency
AMT_T = amount of tracer activity added (dpm)
NCR_T = net count rate of tracer (cpm)
ABN_T = abundance fraction of the tracer emissions used for quantification of the tracer

C.12 RE_R—Radiometric—Sample Concentrations without Blank Subtraction

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 + \left(\frac{\sigma_{EFF}}{EFF}\right)^2 + \left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2$$

$$\left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 = \frac{\left(\frac{C_{GT}}{T_{GT}^2} + \frac{C_{TB}}{T_{TB}^2}\right)}{NCR_T^2}$$

$$\left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2 = \left(\frac{\sigma_{STS_T}}{STS_T}\right)^2 + \left(\frac{\sigma_{MASS_T}}{MASS_T}\right)^2 + \left(\frac{\sigma_{VOL_T}}{VOL_T}\right)^2 + \Sigma\left(\frac{\sigma_{DIL_T}}{DIL_T}\right)^2 + \Sigma\left(\frac{\sigma_{ALI_T}}{ALI_T}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

RE_R = relative error of the tracer recovery
σR = standard deviation of the tracer recovery
σNCR_T = standard deviation of the tracer's net count rate
σEFF = standard deviation of the detector efficiency
σAMT_T = standard deviation of the amount of tracer activity added
σSTS_T = standard deviation of the amount of tracer activity taken for stock tracer solution (provided with certificates received with standards)
$\sigma MASS_T$ = standard deviation of the mass of standard solution used to prepare stock tracer
$\sigma DILT_T$ = standard deviation of the volume(s) of the dilution(s) made to prepare the working tracer solution
σVOL_T = standard deviation of the volume of the stock tracer solution
σALI_T = standard deviation(s) of the aliquot(s) of tracer solution(s) diluted to prepare tracer working solution
C_{GT} = gross count of tracer
$T2_{GT}$ = square of tracer count time
C_{TB} = background count of tracer ROI
T^2_{TB} = square of background count time
R = tracer recovery
NCR_T = net count rate of the tracer
EFF = detector efficiency
AMT_T = amount of tracer activity added (dpm)
STS_T = amount of tracer activity (dpm) in stock tracer solution
$MASS_T$ = mass (grams) of standard solution used to prepare stock tracer solution
VOL_T = volume of tracer solution added
DIL_T = volume(s) of dilution(s) made to prepare the working tracer solution
ALI_T = aliquot(s) of trace solution(s) taken to prepare serial tracer solution dilution(s)

NOTE: Certificates, such as those from NIST, may give two or even three sigma uncertainty. Only one sigma should be used for σSTS_T.

C.13 Calculation of Recovery and RE_R—Gravimetric—Sample Concentrations without Blank Subtraction

$$R = \frac{WT_c}{CONC_{CS} * VOL_{CS}}$$

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{PPT.WT.}}{PPT.WT.}\right)^2 + \left(\frac{\sigma_{CONC_{CS}}}{CONC_{CS}}\right)^2 + \left(\frac{\sigma_{VOL_{CS}}}{VOL_{CS}}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

R	=	carrier recovery
RE_R	=	relative error in recovery
WT_C	=	weight of carrier present in final precipitate
$CONC_{CS}$	=	concentration of carrier solution
VOL_{CS}	=	volume of carrier solution added
σPPT.WT.	=	standard deviation in weight of precipitate
$\sigma CONC_{CS}$	=	standard deviation in carrier concentration
σVOL_{CS}	=	standard deviation in carrier volume
PPT.WT.	=	weight of final carrier precipitate

Counting Uncertainty (CU) at the 95% Confidence Level

$$CU_{B_{1.96\sigma}} = \frac{1.96 * (\sigma_{NBCR_S})}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

$\sigma NBCR_S$	=	standard deviation of the net background corrected count rate
EFF	=	detector efficiency
ALI	=	sample aliquot volume or mass
R	=	sample tracer/carrier recovery
ABN_S	=	abundance fraction of the emissions used for identification/quantification
λ	=	analyte decay constant - ln 2/(half-life) (Same units as the half-life used to compute λ)
t	=	time from sample collection to radionuclide separation or mid-point of count time (same units as half-life)
CF	=	other correction factors as appropriate (i.e., ingrowth factor, self-absorption factor)

C.14 Sample Activity Concentration TPU—Sample Concentrations Without Blank Subtraction

$$TPU_{B_{1\sigma}} = \sigma_{ACT} = \frac{\sqrt{\sigma_{NBCR_S}{}^2 + (NBCR_S)^2 * (RE_{EFF}^2 + RE_{ALI}^2 + RE_R^2 + \Sigma\, RE_{CF}^2)}}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

EFF	=	detector efficiency
ALI	=	sample aliquot volume or mass

R	=	sample tracer/carrier recovery
ABN_S	=	abundance fraction of the emissions used for analyte identification/quantification
$\sigma NBCR_S$	=	variance of the net background corrected count rate
$NBCR_S$	=	net background corrected count rate
RE^2_{EFF}	=	square of the relative error of the efficiency term
RE^2_{ALI}	=	square of the relative error of the aliquot
RE^2_R	=	square of the relative error of the sample recovery
RE^2_{CF}	=	square of the relative error of other correction factors
λ	=	analyte decay constant - ln 2/(half-life) (Same units as the half-life used to compute λ)
t	=	time from sample collection to radionuclide separation or mid-point of count time (same units as half-life)
CF	=	other correction factors as appropriate (i.e., ingrowth factor, self-absorption factor)

NOTE: For methods using a tracer or carrier, the inclusion of efficiency and recovery terms in the equation above may result in overestimation of the TPU.

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