WORKING COPY

VERIF. DATE: _____

INTIALS: _____

CP2-ES-5102/FR1A

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

FOUR RIVERS NUCLEAR PARTNERSHIP, LLC

This document is approved for public release per review by:

N/A

FRNP Classification Support _____ Date _____

Radiochemical Analyses Data Verification and Validation Paducah Gaseous Diffusion Plant, Paducah, Kentucky

Date Issued—December 2017

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

THIS PAGE INTENTIONALLY LEFT BLANK

APPROVALS

Radiochemical Analyses
Data Verification and Validation
Paducah Gaseous Diffusion Plant,
Paducah, Kentucky

CP2-ES-5102/FR1A

Lisa Crabtree
Environmental Monitoring Project Manager

12/12/17
Date

Curt Walker
Environmental Services Project Director

12/13/17
Date

DOE Approval Letter: N/A Date: N/A

Nuclear Safety Documentation: N/A

Non-intent changes per CDL- USQ not required

THIS PAGE INTENTIONALLY LEFT BLANK

REVISION LOG

REVISION NUMBER	DATE	DESCRIPTION OF CHANGES	PAGES AFFECTED
FR0	11/14/17	Bluesheet	ALL
FR1	12/13/17	Non-Intent Changes for Bluesheet Incorporation	ALL
FR1	7/6/2021	In accordance with the Corrective Action Plan for CAPA CA-003116, Action Item AI-0004735 and CAPA CA-003086, Action Item AI-0004709, the periodic review date for this procedure has been extended to December 13, 2022.	1
FR1A	12/13/2022	Periodic Review has been completed with no changes identified in procedure technical content. Nonintent changes have been incorporated per CP3-NS-2001. Date for review cycle has been reset	ALL

THIS PAGE INTENTIONALLY LEFT BLANK

CONTENTS

TABLES

THIS PAGE INTENTIONALLY LEFT BLANK

ACRONYMS

ANSI	American National Standards Institute
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	chain of custody
DOE	U.S. Department of Energy
DQO	data quality objective
EDD	electronic data deliverable
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
LLD	lower limit of detection
MD	mean difference
MDA	minimum detectable activity
MS	matrix spike
MSD	matrix spike duplicate
NIST	National Institute of Standards and Technology
NORM	naturally occurring radioactive material
QAPP	quality assurance project plan
QC	quality control
RL	reporting limit
RPD	relative percent difference
SDG	sample delivery group
SMO	Sample Management Office
SOW	statement of work
SRM	standard reference material
TPU	total propagated uncertainty
TRM	traceable reference material
WCRM	well characterized reference material
%R	percent recovery

THIS PAGE INTENTIONALLY LEFT BLANK

DEFINITIONS

NOTE 1: Qualifier 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 transformation that occur in a quantity of a radioactive nuclide per unit time.

AFFECTED SAMPLE RESULT—A sample result is considered to be affected when it is significantly influenced by a quality deficiency and is qualified accordingly through analytical data validation.

ANALYTICAL DATA VALIDATION—Analytical 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.

ANALYTICAL DATA VERIFICATION—Analytical 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.

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.

CALIBRATION VERIFICATION—Calibration verification, as described in this plan, is defined as a periodic evaluation of instrument standardization established during initial calibration. Using tolerance or statistical control charts, calibration verification can alert the instrument user of the occurrence of out-of-control instrumental conditions.

CARRIER—A carrier is a stable element/compound, introduced into the sample preparation/analysis process that will behave chemically similar to 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. The amount of the carrier recovered at the end of the analysis compared to that added initially is always used in the calculation of the final result.

CASE—A finite, usually predetermined number of samples, that have 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 (COC)—The history of the transfer of samples from the time of sample acquisition through archival and disposal of samples. COC documentation is required as evidence of sample integrity.

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 revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory. Correctable problems 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, as described in this plan, is defined as the statistical sample standard deviation, which is an approximation of the population standard deviation and is numerically defined as the square root of the number of counts obtained from a detector. This relationship holds true, provided that the distribution that the counts follows the Poisson distribution. Units for counting uncertainty are the same as for the reported result, minimum detectable activity (MDA), and total propagated uncertainty (TPU).

DATA QUALITY OBJECTIVES (DQO)—DQOs are qualitative and quantitative statements derived from the outputs of each step of the DQO 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 DQO process is a quality management tool based on the scientific method and developed by U.S. Environmental Protection Agency (EPA) to facilitate the planning of environmental data collection activities. The DQO 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.

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

LABORATORY CONTROL SAMPLE (LCS)—The LCS is a control sample of known composition. Aqueous and solid LCSs are analyzed using the same preparation, reagents, and method employed for field samples.

LABORATORY DUPLICATE (LCSD) —The laboratory control sample 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.

LOWER LIMIT OF DETECTION (LLD)—LLD is defined as the amount of analyte material that has a 95% chance of being detected when the decision that some amount of analyte is present is made when a signal occurs at or above the decision level. It has the same meaning as minimum detectable activity (MDA), which is preferred terminology.

MATRIX SPIKE (MS)—The MS 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 MS is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation. The purpose of a MS 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 (MDA)—The MDA is the amount of a radionuclide, which if present in a sample, would be detected with a probability of nondetection while accepting a probability of erroneously detecting that radionuclide in an appropriate blank sample. For this plan, the probabilities are both set at 0.05. As defined here, the MDA applies to the nominal concept of detection (i.e., specific to an instrument, radioanalytical method, and typical sample type).

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

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.

QUALITY-INDICATOR SAMPLE—Quality-indicator samples are those samples made ready in the laboratory that provide direct or indirect evaluation of the status of the analytical system and resulting data quality. Collectively, quality indicator samples are the LCS, laboratory duplicate, MS, and method blank.

REPORTING BATCH—A reporting batch is a group of sample results reported together in a single data package. The reporting batch may be comprised of samples prepared and analyzed together in the same preparation and counting batches or samples prepared and analyzed in different preparation and counting batches.

REPORTING LIMIT (RL)—The RL is a contractually specified detection limit that, under typical analytical circumstances, should be achievable.

SAMPLE DELIVERY GROUP (SDG)—An SDG 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 SDG.

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.

STANDARD REFERENCE MATERIAL (SRM)—A SRM is a material or substance of which one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. The SRM is characterized by the U.S. National Institute of Standards and Technology (NIST) or other certified testing authority, and issued with a certificate providing the results of the characterization.

STATEMENT OF WORK (SOW)—The validation SOW is a document prepared to function as the mechanism by which validation requirements are communicated from the project to the validation organization.

TOLERANCE CHART—For purposes of this plan, a tolerance chart is a representation of measured values with statistical descriptors, (i.e., mean and standard deviation limits) and is based upon maintaining a change of instrument response to a tolerance level judged acceptable to meet overall quality

requirements for the technique; a tolerance level should never be more restrictive than what is statistically possible.

TOTAL PROPAGATED UNCERTAINTY (TPU)—TPU is the addition of the square root of the sum of the squares of random components of the individual uncertainties, plus the magnitude of the estimated individual systematic relative uncertainties. TPU may include uncertainties introduced through field sampling and analytical laboratory procedures. For the purposes of this plan, TPU includes only those random and systematic uncertainties associated only with laboratory preparation and analysis. Refer to Appendix C for a full description of TPU.

$$TPU = \sqrt{\Sigma R_i^{\,2} + \Sigma S_j^{\,2}}$$

R = random components of individual relative uncertainties
S = magnitude of the estimated individual systematic relative uncertainties

TRACEABLE REFERENCE MATERIAL (TRM)—A TRM is a NIST prepared standard reference material or a sample of known activity or concentration prepared from a NIST standard reference material (derived standard material).

TRACER—A tracer is a radioactive isotope, introduced into the sample preparation/analysis process that will behave chemically similar to the analyte isotope(s). The tracer isotope is of the same element as the analyte isotope(s), except where the decay mode, half-life, or availability dictates the use of the isotope of a different element. The activity of tracer detected at the end of the analysis compared to that added initially is used in the calculation of the final result.

TURN-AROUND TIME—Turn-around 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 QUALIFIER—A qualifier is an alphabetic character 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 SOW is a document prepared to function as the mechanism by which validation implementation requirements are communicated from the project to the validation organization.

WELL CHARACTERIZED REFERENCE MATERIAL (WCRM)—The WCRM may be derived from a field sample that has been well characterized through multiple analyses, providing a high level of confidence of the concentration in the sample. The WCRM may be submitted to NIST for characterization and classification as a TRM.

1. INTRODUCTION

1.1. PURPOSE, SCOPE, AND APPLICATION

1.1.1 Purpose

This plan defines the minimum requirements, responsibilities, and methodology for radiochemical analyses data verification and validation processes for evaluating analytical data generated using industry standard measurement techniques.

This plan is applicable to radionuclide contaminants routinely analyzed by common radiochemical methods primarily in aqueous and soil/sediment matrices. It applies to environmental radiochemical data produced through methods that use instrumentation for detecting radioactivity. This plan is not applicable to mass spectrometric or fluorimetric methodologies.

Specifications in this plan should be incorporated into project documentation such as the quality assurance project plan (QAPP), into contractual statements of work (SOWs) between the project and the analytical laboratories, and into contractual validation SOWs between the project and the organization chosen to validate the data. If data validation is performed by individuals within the project, the SOW is not required, but a mechanism to specify data validation requirements is recommended. This plan shall be used as a baseline to create project-specific reports needed to perform radiochemical data verification and validation.

1.1.2 Scope and Application

This plan applies to radiochemical data verification and validation activities performed by the Sample Management Office (SMO) or its subcontractors.

2. RESOURCES

- Analytical Method
- Laboratory SOW
- Data Validation SOW
- Project-Specific QAPP

3. PREPERFORMANCE ACTIVITIES

The project manager shall ensure that individuals who perform radiochemical data verification and validation are knowledgeable of the latest version of this plan before beginning any activities.

4. GENERAL INFORMATION

4.1 REQUIRED ELEMENTS OF REVIEW AND VALIDATION

To the extent possible, all laboratory data packages will be produced by the laboratory performing the analysis as Level IV [i.e., U.S. Environmental Protection Agency (EPA) Stage 4] laboratory data deliverables. One hundred percent of the data deliverables will undergo a data quality review and validation comparable to a Level I validation (depending on analyte and method). As required by project specific requirements, the data review and validation effort may be increased to cover a Level II, Level III or a full Level IV validation of the data package. The activities included in the review and validation effort for each level are provided in Table 1.

Table 1. Required Elements of Review and Validation

Report Elements to be Reviewed*	**Level I**	**Level II**	**Level III**	**Level IV**
Cover/Signature Page	x	x	x	x
Table of Contents			x	x
Report Narrative	x	x	x	x
Executive Summary (if included)			x	x
Method Summary/Analyst Summary			x	x
Sample Summary/Sample Data Sheets	x	x	x	x
Shipping and Receiving Documents	x	x	x	x
Client Chain of Custody (COC)	x	x	x	x
Sample Receipt Checklist	x	x	x	x
Interlab COC (where applicable)		x	x	x
Internal COC (if required)			x	x
Glossary of Abbreviations	x	x	x	x
QUALITY CONTROL (QC) RESULTS				
QC Association Summary		x	x	x
Laboratory Chronicle			x	x
Surrogate and/or Tracer and Carrier Recovery Report		x	x	x
Blank Reports		x	x	x
Laboratory Control Sample (LCS) Reports		x	x	x
Matrix Spike (MS)/Matrix Spike Duplicate (MSD) and Duplicate Reports		x	x	x
Hold Times and Preservation Requirements	x	x	x	x
(Extended Data Deliverables/Forms)				
CLP-Like Organics				
SUMMARY FORMS			x	x
Summary Forms (Org I–X)			x	x
QC SUMMARY			x	x
QC Forms (Org I–IV,VIII)			x	x
SAMPLE DATA			x	x
Quant Rpt + Chro + spectra				x
STANDARDS DATA			x	x
Calibration Forms (VI–VII; for GC, VIII–X)			x	x
(Quant + Chro Follows Each Form Set)				x
QC DATA			x	x
Tune			x	x

Table 1. Elements of Review and Validation (Continued)

Report Elements to be Reviewed*	Level I	Level II	Level III	Level IV
Blank Form I			x	x
Blank Quant Rpt + Chro + Spectra				x
LCS/ Laboratory Control Sample Duplicate (LCSD) Form I			x	x
LCS/LCSD Quant Rpt + Chro + Spectra				x
MS/MSD Form I			x	x
MS/MSD Quant Rpt + Chro + sSpectra				x
GEL Permeation Data				x
Florisil Data				x
Logs—Instrument, Prep, Standard			x	x
CLP-Like Inorganics				
Cover Page			x	x
Sample Forms (I) (CLP-like)			x	x
Calibration + QC Forms (ex: II–XIV)			x	x
Instrument Data				x
Preparation Data				x
SHIPPING/RECEIVING DOCUMENTS				
Internal COC (if required)			x	x
Interlab COC (where applicable)			x	x
Client COC	x	x	x	x
Sample Receipt Checklist	x	x	x	x

*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 data package.

The requirements of the Level I and Level II review and validation effort will be referred to as "Data Verification" and will be performed by a member of the SMO. The requirements of the Level III and Level IV review and validation effort will be referred to as "Data Validation," and is typically performed by an entity external to the project. This can be an internal staff member who is not associated with the project, or it may be an independent third party external to Paducah. The following sections summarize the requirements of each type of review and validation efforts.

4.2 DATA VERIFICATION REQUIREMENTS

Data verification is defined as systematic process, performed either by the data generator (on-site or fixed-base laboratory) or by an entity external to the data generator, which results in evaluation of the completeness, correctness, consistency, and compliance of a data set against a standard or contract.

If data verification is performed by the data generator, a project-level surveillance must be established by which the performance of the data verification process is evaluated.

Data verification, at the project level, is conducted by an SMO representative to expedite the review process. If data verification is conducted independently of the data validator, it includes two activities. The first activity entails inventory of the data package to ensure compliance with the contract and SOW in terms of the required deliverables. The second activity entails various checks of the data quality to determine the need for qualification. This process is commonly referred to as the "contractual screen" and is the beginning of the data validation process in that it encompasses the review of the Level I and some Level II validation elements identified in Table 1. The data verifier will qualify data based on the review

and validation elements in accordance with Section 5 of this plan. If the data set is being reviewed and validated at the Level III or IV requirements, then the data verifier will provide a copy of the data verification checklist to the data validator to expedite the validation process, or the data validator will perform both the data verification and the data validation processes.

Data verification should provide a mechanism for problem resolution with the laboratory; it should not be exclusively an after-the-fact identification of noncorrectable deficiencies.

A data verification checklist is completed by the data verifier and takes, as input, the steps in this plan that are listed as "Data Verification." The data verifier shall complete Form CP3-ES-5003-F03, "Data Verification Checklist," in accordance with CP3-ES-5003, *Quality Assured Data,* for all Level II, III, and IV validations.

4.3 ANALYTICAL DATA VALIDATION REQUIREMENTS

Analytical data validation, including laboratory data review, is defined as a systematic process, performed externally from the data generator, which applies a defined set of performance-based criteria to a body of data to determine the quality of reported results. Data validation is not performed by the analytical laboratory. Data validation provides a level of assurance, based on a technical evaluation, that an analyte is present or absent and, if present, the level of uncertainty associated with the measurement. Analytical data validation for radiochemical methods includes a technical review of the laboratory data package specified in the laboratory SOW. Data validation incorporates an evaluation of sample custody, sample handling and preparation, holding times, instrument calibration, instrument performance, batch quality control samples (e.g., LCS), the identification and quantitation of target analytes, performance standards (e.g., surrogates, internal standards) and the effect QC performance and/or deficiencies have on the quality of analytical sample data.

A data validation report that includes the results of data validation activities must be completed by the data validator for Level III and Level IV data validation requests and takes, as input, the data verification checklist (or equivalent) and the steps in this plan that are listed as "Data Validation." Data validation requires that personnel performing it have the appropriate level of training and experience to ensure data review and qualification is completed in a reasonable manner and in accordance with industry practices. Professional judgment may be required when performing data validation. Where professional judgment is used, resulting in either qualification of data or data left unqualified, the rationale for the selection of this path will be fully documented in the data validation report. Documentation will include the following: citations from this plan, other industry standards, and/or the literature demonstrating the reasonableness of the evaluation.

The actions described in this plan must serve as the baseline for incorporation into project verification/validation activities. Project-specific procedures applying to analytical methods not covered in this plan must be reviewed and approved prior to use.

Implementation of this plan is expedited through the agreement of work to be performed by an analytical laboratory in the form of a project-specific laboratory SOW. Deliverable requirements specified in the analytical SOW must be consistent with the requirements of this plan and with the Basic Ordering Agreement contract with the laboratory.

The validation SOW must be written consistent with the requirements and specifications of this plan. The validation SOW is prepared by an SMO representative and communicated to the validation organization (for Level III and Level IV validations only).

The validation SOW will include as attachments full copies of the analytical data package, as well as an electronic data deliverable (EDD) in the form of a Microsoft Excel file. Placement of the data validation qualifier may be assigned by hand writing on the laboratory report form, initialed and dated, or electronically on provided EDDs in the Validation Code field. If data are not qualified during data validation, an equals sign ("=") shall be entered on the sample result or placed in the Validation Code field of the provided EDD.

Form CP3-ES-5003-F03, "Data Verification Checklist," (in accordance with CP3-ES-5003, *Quality Assured Data*) must be completed for every sample delivery group (SDG) that undergoes Level II, III, or IV data validation. In addition to the data verification checklist, a data validation report must be completed for every SDG that undergoes Level III or IV data validation.

5. PROCEDURE

NOTE: Refer to Appendix A for qualifier descriptions. Refer to Appendix B for qualification guidance due to multiple quality deficiencies. Refer to Appendix C for a listing of relevant equations to use with this plan.

The following is a step-by-step approach to implement analytical data verification and data validation activities. This approach is based on current industry accepted standards. Because changes to methodology and the referenced guidance documents are not within the data verifier's or data validator's control, the data verifier and the data validator should always follow the most current methodology and associated guidance documents referenced throughout this text to perform the review and validation of associated data.

5.1 VALIDATION STRATEGY AND SOW DEVELOPMENT

The project team, with input as needed from a quality assurance specialist and/or a representative of the 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 will define the DQOs and the framework for performing data validation. An SMO representative shall prepare a validation SOW to communicate data verification and validation requirements to the organization performing the work (for Level III and Level IV validation only).

5.2 CUSTODY OF SAMPLES AND SAMPLE DOCUMENTATION

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 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 the sample truly has been in custody from the field to the final result), an evaluation of field notes, laboratory records, and the COCs provides the best available indicator of sample traceability. A sample is defined as being under proper custody of any of the following conditions is met:

- The sample is within the possession of an authorized person (e.g., field personnel, laboratory personnel, etc.);
- 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: Data verification of sample documentation includes result report header checks for accuracy from the COC. If sample identity is in question, every attempt should be made to verify the true identity of each sample. When custody problems cannot be resolved, they will affect the defensibility of the sample.

5.2.1 Data Verification

The data verifier shall trace custody of all samples in the reporting batch from field sampling through receipt at the laboratory by reviewing the COCs. If the information is missing, 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, etc.), indicate the problem on the data verification checklist and provide this information to the data validator.

5.2.2 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 qualify associated results rejected "R."

Custody of Samples	Yes	No	NA
1. Does the data verification checklist or associated attachments in the data report indicate that samples are traceable?			

5.3 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, the result must have been generated within the prescribed holding time in order for the result to be considered definitive.

5.3.1 Deliverables

- Field Sampling Notes
- Field COCs
- Laboratory COCs
- Laboratory reports and/or raw data containing the following: dates of collection, preparation, and analysis for all samples, dilutions, and reextractions.

5.3.2 Criteria

Gross alpha and beta activity are subject to a holding time of six months, as specified by 40 *CFR* § 136.

Physical characteristics and matrix influences also must be considered when setting holding times. These characteristics must be considered when planning for data validation implementation.

Table 2 presents commonly analyzed radionuclides on the U.S. Department of Energy (DOE) complex that have characteristics affecting holding time establishment decisions. In all cases the data verifier or data validator shall always follow the most current methodology guidance for sample holding time, temperature, and preservation requirements.

Table 2. Physical and Matrix-Related Characteristic

Nuclide	Physical or Matrix-Related Characteristic
Anionic radionuclides	Volatile when placed in acid solution
H-3	Volatile
I-131	Volatile
Tc-99	Volatile

Table 3 presents commonly analyzed radionuclides on the DOE Complex that are particularly susceptible to holding time and reporting limit (RL) exceedances due to short half-life.

Table 3. Commonly Analyzed Short Half-Life Radionuclides

Nuclide	T*
I-131	8.04 days
Rn-222	3.82 days
Po-210	138 days
Sr-89	50.5 days

*Source: Kocher, D. C. Radioactive Decay Data Tables. DOE/TIC-11026

Half of the half-life shall be used to determine the impact of sample holding times on short-lived radionuclides unless otherwise addressed in the DQO process along with specific criteria for assessment.

When appropriate, or in absence of known preservation techniques, the preservatives, container types, and holding times listed in Table 4 shall be used for samples. It is recommended that samples of any matrix not be allowed to be stored in high temperature areas to prevent potential loss of volatile radionuclides.

Table 4. Holding Time, Preservation, and Container Requirements

Parameter	Matrix	Volume/Container	Preservation	Holding Time
Americium	Soil, vegetation, air filters	8 ounces/P, G	None	6 months
Americium	Water	1 L/P, G	HNO_3 to pH < 2	6 months
Carbon-14	Soil	4 ounces/P, G	4 ± 2°C	6 months
Carbon-14	Water	1 L/P, G	4 ± 2°C	6 months
Curium	Soil, vegetation, air filters	8 ounces/P	None	6 months
Curium	Water	8 ounces/P	None	6 months
Gamma Radionuclides	Soil	8 ounces/P, G	None	6 months
Gamma Radionuclides	Water	1 L/P, G	HNO_3 to pH < 2	6 months
Gross Alpha	Water	500 mL/P, G	HNO_3 to pH < 2	6 months
Gross Alpha/Beta	Soil	4 ounces/P	None	6 months
Gross Alpha/Beta	Water	200 mL/P, G	HNO_3 to pH < 2	6 months
Iodine-131	Water	P	None	8 days
Iodine-129	Soil	8 ounces/P	None	6 months
Iodine-129	Water	P	None	6 months
Iron-55	Soil	8 ounces/P	None	6 months
Iron-55	Water	P	HNO_3 to pH < 2	6 months
Lead-210	Soil	4 ounces/P, G	None	6 months
Lead-210	Water	1 L/P, G	HNO_3 to pH < 2	6 months
Neptunium-237	Soil, vegetation, air filters	4 ounces/P	None	6 months
Neptunium-237	Water	1 L/P	HNO_3 to pH < 2	6 months
Nickel-59	Soil	4 ounces/P	None	6 months
Nickel-59	Water and soil	1 L/8 ounces/P	None	6 months
Nickel-63	Soil	4 ounces/P	None	6 months
Nickel-63	Water and soil	1 L/8 ounces/P	None	6 months
Phosphorus-32	Soil	4 ounces/P	None	6 months
Phosphorus-32	Water	1 L/P	HNO_3 to pH < 2	6 months
Plutonium	Soil, vegetation, air filters	8 ounces/P, G	None	6 months
Plutonium	Water	1 L/P, G	HNO_3 to pH < 2	6 months
Polonium-210	Soil	4 ounces/P, G	None	6 months
Polonium-210	Water	1 L/P, G	HNO_3 to pH < 2	6 months
Promethium-147	Soil	4 ounces/P	None	6 months
Promethium-147	Water	1 L/P	HNO_3 to pH < 2	6 months
Radium-226/228	Soil	8 ounces/P, G	None	6 months
Radium-226/228	Water	1 L/P, G	HNO_3 to pH < 2	6 months
Radium-223	Water	P	None	6 months
Radium-224	Water	P	None	6 months
Radon-222	Soil	8 ounces/P	4 ± 2°C	6 months
Radon-222	Water	40 mL volatile bottle	4 ± 2°C, zero headspace	7 days
Sr-89/90	Soil	8 ounces/P, G	None	6 months
Sr-89/90	Water	1 L/P, G	HNO_3 to pH < 2	6 months
Technetium-99	Soil	8 ounces/P, G	None	6 months
Technetium-99	Water	1 L/P, G	HNO_3 to pH < 2	6 months

Table 4. Preservation and Container Requirements (Continued)

Parameter	Matrix	Volume/Container	Preservation	Holding Time
Thorium	Soil, vegetation, air filters	8 ounces/P, G	None	6 months
Thorium	Water	1 L/P, G	HNO_3 to pH < 2	6 months
Total Alpha Radium	Soil	8 ounces/P	None	6 months
Total Alpha Radium	Water	P	HNO_3 to pH < 2	6 months
Total Uranium	Soil	4 ounces/P, G	None	6 months
Total Uranium	Water	5 mL/P, G	HNO_3 to pH < 2	6 months
Tritium	Soil	8 ounces/G	None	6 months
Tritium	Water	120 mL/G	None	6 months
Uranium	Soil, vegetation, air filters	8 ounces/P, G	None	6 months
Uranium	Water	1 L/P, G	HNO_3 to pH < 2	6 months

P = polyethylene (preferred when acceptable); G = borosilicate glass with Teflon lined cap; L = liter
Source: U.S. Army Corps of Engineers, Louisville District, Louisville Radiological Guideline (Data Analysis and Validation Guidelines), version 4, 2004.

5.3.3 Data Verification

The data verifier shall trace custody of all samples in the reporting batch from field sampling through receipt at the laboratory by reviewing the COCs. If the information is missing, 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, etc.), indicate the problem on the data verification checklist and provide this information to the data validator.

5.3.4 Data Validation

The data validator shall review the COC forms and laboratory raw data to determine the elapsed time from sample collection through analysis. If samples have been analyzed within the prescribed holding time, no action is warranted.

If holding times are exceeded, qualify as follows:

- If the holding time has been exceeded by a factor of < 2, qualify detected compounds “J” and nondetected compounds “UJ.”

- If the holding time has been exceeded by a factor of > 2, qualify detected results “J” and nondetected results “R.”

The data validator shall review laboratory receiving records to determine if samples were received at the appropriate temperature and that proper preservative addition has resulted in the appropriate pH adjustment(s). If records demonstrate samples were received at the proper temperature and with the appropriate pH adjustment, no action is warranted.

If samples with radionuclides amenable to preservative with acid have not been acidified in the field but have been acidified in the laboratory prior to subsampling, qualification may not be necessary. The matrix and container type will not affect the radioactive characteristics of the radionuclides in the sample. For

this reason, neglecting to acidify samples prior to shipment to a laboratory should not necessarily result in qualification; however, as radionuclides will adhere to the container walls, acidification (of aqueous samples), either during the sampling event or at the laboratory prior to subsampling, is critical to ensure that all radioactive components are in solution and the representativeness of the sample is maintained.

If temperatures are exceeded and/or pH adjustment is incorrect, qualify as follows:

- If samples have not been preserved properly in the field or have been stored in an improper container, qualify results < minimum detectable activity (MDA) "UJ." Sample results ≥ MDA may not require qualification, but may be qualified "J" if necessary.

- If samples have not been preserved properly or have been stored improperly, qualify results < MDA "J" and qualify results ≥ MDA "J" only if it is suspected that improper preservation or container type have had the affected the presence of the radionuclide in the sample.

Holding Times and Sample Preservation				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Does the data verification checklist indicate that all samples were analyzed within the appropriate holding time?				J	UJ/R
2. Were all samples preserved properly?*				J	UJ

*Data may be qualified "R" if the reviewer determines the effect of improper preservation has had a significant effect on the accuracy of reported sample results.

5.4 INITIAL CALIBRATION

It is outside the scope of this document to prescribe calibration requirements for the laboratory. This section provides recommended frequencies, performance, and evaluation criteria based on existing American National Standards Institute (ANSI) standards. Initial calibration requirements should be found in the analytical methods used to generate the analytical data being reviewed. Decisions regarding calibration evaluation during data validation should be influenced by the strategy outlined in this section in order to provide a consistent approach to data evaluation with respect to calibration and to expedite the data verification and data validation processes.

5.4.1 Deliverables

- Initial calibration data for all detector systems
- Raw data (required for confirmation)

5.4.2 Frequency

Typically the initial calibration is required to be performed within one year (± 30 days) of the last initial calibration. Initial calibration must be performed before any samples are analyzed for radionuclides. Initial calibration is also required if any continuing (routine) calibration does not meet the required criteria.

5.4.3 Criteria

The following sections present the most common requirements for calibration information related to radiochemical analysis; however, the data validator will need to review the requirements of a specific

method and/or the laboratory method that is being reviewed and follow the requirements for that method when validating data. This may mean that the laboratory method will need to be obtained and reviewed prior to data validation. In all cases specific analytical method requirements for calibration should be used as the primary guidance when evaluating radiochemical data.

5.4.3.1 Check source statistics

The instrument calibration sources should provide adequate counting statistics (< 1% Poisson statistic uncertainty) over the time period for which the source is to be counted; however, the source shall not be so radioactive as to cause the following:

- Pulse pileups;
- Dead time that is significantly different from that to be expected from routine analysis; or
- Gain shift in the case of pulse height analyzer systems.

5.4.3.2 Radioactive sources

Commercially prepared and sealed standards shall not be used after their stated expiration dates, which are based on radionuclide half-life or physical form of the standard (e.g., sealed source or plated planchet). Standards prepared from certified radioactivity standards at the laboratory or those purchased without expiration dates should be recertified against a certified radioactivity standard source or replaced yearly.

When using a mixed gamma source, typically two radionuclides decay within 6 months, but the remaining 11 isotopes remain present in measureable quantities. The standard remains usable after significant decay of the 2 radionuclides, but still is subject to the recertification criteria stated in the above paragraph.

The standard source(s) used in initial calibration shall be National Institute of Standards and Technology (NIST) or International Atomic Energy Agency (IAEA)-traceable Standard Reference Materials (SRM), or equivalent; however, source(s) used in calibration verification are not required to be NIST traceable unless measurements of these sources are directly used in calculation of analytical sample data results.

5.4.3.3 Control criteria

The scope of this plan does not include prescriptive requirements for calibration; however, quality of analytical data is highly dependent on control of the calibration process. To facilitate a framework for defining control of the calibration process, the three following strategies may be incorporated dependent on what instrumentation is being used.

1. Tolerance charts may be established based on consideration of specific performance characteristics of the instrument and radiochemical method. The required precision of tolerance charts must never be more restrictive than that of a quality control chart.

2. Statistical quality control charts may be established based only on a level of confidence considered necessary for statistical quality control.

3. Fixed limits may be used by consideration of percent deviation from a known value. With some radiochemical methodologies (e.g., alpha and gamma spectroscopy), establishment of tolerance or

statistical quality control charts may provide unrealistic precision goals [e.g., 5% relative percent difference (RPD)] or may exceed a ± 3σ control chart limit, but still provide adequate instrumental precision. In these cases evaluation of measured values using a percent deviation approach may provide realistic evaluation of detector precision.

5.4.3.4 Establishment of control points

Establishment of control points for use with a tolerance or statistical quality control chart may be approached in two differing strategies: fixed range or moving range.

Fixed range control charts are established by acquiring a predetermined number of points with associated mean and standard deviation, and comparing subsequent data point acquisitions to those statistical descriptors. This allows for evaluation of instrumental control over time, but may not represent true precision over time.
Moving range control charts are established by acquiring a predetermined number of points with associated mean and standard deviation, and as subsequent points are acquired, they are included for an up-to-date evaluation of system precision. In using a moving range control chart, only the most recent 20 points are considered in establishing statistical descriptors. The use of moving range control charts allows for real-time evaluation of detector control, but does not allow for evaluation of detector control in relation to initial calibration.

5.4.3.5 Control of background

The control limits for check sources and backgrounds (where applicable) shall be established using a minimum of 20 sequentially measured data points. For extended background count periods, a series of at least 10 single background measurements are acceptable. No samples subject to these specifications may be counted until these warning and control limits have been established.

Background count time should be ≥ sample count time unless precluded by extended low-level sample count times. In this case, background count time may be < sample count time.

5.4.3.6 Geometry

With all methods of detection, the calibration counting geometry used must be the same as that used with the analytical samples.

5.4.3.7 Background subtraction

Calibration data must be background subtracted, whether data is used in generation of efficiencies, crosstalk, or resolution evaluation

5.4.3.8 Recalibration

Two potential justifications exist for recalibrating an instrument: (1) according to manufacturer's specifications and (2) according to failure in response to a calibration QC test.

Recalibration shall occur within the manufacturer's specifications or as defined by laboratory procedures. Recalibration shall occur after major components are changed or repaired in such a fashion that there is a potential effect on instrument performance. All defined performance criteria shall be reperformed as discussed in this section, including the creation of new control charts.

Recalibration is necessary in the event that the instrument/system has malfunctioned and the repaired equipment has responded to a calibration QC test in a fashion that the tolerance level of a control chart has been exceeded (i.e., the operating or response characteristics of the instrument/system have changed more than the tolerance/control limits permit). Detector calibration is verified according to frequencies that will satisfy contractual criteria and according to criteria defining the warrant of corrective action.

5.4.4 Data Verification

The data verifier shall verify the presence of required reporting forms. If they are not provided the data verifier shall contact the laboratory and request the information be provided. If these occurrences cannot be resolved with the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way in the data.

5.4.5 Data Validation

The data validator shall evaluate the control charts for out-of-control conditions and quality affected data "J" or "R" based on specific conditions in the counting batch.

When QC warning limits are exceeded, professional judgment should be used to determine if the limits are reasonable based on achievable instrument precision by calculation of the RPD. Affected data should be qualified "J/UJ" if warning limits are exceeded, and nondetects qualified "R" if the control limits are exceeded.

The data validator shall evaluate standard source age(s). If standard source(s) has/have aged greater than the expiration date on the certificate(s), affected sample results shall be qualified "J" or "R" using professional judgment. Indicate in the data validation report as to why data was qualified "R."

Standard Traceability				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Were standard certificates included?				--	--
2. Were standards traceable?				--	--

5.5 CONTINUING CALIBRATION

Continuing calibration requirements should be found in the analytical methods. The following subsections present the most common requirements for continuing calibration information related to radiochemical analysis; however, the data validator will need to review the requirements of a specific method and/or the laboratory method that is being reviewed and follow the requirements for that method when validating data. This may mean that the laboratory method will need to be obtained and reviewed prior to data validation. In all cases, specific analytical method requirements for continuing calibration should always be used as the primary guidance when evaluating radiochemical data.

5.5.1 Deliverables

- Initial calibration data for all detector systems
- Raw data (required for confirmation)

5.5.2 Frequency

Continuing calibration must be performed with each reporting batch at the frequency indicated by the analytical method being followed.

5.5.3 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, the laboratory should recount the check source to verify the out-of-control condition. If the recount again exceeds the control limit, 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, 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, reference must be made to quality control 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.

5.5.4 Data Verification

The data verifier shall verify the presence of required reporting forms. If they are not provided, the data verifier shall contact the laboratory and request the information be provided. If these occurrences cannot be resolved with the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist.

5.5.5 Data Validation

The data validator verifies that the continuing calibration sample has been analyzed at the appropriate frequency. The data validator shall evaluate the supplied continuing calibration results against the analytical method requirements for out-of-control conditions. Qualify affected data "J," "UJ," or "R" based on specific requirements for each analytical method.

Evaluate Standard Source Age(s). If standard source(s) have aged greater than the expiration date on the certificate(s), affected sample results shall be qualified "J" or qualified "R," using professional judgment. Indicate in the data validation report as to why data was qualified "R."

Calibration Verification				
Validation Step	Yes	No	NA	**Qualification Guidance**
1. Do control charts indicate out-of-control conditions?				J/R
2. Are standard source age(s) greater than the expiration date on the certificate(s)?				J/R

5.6 QUALITY-INDICATOR SAMPLES

Quality-indicator samples 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 total propagated uncertainty (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 and 2.58, 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. Table 5 provides examples of these decision-making factors, which are applied as decision-making tools through this plan.

Table 5. Examples of Confidence Levels for Qualification Decision Making

Confidence Level, %	Decision-Making Factor	Decision-Making Level
50	0.68	More conservative
67	1.00	
75	1.15	
90	1.645	
95	1.960	
99	2.575	Less conservative

Adapted from: Taylor, J. K., *Quality Assurance of Chemical Measurements*, Lewis Publishers, 1997.

Listed in this section is guidance for qualification for single-quality-indicator samples being outside control criteria based on a 95% and 99% level of confidence. Analytical samples shall not be rejected based on a singular quality control sample. Effects of other QC sample deficiencies must be considered to evaluate whether conditions are such to justify rejection of data. Appendix B provides analytical decision-making guidance for situations where multiple quality deficiencies are encountered.

The tests in this section are meaningful only if the radioanalytical method functions properly. If a method is deemed seriously out of control, the tests in this section are not appropriate, and no further data validation needs to be done; all results may be considered unusable.

Analytical samples and the respective quality indicator samples may be counted on differing detectors within the same detector system, provided that calibration is within appropriate control limits. However, quality indicator samples counted on differing detector systems from the analytical samples may not provide meaningful data, and qualification may be appropriate.

5.6.1 Total Propagated Uncertainty

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 providing 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.

In the event that not all the requested uncertainties are available, 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 $\pm 2\sigma$ counting uncertainty, and a $\pm 2\sigma$ TPU (factors for which are presented in Appendix C) and a minimum detectable activity.

5.6.2 Standard Traceability

Standards used in the preparation of QC samples (e.g., LCS, MS) or sample-specific spikes (tracers or carriers) shall be shown to be traceable to a reliable source (e.g., NIST, IAEA).

5.6.2.1 Deliverables

- QC data forms
- Standard certificates

5.6.2.2 Data verification

The data verifier shall verify the presence of required reporting forms. If they are not provided, the data verifier shall contact the laboratory and request the information be provided. If these occurrences cannot be resolved with the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist.

5.6.2.3 Data validation

The data validator shall verify the identity of standard(s) used in the relevant quality-indicator sample preparation and sample-specific spiking by tracing the standard control number from the certificate to sample preparation documentation.

If a standard is not traceable, a qualification is not required; however the data validator should place qualification code “E02” on affected data if noncorrectable deliverable deficiencies have occurred or sample data is affected by nontraceable standards. The qualification code must be placed on all sample data related to nontraceable quality-indicator samples.

5.7 LABORATORY CONTROL SAMPLE

The purpose of the LCS is to monitor the accuracy of the preparation and analysis of the analytical samples, provided that LCS is fully homogenized prior to preparation and analysis. To the extent possible, the LCS should be the same matrix type as the analytical samples.

5.7.1 Deliverables

- Traceable reference material certificates
- SRM certificates
- Well characterized reference material certificates
- Raw data (required for confirmation)

5.7.2 Frequency

LCS must be analyzed at a frequency of at least one per analytical batch for each matrix and analysis type. The LCS must be analyzed on the same detection system as the samples with which it was prepared.

5.7.3 Criteria

The laboratory is required to analyze an LCS for each analysis type reported in the SDG. The LCS matrix should be equivalent (as can be reasonably achieved) to that of the samples analyzed. It is recognized that the LCS matrix may not simulate that of some sample matrices. Exceptions should be made in cases of novel matrices (e.g., sludge, oil, biota).

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.

The spike in the LCS should be of a level near that of the analytical samples.

The LCS is at least 5 ×, but not greater than 20 ×, the RL with the following exceptions: For RLs of low activity, the analyte is at a level where the random counting error does not exceed 10% in the counting time required to attain the RL.

The measured results of the LCS are reported along with the known (reference) value.

The LCS % recoveries (%Rs) typically have acceptability 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 limits are recommended:

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

- For gross alpha/gross beta analysis, the 75–125% acceptance range is applicable when the analyte in the LCS is the same analyte used for the calibration curve. The %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.

5.7.4 Data Verification

The data verifier shall verify the presence of LCS results. If results are omitted from the laboratory report, the data verifier will contact the laboratory to obtain the omitted information. Upon receipt, this

information will be placed in the data package for delivery to the data validator. If LCS analysis was required but not performed, this is considered a noncorrectable problem and shall be indicated on the data verification checklist. As this is a contract compliance issue, the occurrence should be communicated to the SMO and the data validator on the data verification checklist.

5.7.5 Data Validation

The data validator shall verify that a LCS was analyzed at the appropriate frequency for each analysis type within the analytical batch. In addition, the data validator shall confirm that the LCS matrix was equivalent (or similar, to the extent possible) to the matrix of the samples analyzed. The %R for the LCS for all matrices must be within the applicable laboratory acceptance limits, or the recommended limits of this document if laboratory limits have not been established.

- If the LCS was not analyzed with the analytical batch, the data validator will need to use professional judgment to determine the effect this has on the data. In this case the data validator may determine that the data is not usable and reject the data, or the data validator may qualify statistically detected results "J" and statistically nondetected results "UJ."

- If the LCS %R < the lower acceptance limit, qualify statistically detected results "J" and statistically nondetected results "UJ."

- If the LCS %R > the upper acceptance limit, qualify statistically detected results "J" and apply no qualification to statistically nondetected results.

- If the LCS activity is within the required range (or for low level samples, the random counting error < 10%), apply no qualification.

Laboratory Control Sample				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Has at least one LCS been prepared with up to 20 samples?				J	UJ
2. Is the LCS the same matrix as the samples in the reporting batch?				NA	NA
3. Is the LCS recovery:					
%R < lower acceptance limit?				J	UJ
%R > upper acceptance limit?				J	NA
4. Is the LCS activity:					
within the required range (or for low level samples, the random counting error < 10%)?				NA	NA
not within the required range?				NA	NA

Note: Evaluate other QC prior to qualifying "R." See Appendix B for guidance on qualification for multiple QC deficiencies.

$$\frac{LCS_{meas.} - LCS_{\exp.}}{\sqrt{(TPU_{meas.})^2 + (TPU_{\exp.})^2}}$$

$LCS_{meas.}$ = measured LCS results
$LCS_{exp.}$ = expected result of LCS
$TPU_{meas.}$ = 1σ total propagated uncertainty of measured result
$TPU_{exp.}$ = 1σ total propagated uncertainty of expected result

5.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

The purpose of the MS is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation, provided that samples are fully homogenized prior to

preparation and analysis. The MS 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.

NOTE: For a MS that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification) and field test data in determining similarity. The reviewer also should use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the data package. The reviewer 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. Or the reviewer may determine that no samples are sufficiently similar to the sample used for the MS, and thus that only the field sample used to prepare the MS sample should be qualified.

5.8.1 Deliverables

- MS data
- Raw data (required for confirmation)

5.8.2 Frequency

The MS sample must be analyzed at a frequency of at least one per 20 field samples for each matrix and analysis type.

5.8.3 Criteria

MSs are run on a separate sample aliquot using the same analyte as that being analyzed whenever possible.

Due to difficulties in homogenization of solid samples for gamma analyses, a MS may not present useful information. MSs may present useful information for aqueous samples but should not be used for energies < 100 keV.

MSs 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.

The MS is added at a concentration of at least 5 but not > 20 × RL. In samples having known significant activity of radionuclides to be analyzed, more than 20 × RL may be added to minimize the effect of the sample activity on determination of spike recoveries.

The measured results of the MS are reported along with the known spike value. The MS %Rs typically have acceptability criteria established by the laboratory based on an average of MS data generated by the laboratory for a specific method. In the absence of laboratory established limits, the following limits are recommended:

- The MS %R acceptance range is established as the laboratory's established limits or at 100 ± 40% (60–140%) as per ER-SOW-394, whichever is stricter. For samples where the native sample activity is greater than 5 × the spiking level, the MS is not required to meet this criterion.

Consideration should be given to the similarity in matrix type among samples in the preparation batches. If the matrices differ notable (particularly in soil particle size), 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, qualify using guidance provided in Appendix B.

5.8.4 Data Verification

The data verifier shall verify that MS/MSD samples were analyzed at the required frequency and that results are provided for each sample. If results are omitted from the laboratory report, the data verifier will contact the laboratory to obtain the omitted information. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If results cannot be obtained or the frequency of analysis is not satisfied, these occurrences are considered noncorrectable problems and will be identified as such on the data verification checklist. As this is a contract compliance issue, the occurrence will be communicated to the SMO and the data validator on the data verification checklist.

5.8.5 Data Validation

If MS/MSD analysis was required but not performed, 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 a MS/MSD analysis in a data package that is performed on a parent sample that is not from the sample set being reviewed. This is commonly called a “batch QC sample.” Data validation will not be made based on batch QC that is generated from a sample that is not from the data set being reviewed. In this case, the LCS will be used to determine the accuracy and precision of the sample set.

The data validator shall verify that a MS was analyzed for each applicable analysis type within the analytical batch and that the MS ID is traceable to an original sample in the SDG. The data validator also shall verify the spike recovery is within the acceptance limits of the laboratory or within 60–140%, whichever is stricter.

For samples having known significant activity of the radionuclide to be analyzed, more than 20 × the RL may be added to minimize the effect of the sample activity on determination of spike recoveries.

The uncertainty associated with the preparation of the MS should be small (> one-third of required MS %R) compared to the MS %R.

- If a MS was not analyzed within the analytical batch, qualify statistically detected results “J” and statistically nondetected results “UJ.”

- If a MS recovery is below the lower acceptance range, qualify statistically detected results “J” and statistically nondetected results “UJ.”

- If a MS recovery is above the upper acceptance range, qualify statistically detected results “J” and apply no qualification to statistically nondetected results.

- If the MS activity is not within the required range, note the noncompliance in the report and apply no qualification to results.

For Level IV data validations only, verify MS recoveries are calculated correctly using the following equation:

$$MS\ \%R = \frac{|SSR - SR|}{SA} * 100$$

Where:

SSR = spiked sample result
SR = sample result
SA = spike added

Matrix Spike				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Has at least one MS been prepared with up to 20 samples?				J	UJ
2. Is the MS recovery:					
%R < lower acceptance range?				J	UJ
%R > upper acceptance range?				J	UJ
3. Is the MS activity not within the required range?				NA	NA
4. Verify MS recoveries are calculated correctly using the equation below (Level IV validations only).					

Note: Evaluate other QC prior to qualifying "R." See Appendix B for guidance on qualification for multiple QC deficiencies.

$$MS\%R = \frac{(SSR - SR)}{SA} * 100$$

SSR = spiked sample result
SR = sample result (unspiked)
SA = spike added

5.9 DUPLICATES

The purpose of a laboratory duplicate is to monitor the precision of the analytical method, provided the sample is fully homogenized prior to preparation and analysis. The laboratory duplicate is a randomly chosen split of an analytical sample into two aliquots prior to sample preparation. The laboratory duplicate is used to evaluate the reproducibility of the complete laboratory process.

Field duplicate samples are collected to monitor the overall precision of both the field and laboratory practices.

5.9.1 Deliverables

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

5.9.2 Frequency

Laboratory duplicate must be analyzed at a frequency of at least one per 20 field samples for each matrix and analysis type (including gamma isotopes).

Field duplicates are collected at a rate determined by project specific requirements, but in most cases the frequency is one per 20 field samples.

5.9.3 Criteria

To provide for relevancy of duplicate data, the sample chosen should have measureable activity (i.e., > MDA and 2σ counting uncertainty); however, the test provided in this section may be performed on results < MDA or 2σ counting uncertainty.

In analytical methods where no sample preparation or separation is performed (e.g., gamma spectrometry), a sample recount may be performed in lieu of a laboratory duplicate, although qualification under these conditions should be based on instrumental performance, as most gamma spectrometry entails minimal sample preparation.

A limitation that applies to duplicate analysis performed in solid sample matrices is that the acceptance criteria requirement for duplicates of solids should not be used to disqualify (reject) project sample analysis data because it is recognized that analysis results of laboratory-generated splits of solids can vary drastically due to matrix heterogeneity.

All laboratory-generated duplicates are traceable to the sample number of the original project sample. A member of the SMO will provide the ID of the field duplicate and its parent sample to the data validator so that a comparison of the two can be made.

The duplicate results satisfy the acceptance criteria established by applying the mean difference (MD) and/or RPD comparison. The MD acceptance criterion is ≤ 3. The RPD acceptance criteria for aqueous samples is ≤ 25% for laboratory and field duplicates The RPD acceptance criteria for solid samples for is ≤ 25% for laboratory duplicates and ≤ 50% for field duplicates. The following deviations are pertinent when applying RPD criteria:

- The RPD acceptance criteria becomes less exacting when the sample matrices are something other than water or soil; therefore, some deviations from the RPD criteria are allowable for nonroutine matrices. In such cases, the duplicate should be evaluated using the MD equation and professional judgment shall be used to qualify any samples.

- If one of the results is not statistically positive, the RPD is calculated using one-half RL value for the nonpositive radionuclide result.

5.9.4 Data Verification

The data verifier shall verify that field blanks were not analyzed as laboratory duplicates. If a field blank has been used, the SMO will be notified immediately to ensure timely corrective action. If reanalysis cannot be completed, this issue will be identified as noncorrectable on the data verification checklist.

The data verifier shall verify the presence of laboratory and/or field duplicate results. If they are not provided, or if the required frequency of analysis is not demonstrated in the laboratory deliverable, the data verifier will seek to obtain the missing information from the laboratory. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If the missing information cannot be obtained from the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist. As they are

contract- compliance related, all such occurrences shall be communicated to the SMO and to the data validator on the data verification checklist.

5.9.5 Data Validation

The data validator shall verify that a laboratory duplicate was analyzed for each matrix and analysis type in the SDG. The data validator shall verify that the duplicate sample ID is traceable to an original sample in the SDG for both laboratory and field duplicate analyses. Calculate the mean difference and determine if it is ≤ 3.

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

Where:

MD = the mean difference 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 combined propagated 1σ uncertainty of the original results (as a standard deviation)
σ_d = the associated combined propagated 1σ uncertainty of the duplicate results (as a standard deviation)

If one of the results is not statistically positive, calculate the MD by using one half RDL value for the nonpositive radionuclide result:

$$MD = \frac{\left|Positive\ \mathrm{Re}sult - \frac{1}{2}RL\right|}{\sqrt{\left(\sigma_{POS}^{\,2} + \tfrac{1}{2}RL^2\right)}}$$

Where:

MD = the mean difference of the duplicate results
Positive Result = the positive sample results (as pCi/g or pCi/L)
½ RL = one-half the appropriate RL (as pCi/g or pCi/L)
σ_{POS} = the associated combined propagated to 1σ uncertainty of the positive result (as a standard deviation)
½ RL^2 = the ½ RL value is the assumed uncertainty

If the MD is > 3, calculate the RPD and determine whether or not it is ≤ 20% for water and ≤ 30% for soils:

$$RPD = \frac{High\ \mathrm{Re}sult - Low\ \mathrm{Re}sult}{\left(Average\ \mathrm{Re}sult\right)} * 100$$

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

If one of the results is not statistically positive, calculate the RPD using one-half RL value for the nonpositive radionuclide result:

$$RPD = \frac{\left|Positive\,\mathrm{Re}sult - \tfrac{1}{2}RL\right|}{\left(Positive\,\mathrm{Re}sult + \tfrac{1}{2}RL\right)/2} * 100$$

NOTE: Action determinations for duplicate analyses results that do not satisfy the acceptance criteria must be carefully dealt with. Sample conditions may cause poor duplicate agreement (e.g., heterogeneity) but have no adverse effect or impact on the actual project sample analysis results. In such cases, the data validator can only qualify the sample and corresponding duplicate. It can be difficult for the data validator to determine why the duplicate results did not agree well based solely on reported results. Such conditions and expected causes typically are described by the laboratory in the case narrative section of the data package. In some situations, the data validator may need to apply professional judgment to determine how associated sample data should be qualified. Heterogeneity can be anticipated with sample matrices that include soil, solid wastes, and liquids with suspended solids or multiphases.

If a laboratory and/or field duplicate were analyzed for each analysis type in the SDG, the duplicate is traceable to a sample within the SDG and the MD and/or RPD criteria are met, apply no qualification.

If the MD and RPD have been calculated and one or both criteria (with consideration given to the matrix concerns) were met, the MD takes precedence over the RPD value.

If the MD is > 3 and/or the RPD is outside the prescribed matrix criteria, qualify statistically detected results "J" and statistically nondetected results "UJ."

The data validator will have to use professional judgment in applying the results of the duplicate analysis to all samples in the batch. If it can be definitely determined that the duplicate imprecision was due to analytical or heterogeneity problems that may have affected all other related sample analysis results in the same preparation or analysis batch, qualify associated sample results "J" or "UJ." The data validator should indicate in the data validation report as to why data was qualified.

Laboratory Duplicate				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Is the MD > 3 and/or the RPD outside the prescribed matrix criteria?				J	UJ
2. Is the duplicate imprecision due to analytical or heterogeneity problems that may have affected all other related sample analysis results in the SDG?				J	NA

Note: Evaluate other QC prior to qualifying "R." See Appendix B for guidance on qualification for multiple QC deficiencies.

$$\frac{S - D}{\sqrt{(TPU_S)^2 + (TPU_D)^2}}$$

S = sample result
D = laboratory duplicate result
TPU_s = 1σ total propagated uncertainty of the sample
TPU_D = 1σ total propagated uncertainty of the duplicate

5.10 BLANKS

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

5.10.1 Deliverables

- Blank results

- Sample preparation log and/or analytical run log
- Raw data for each blank (required for confirmation)

5.10.2 Frequency

Method blanks must be analyzed at a frequency of at least one per analytical batch of 20 samples or less. The frequency of field and equipment blank collection will be project specific.

5.10.3 Criteria

The method blank matrix should be equivalent (or similar to the extent possible) to that of the samples analyzed.

No detectable target radionuclide activity should be found in the blank (i.e., the activity should be less than the 2σ total propagated uncertainty and its MDA). The MDA of the batch blank should be < the RL, unless all samples in the batch are positive. If all sample results in the batch are > the RL, then the batch blank MDA should be < the activity of the least active sample in the batch. If all of the samples in the batch are < the RL, the activity of the blank should be < the MDA.

5.10.4 Data Verification

The data verifier shall verify the presence of the pertinent deliverables for blank analyses. If the required information is not present in the laboratory report, or if the frequency of analysis is not satisfied, the data verifier will contact the laboratory to obtain the omitted information. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If the information cannot be obtained, these occurrences are considered noncorrectable problems and will be identified as such on the data verification checklist. As this is a contract compliance issue, the occurrence should be communicated to the SMO and to the data validator on the verification checklist.

5.10.5 Data Validation

The data validator shall verify that a laboratory-generated blank was analyzed with each matrix and analysis type in the analytical batch.

NOTE: In the absence of blank data (or questionable blank results), it should be noted that sample data, which is free of unwanted interferences/activity and is representative of the sample matrices being evaluated, can provide useful information to assess analytical or detector contamination problems.

Verify that the blank matrix was equivalent (or similar, to the extent possible) to that of the project samples analyzed.

Verify that the measured blank activity is not statistically positive and < than its MDA and the reported MDA is < the RL.

If the method blank is < its MDA or < its 2σ counting uncertainty, or the method blank result is > its MDA with the sample result < its MDA, apply no qualification to results.

If both the method blank and sample results are statistically positive or are > their respective MDA, perform the following mathematical test to determine the significance of the contamination on the samples.

NOTE: This test is the standard statistical method of assessing differences between radioactivity

measurements and determining the significance of those differences. This test shall not be performed if the QC blank has been subtracted from the sample result.

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

Where:

MD = the statistical difference used to define the significance of the blank contamination on sample results

S = the sample result (as pCi/g or pCi/L)

B = the blank sample result (as pCi/g or pCi/L)

σS = the associated combined propagated 1σ uncertainty of the sample result (as a standard deviation)

σB = the associated combined propagated 1σ uncertainty of the blank sample result (as a standard deviation)

Determine the magnitude of the contamination interference by performing the following mathematical test:

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

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 spectroscopy, one of the common interferences is with radium-226 and uranium-235. In alpha spectroscopy, one of the common interferences is the thorium-229 tracer peak tailing into the thorium-230 energy region of interest.

If batch method blank analysis was not performed at the specified frequency, qualify statistically detected results “J” and apply no qualification to statistically nondetected results.

If both the blank and sample results are statistically positive or are > 3 and the sample and blank activity differ by a factor of 10 (i.e., ≥ 10), apply no qualification to statistically nondetected 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]. There are cases where the mean difference can be > 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 qualifier for such cases, the factor-of-10 criteria also are applied.

If both the method blank and sample results are statistically positive or are > their respective MDA and the MD is between 2 and 3 and the sample and blank activity differ by < a factor of 10, qualify statistically detected results “J.”

NOTE: 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). See Note above.

If both the method blank and sample results are statistically positive or are > their respective MDA and the mean difference is > 3 and the sample and blank activity differ by < a factor of 10, qualify statistically detected results "J."

If both the method blank and sample results are statistically positive or are > their respective MDA and the MD is between 0 and 2 and the sample and blank activity differ by < a factor of 10, qualify statistically detected results "UJ."

NOTE 1: 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).

NOTE 2: The typical statistical values for "MD" are 1.96 (at the 95% confidence interval) and 2.58 (for the 99% confidence interval). However, this guide 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.

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.

Method Blank				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Has at least one method blank been prepared with up to 20 samples?				J	NA
2. Is the method blank the same matrix as the samples in the reporting batch?				NA	NA
3. If the method blank and sample results are statistically positive or are greater than their respective MDA, and:					
• The MD > 3 and the sample and blank activity differ by 10?				NA	NA
• The MD = 2–3 and the sample and blank activity differ by < 10?				J	NA
• The MD > 3 and the sample and blank activity differ by < 10?				J	NA
• The MD = 0–2 and the sample and blank activity differ by < 10?				UJ	NA
4. Verify aliquot size, volume/mass of reagents, dilution, and counting times. (Level IV validations only)					

$$\frac{|S - B|}{\sqrt{(TPU_S)^2 + (TPU_B)^2}}$$

S = sample result
B = method blank result
TPU_s = 1σ total propagated uncertainty of the sample
TPU_B = 1σ total propagated uncertainty of the method blank

5.11 ELEVATED UNCERTAINTY

At some 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.

The data validator shall compare sample results and TPUs for each result. If the TPU is ≥ 80% of the sample result, the data validator should qualify detected results "J." Nondetected results do not require qualification.

Elevated Uncertainty				Qualification Guidance	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Is the TPU ≥ 80% of the sample result?				J	NA

5.12 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 %R of the tracer and 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 standard operating procedure in the validation of radiochemical data.

5.12.1 Deliverables

- Matrix spike data
- Raw data (required for confirmation)

5.12.2 Frequency

Chemical yields will be analyzed on each sample tested by a laboratory based on the isotopic specific method being followed for analysis.

5.12.3 Criteria

Chemical yield is evaluated through the %R of chemical species spiked into samples. It is evaluated radiometrically with a tracer and gravimetrically with a carrier. Each sample is spiked with either a carrier or tracer, and sample results are adjusted for yields ≥ 100%.

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.

The recovery range of isotopic tracers is typically 30–110%. The recovery range of stable carriers is typically 40–110%. If the laboratory has established limits based on instrument or method specific capabilities, the reviewer should take these into consideration.

Alpha spectrometry tracer criteria are full width half maximum (FWHM) for the tracer peak < 100 keV and/or the peak energy within ± 50 keV of the known energy.

Criteria for qualification shall be based on what magnitude of correction has been applied to the sample result (e.g., 20% recovery implies a sample result correction of 5), although a point of debate exists concerning usability of radionuclide data with yields near 0%.

Yield criteria also may be established from existing sample yield data from previous sampling at the site, if this data are available.

NOTE**:** Abnormally low chemical yields can cause a large uncertainty in affected sample results. Yields greater than expected (> 100%) can add negative bias of at least the amount greater than 100 and may indicate the presence of the radionuclide in the sample, contamination, or instrument problems.

5.12.4 Data Verification

The data verifier shall verify the presence of required reporting forms. If they are not provided, the data verifier shall contact the laboratory and request the information be provided. If these occurrences cannot be resolved with the analytical laboratory, they are considered noncorrectable problems and shall be identified in this way on the data verification checklist.

5.12.5 Data Validation

The data validator shall 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.

If the sample-specific tracer recovery is > 110% and < 120%, qualify statistically detected results "J" and statistically nondetected results "U."

If the sample-specific tracer recovery is > 120%, qualify statistically detected results "R" and statistically nondetected results "U."

If the sample-specific carrier recovery is < 40% but > 10%, or the tracer recovery is < 30% but > 10%, qualify statistically detected results "J" and statistically nondetected results "UJ."

If the sample specific carrier recovery or tracer recovery is < 10%, use professional judgment to qualify the result "R."

For alpha spectrometry data, verify the FWHM for the tracer peak is < 100 keV and/or the peak energy falls within ± 50 keV of the known peak energy. (Level IV validations only)

Chemical Yield—Tracers and Carriers				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Is yield reported for all samples and QC samples in the reporting batch?				NA	NA
2. Are percent recovery criteria satisfied for all yield results?				NA	NA
3. Was the sample tracer recovery > 110% and < 120%?				J	U
4. Was the sample tracer recovery > 120%?				R	U
5. Was the sample carrier recovery < 40% but > 10%, or the sample tracer recovery < 30% but > 10%?				J	UJ
6. Was the sample carrier or tracer recovery < 10%?				R	NA
7. For alpha spec data, verify FWHM for tracer peak to be less than 100 keV and/or peak energy within ± 50 keV. (Level IV validations only)					

5.13 NUCLIDE IDENTIFICATION AND QUANTITATION

The data validator shall verify whether a sample result is > its MDA.

Verify whether the sample result is > its 2σ counting uncertainty. If the sample result is < the MDA or < 2σ counting uncertainty and qualified "U" by the laboratory, apply no qualification.

If the sample result is < the MDA or < 2σ uncertainty and is not qualified "U" by the laboratory, notify the SMO to have the data reviewed by the laboratory.

Nuclide Identification and Quantitation				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
1. Are sample results < MDA and "U" qualified by the laboratory?				NA	NA
2. Are sample results less than 2σ counting uncertainty and "U" qualified by the laboratory?				NA	NA
3. For Level IV data validation only, if sample results are produced manually at a 10% frequency.					

5.14 SAMPLE RESULT RECALCULATION

Sample result calculation by the laboratory can be performed either by software processing or manual transcription. If sample results are produced primarily through software processing, the data system will be evaluated during audits. If sample results are produced primarily through manual transcription, then sample results will be evaluated by manual recalculation at a 10% frequency (for Level IV data validation only).

If sample results cannot be reproduced through manual recalculation, a member of the SMO shall contact the laboratory and request the appropriate information be provided. If the result is verified as incorrect, then a member of the SMO shall contact the laboratory to obtain corrected data sheets and Form I.

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 follows. If calculations are not in the method, the laboratory will be requested to provide sample calculations such that the data validator will be able to check any necessary calculated results as needed. The data validator shall apply qualifiers using professional judgment.

5.15 INSTRUMENT-SPECIFIC SAMPLE CONSIDERATION

The data validation process should take into consideration analytical method-specific requirements as well as instrument-specific requirements that might be applicable to the analytical result. There is always potential that analytical methods will vary from laboratory to laboratory and from instrument to instrument, so a copy of the laboratory specific method should be reviewed and utilized during data validation. The following subsections present several common instrument-specific items that should be reviewed during the data validation process.

5.15.1 Gas Proportional Counting

A relationship between standard weight and activity used to calculate sample-specific efficiencies is developed from the mass attenuation curve. A representative sample aliquot must be chosen to ensure the dissolved solid content of the sample falls within the mass range of the appropriate curve.

If the aliquot weight is outside the mass attenuation curve and not reanalyzed with a smaller aliquot, the data validator shall qualify results "J."

5.15.2 Gamma Spectrometry

The data validator shall verify radionuclide identification and quantitation by evaluating the following:

- Review sample-specific spectra for changes in energies positions of target radionuclides and for significant peak overlap.
- Where more than one isotope of a single naturally occurring radioactive material (NORM) series is reported, reported results for that series should demonstrate secular equilibrium.
- For soil samples, two peaks that almost always are observed are the 511 keV annihilation peak and the 1,460 keV peak of K-40. The appearance of these peaks at the respective energy, and the respective peak shape should be checked.
- If K-40 is quantitated in the analysis, the reported value should be checked against the activity expected in site's soil (if those data are available).
- For nuclides identified and/or quantitated with two or more gamma energies (i.e., 1,173 and 1,332 keV for cobalt-60), the count rate at each energy can be observed to ensure that the count rate at each respective energy is reasonable to confirm the presence of the isotope.
- Where isotopes are verified by software using several energies, ensure isotopes are not disqualified by low abundance energies when counting times limit detection for those energies.
- The data validator shall apply qualifiers using professional judgment.

5.15.3 Alpha Spectrometry

The data validator shall verify of radionuclide identification and quantification by evaluating the following:

- Target peaks should be in the energy range of interest.
- Peak tailing should not significantly overlap peaks at lower energies.

The data validator shall apply qualifiers using professional judgment.

Instrument-Specific Sample Consideration				**Qualification Guidance**	
Validation Step	Yes	No	NA	Detects	Nondetects
Gas Proportional Counting					
1. Is the aliquot weight outside the mass attenuation curve and not reanalyzed with a smaller aliquot?				J	NA
Gamma Spectrometry					

Instrument-Specific Sample Consideration				Qualification Guidance	
Validation Step	Yes	No	NA	Detects	Nondetects
2. Are there changes in energy positions or significant peak overlap?				*	*
3. Do the reported results for NORM series demonstrate secular equilibrium?				*	*
4. Verify peak shape of 500 keV annihilation peak and 1460 keV peak of K-40 for soil samples are below the lower acceptance limit?				*	*
5. Verify K-40 value against activity expected in site's soil, if available				*	*
6. Verify energy count rates for nuclides identified with two or more gamma energies to confirm presence of isotope.				*	*
7. Verify isotopes are not disqualified by software by low abundance energies when counting times limit detection.				*	*
Alpha Spectrometry					
8. Verify that target peaks are in the energy range of interest.				*	*
9. Verify that peak tailing does not significantly overlap peaks at lower energies.				*	*

*Apply qualifiers using professional judgment.

6. RECORDS

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

- Data Verification Checklist
- Data Validation Report (for Level III and Level IV validation only)
- Copies of qualified or unqualified results reports (if applicable)

7. REFERENCES

NOTE: The most current versions of the references listed below should be utilized when using this plan for the data review, verification and validation process.

American National Standard Calibration and Usage of Sodium Iodide Detector Systems, ANSI N42.12-1980, April 28, 1980.

American National Standard Performance Verification of Liquid-Scintillation Counting Systems, ANSI N42.15-1990, April 23, 1990.

Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides, ANSI N42.14, October 30, 1991.

Currie, L. A. *Limits for Qualitative Detection and Quantitative Determination, Application to Radiochemistry,* Analytical Chemistry. 40, 3 (1968).

Data Quality Objectives Process for Superfund, EPA/540/G-93/071, U.S. Environmental Protection Agency.

DOE Methods for Evaluating Environmental and Waste Management Samples, DOE/EM-089T, Rev. 2, October 1994.

EML Procedures Manual, HASL-300, 27th Edition.

FEMP Data Validation Procedure, SSOP-1004, June 23, 1993.

Guidance for the Data Quality Objectives Process, EPA QA/G-4, U.S. Environmental Protection Agency, August 1994.

Hazardous Waste Site Field Sampling Workshop, U.S. Environmental Protection Agency, Region IV, June 1993.

Kanipe, L. G., *Handbook for Analytical Quality Control in Radioanalytical Laboratories*, EPA-600/7-77-088.

Karnofsky, J., *Radiochemistry Data Validation*, Draft #4, November, 1993.

Knoll, G. F., *Radiation Detection and Measurement*, 2nd ed., John Wiley and Sons, 1989.

Laboratory Data Validation Guidelines for Evaluating Radionuclide Analyses, Rev. 05A, Science Applications International Corporation.

Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures Quality Assurance, Third Edition, EPA/570/9-90/008, U.S. Environmental Protection Agency, Washington, DC.

Measurement Quality Assurance for Radioassay Laboratories, ANSI N42.2, Rev. 12, June 1, 1993 (Draft).

Montgomery. D., and D. E. McCurdy, *Specifications for WEMCO Environmental Radioanalytical Services Contract*, October 23, 1992.

Multi-Agency Radiation Survey and Site Investigation Manual, Rev 1, U.S. Environmental Protection Agency, Washington, DC, June 2001.

Multi-Agency Radiological Laboratory Analytical Protocols Manual, EPA 402-B-04, U.S. Environmental Protection Agency, Washington, DC, July 7, 2011.

Paducah Gaseous Diffusion Plant Programmatic Quality Assurance Project Plan, DOE/LX/07-1269&D2/R2, U.S. Department of Energy, Paducah, KY.

Performance Criteria for Radiobioassay, ANSI N13.30, January 15, 1993.

Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, U.S. Environmental Protection Agency, Washington, DC.

Quality Assured Data, CP3-ES-5003.

Quality Systems for Analytical Services, Rev 2.9, U.S. Department of Energy, May 2013.

Radiochemical Data Validation Guidelines, EG&G Rocky Flats, Environmental Monitoring and Assessment Division, May 1990.

Radiochemical Determination of Gross Alpha and Gross Beta Particle Activity in Water, Radiochemical Procedures Manual, EPA-520/5-84-006, U.S. Environmental Protection Agency.

Standard Operating Procedure for Radiological Data Validation, MSO-SOP-12.1.2, EG&G Idaho National Engineering Laboratory, January 1993.

Taylor, J. K., *Quality Assurance of Chemical Measurements*, Lewis Publishers, 1987.

Upgrading Environmental Radiation Data, Health Physics Society Committee Report HPSR-1 (1980), EPA 520/1-80-012, U.S. Environmental Protection Agency.

APPENDIX A

DATA VALIDATION QUALIFIERS AND QUALIFICATION CODES

THIS PAGE INTENTIONALLY LEFT BLANK

DATA VALIDATION QUALIFIERS AND QUALIFICATION CODES

Data Validation Qualifiers

U Analyte compound or nuclide considered not detected above the reported detection limit.
J Analyte compound or nuclide identified; the associated numerical value is approximated.
NJ Analyte compound or nuclide presumptively present at an estimated quantity.
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 is not usable for its intended purpose.
= "Equals" sign, indicates that no qualifier is necessary.

Data Validation Qualification Codes

Blanks

B01 Sample concentration was ≤ the RL, and < 5× the blank concentration (10× for common contaminants).
B02 Sample concentration was > 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 RRF was < 0.05
C02 Initial calibration %RSD was exceeded
C03 Initial calibration sequence was not follows as appropriate
C04 Continuing calibration RRF was < 0.05
C05 Continuing calibration %D was exceeded
C06 Calibration or performance check was not performed at the appropriate frequency
C07 Calibration data not reported
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 RSD criteria were not satisfied
C14 Retention time of compound outside window
C15 Initial calibration % 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 CRI %R was below the lower acceptance limit
C22 CRI %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 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)

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

Laboratory Control Sample
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 and MS/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 (PEM) 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 GC/MS window
Q09 TIC result was not above 10× the level found in the blank
Q10 TIC reported as detect in another fraction
Q11 Common artifact reported as a TIC
Q12 No raw data were provided to confirm quantitation
Q13 MDA > RL
Q14 Inappropriate aliquot sizes were used
Q15 Sample result < MDA
Q16 Sample result < 2σ uncertainty
Q17 Negative result
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 RT outside calculated RT window
Q28 Neither RL or the SQL are reported for a nondetect result
Q29 SQL > RL
Q30 Compound detected at < 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 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
V05 Cleanup check not performed at the appropriate 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)

APPENDIX B

QUALIFICATION TABLES FOR MULTIPLE QUALITY DEFICIENCIES

THIS PAGE INTENTIONALLY LEFT BLANK

GUIDANCE FOR DATA QUALIFICATION DUE TO MULTIPLE QUALITY DEFICIENCIES

This appendix provides guidance in the qualification of data due to instances of multiple quality deficiencies. Quality deficiencies can be categorized based on 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 qualifier should not be placed on sample data until all quality deficiencies have been identified within the reporting batch.

These tables represent validation qualification decisions made based on radioanalytical data quality considerations only. Data quality needs (e.g., risk assessment, remediation technologies) must be considered when using the guidance in these tables. For example, quantitative data needs may necessitate that data be rejected due to multiple quality deficiencies, but qualitative data needs may indicate that the same data shall only be qualified estimated.

Table B.1 provides a mapping scenario for qualification guidance. For example, if the LCS shows a high bias, and chemical yield also is high, choose the letters "A" and "E" and reference Table B.2 for guidance on qualification based on those quality deficiencies. The differing separation lines in Table B.1 indicate relationships among the quality indicators. Double lines indicate an "and" function, in that any combination is possible. Thick lines indicate that a single QC sample can only indicate a bias in one direction.

"High" and "low" bias in Table B.1 represents the confidence level by which the decision has been made to qualify affected data.

Table B.1. Mapping Scenario for Qualification Guidance

Laboratory Control Sample		**Matrix Spike**		**Chemical Yield**		**Method Blank Contamination**	**Lab Duplicate Poor Precision**
High Bias	Low Bias	High Bias	Low Bias	High	Low		
A	B	C	D	E	F	G	H

Table B.2. Qualification Guidance for 2 Simultaneous Quality Deficiencies

Combination	Qualification	
	< MDA	≥ MDA
AC	None	R
AD	UJ	J
AE	None	R
AF	UJ	J
AG	None	J
AH	None	J
BC	UJ	J
BD	R	J
BE	UJ	J
BF	R	J
BG	UJ	J
BH	UJ	J
CE	None	R
CF	UJ	J
CG	None	R
CH	None	R
DE	UJ	J
DF	R	J
DG	UJ	R
DH	UJ	R
EG	None	R
EH	None	J
FG	UJ	J
FH	UJ	J
GH	UJ	J

Table B.3. Qualification Guidance for 3 Simultaneous Quality Deficiencies

Combination	Qualification	
	< MDA	≥ MDA
ACE	None	R
ACF	UJ	R
ACG	None	R
ACH	None	R
ADE	UJ	R
ADF	R	R
ADG	UJ	R
ADH	UJ	R
AEG	None	R
AEH	None	R
AFG	UJ	R
AFH	None	R
BCE	UJ	R
BCF	UJ	R
BCG	UJ	R
BCH	UJ	R
BDE	R	R
BDF	R	R
BDG	R	R
BDH	R	R
BEG	UJ	R
BEH	UJ	R
BFG	R	R
BFH	R	R
CEG	None	R
CEH	None	R
CFG	UJ	R
CFH	UJ	R
DEG	UJ	R
DEH	UJ	R
DFG	R	R
DFH	R	R
EGH	None	R
FGH	UJ	R

Table B.4. Qualification Guidance for 4 Simultaneous Quality Deficiencies

Combination	Qualification	
	< MDA	≥ MDA
ACEG	None	R
ACFG	UJ	R
ACEH	None	R
ACFH	UJ	R
ADEG	UJ	R
ADFG	R	R
ADEH	UJ	R
ADFH	R	R
BCEG	UJ	R
BCFG	R	R
BCEH	UJ	R
BCFH	R	R
BDEG	R	R
BDFG	R	R
BDEH	R	R
BDFH	R	R
CEGH	None	R
CFGH	UJ	R
DEGH	UJ	R
DFGH	R	R

APPENDIX C

RULES, CALCULATIONS, AND EQUATIONS

THIS PAGE INTENTIONALLY LEFT BLANK

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 less than 5, the preceding digit stays the same.
3. If the digit to be removed is equal to or greater than 5, the preceding digit is increased by 1.

Calculations/Equations

The equations in this appendix are provided as examples. It is likely that laboratory data will be reported with variations of these equations. These calculations should be regarded as guidance only.

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

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

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

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

C_{GS} = Sample Counts
T_{GS} = Sample Count Time (minutes)
CSB = Background Counts
TSB = Background Count Time (minutes)
CGB = Gross Method Blank Counts
TGB = Gross Method Blank Count Time (minutes)
CBB = Method Blank Background Counts
TBB = Method Blank Background Count Time (minutes)
EFFSD = Efficiency of the Sample Detector

EFFBD = Efficiency of the Method Blank Detector
RS = Sample Tracer/Carrier Recovery Fraction
R_B = Method Blank Tracer/Carrier Recovery Fraction

C.3 Uncertainty of Net Count Rate

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

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

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

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
CGT = Gross Count of Tracer
TGT = Tracer Count Time (minutes)
CTB = Background Count of Tracer [Region of Interest (ROI)]
TTB = Background Count Time (minutes)
EFF = Detector Efficiency Fraction
AMTT = Amount of Tracer Activity Added (dpm)
NCRT = Net Count Rate of Tracer (cpm)
ABNT = 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
σNCRT	=	Standard Deviation of the Tracer's Net Count Rate
σEFF	=	Standard Deviation of the Detector Efficiency
σAMTT	=	Standard Deviation of the Amount of Tracer Activity Added
σSTST	=	Standard Deviation of the Amount of Tracer Activity Taken for Stock Tracer Solution (provided with certificates received with standards)
σMASST	=	Standard Deviation of the Mass of Standard Solution used to Prepare Stock Tracer Solution
σDILT	=	Standard Deviation of the Volume(s) of the Dilution(s) Made to Prepare the Working Tracer Solution
σVOLT	=	Standard Deviation of the Volume of the Stock Tracer Solution
σ_{ALIT}	=	Standard Deviation(s) of the Aliquot(s) of Tracer Solution(s) Diluted to Prepare Working Tracer Solution
CGT	=	Gross Count of Tracer
T2GT	=	Square of Tracer Count Time (minutes)
CTB	=	Background Count of Tracer [Area or Region of Interest (ROI)]
T2TB	=	Square of Background Count Time (minutes)
R	=	Tracer Recovery
NCRT	=	Net Count Rate of the Tracer
EFF	=	Detector Efficiency
AMTT	=	Amount of Tracer Activity Added (dpm)
STST	=	Amount of Tracer Activity (dpm) in Stock Tracer Solution
MASST	=	Mass (grams) of Standard Solution Used to Prepare Stock Tracer Solution
VOLT	=	Volume of Tracer Solution Added
DILT	=	Volume(s) of Dilution(s) Made to Prepare the Working Tracer Solution
ALIT	=	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 σ_{STS_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
RER	=	Relative Error in Recovery
WTc	=	Weight of Carrier Present in Final Precipitate
CONCcs	=	Concentration of Carrier Solution
VOLcs	=	Volume of Carrier Solution Added
σ PPT. WT.	=	Standard Deviation in Weight of Precipitate
σCONC	=	Standard Deviation in Carrier Concentration
σVOL	=	Standard Deviation in Carrier Volume
PPT. WT.	=	Weight of Final Carrier Precipitate

Counting Uncertainty (CU) at the 95% Confidence Level

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

σ_{NCR_S}	=	Standard Deviation of the Net Sample Count Rate
EFF	=	Detector Efficiency
ALI	=	Sample Aliquot Volume or Mass
R	=	Sample Tracer/Carrier Recovery
ABNS	=	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

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

EFF = Detector Efficiency
ALI = Sample Aliquot Volume or Mass
R = Sample Tracer/Carrier Recovery
ABNS = Abundance Fraction of the Emissions Used for Identification/Quantification
σ NCRS = Variance of the Net sample Count Rate
NCR = Net Sample Count Rate
RE2EFF = Square of the Relative Error of the Efficiency Term
RE2ALI = Square of the Relative Error of the Aliquot
RE2R = Square of the Relative Error of the Sample Recovery
RE2CF = 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
NBCRS = Net Sample Background-Corrected Count Rate
2.22 = Factor for Converting dpm to pCi
EFF = Detector Efficiency
ALI = Sample Aliquot Volume or Mass
ABNS = Abundance Fraction of the Emissions Used for Identification/Quantification
R = Sample Tracer/Carrier 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.9 Net Sample Count Rate (NBCRS) and σNBCRS—Sample Concentrations with Blank Subtraction

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

$NBCR_S$ = Net Background Corrected Count Rate
CGS = Sample Counts
TGS = Sample Count Time (minutes)
CSB = Background Counts
TSB = Background Count Time (minutes)

σ_{NBCR_S}

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

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 * $e^{-\lambda t}$ * 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
CGT = Gross Count of Tracer
TGT = Tracer Count Time (minutes)
CTB = Background Count of Tracer Region of Interest (ROI)
TTB = Background CountTime (minutes)
EFF = Detector Efficiency
AMTT = Amount of Tracer Activity Added (dpm)
NCRT = Net Count Rate of Tracer (cpm)
ABNT = 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
σNCRT = Standard Deviation of the Tracer's Net Count Rate
σEFF = Standard Deviation of the Detector Efficiency
σAMIT = Standard Deviation of the Amount of Tracer Activity Added
σSTST = Standard Deviation of the Amount of Tracer Activity Taken for Stock Tracer Solution (provided with certificates received with standards)
σMASST = Standard Deviation of the Mass of Standard Solution Used to Prepare Stock Tracer Solution
σDILTT = Standard Deviation of the Volume(s) of the Dilution(s) Made to Prepare the Working Tracer Solution
σVOLT = Standard Deviation of the Volume of the Stock Tracer Solution
σALIT = Standard Deviation(s) of the Aliquot(s) of Tracer Solution(s) Diluted to Prepare Tracer Working Solution
CGT = Gross Count of Tracer
T2GT = Square of Tracer Count Time
CTB = Background Count of Tracer ROI
T2TB = Square of Background Count Time
R = Tracer Recovery
NCRT = Net Count Rate of the Tracer
EFF = Detector Efficiency
AMTT = Amount of Tracer Activity Added (dpm)
STST = Amount of Tracer Activity (dpm) in Stock Tracer Solution
MASST = Mass (grams) of Standard Solution Used to Prepare Stock Tracer Solution
VOLT = Volume of Tracer Solution Added
DILT = Volume(s) of Dilution(s) Made to Prepare the Working Tracer Solution
ALIT = 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 σ_{STS_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
RER	=	Relative Error in Recovery
WTC	=	Weight of Carrier Present in Final Precipitate
CONCCS	=	Concentration of Carrier Solution
VOLCS	=	Volume of Carrier Solution Added
σ PPT. WT.	=	Standard Deviation in Weight of Precipitate
σCONC	=	Standard Deviation in Carrier Concentration
σVOL	=	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
ABNS	=	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, etc.)

C.14 Sample Activity Concentration Total Propagated Uncertainty (TPU)—Sample Concentrations Without Blank Subtraction

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

EFF	=	Detector Efficiency
ALI	=	Sample Aliquot Volume or Mass
R	=	Sample Tracer/Carrier Recovery
ABNS	=	Abundance Fraction of the Emissions Used for Analyte Identification/Quantification
σNBCRS	=	Variance of the Net Background Corrected Count Rate
NBCRS	=	Net Background Corrected Count Rate
RE2EFF	=	Square of the Relative Error of the Efficiency Term
RE2ALI	=	Square of the Relative Error of the Aliquot
RE2R	=	Square of the Relative Error of the Sample Recovery
RE2CF	=	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.)

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.

THIS PAGE INTENTIONALLY LEFT BLANK