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THE NELAC INSTITUTE

ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 6: Quality Systems for Radiochemical Testing

TNI Standard

DRAFT Standard for Comment - v0

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PREFACE

This Standard is the result of many hours of effort by those volunteers on The NELAC Institute (TNI) Radiochemistry Expert Committee. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

This Standard supersedes and replaces preceding documents in whole or in part. It supplements Module 2, Quality Systems General Requirements, and may be used by any organization that wishes to implement a program for the accreditation of environmental laboratories.

Standard Revision History

Action	Date
Working Draft Standard Published	May 30, 2014
Modified Working Draft Standard Published	November 1, 2014
Voting Draft Standard Published	March 31, 2015
Interim Standard Published	August 14, 2015
Final Standard Released by Radiochemistry Committee	September 28, 2015
Approved as an American National Standard	December 6, 2016

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VOLUME 1, MODULE 6
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VOLUME 1, MODULE 6
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1.0 Radiochemical Testing

1.1 Introduction

This Standard contains detailed quality assurance (QA) and quality control (QC) requirements for environmental testing involving radiochemical measurements. The evaluation of laboratories for this discipline is in conjunction with a quality system as specified in the general requirements module. Adherence to quality systems requirements will ensure that all QC procedures specified in this module are being followed.

1.2 Scope

Essential QA and QC requirements for laboratories undertaking the examination of environmental samples by radiochemical analysis are defined in this Standard. Radioanalytical determinations involve detection of the radioactive emissions of the analyte (or indicative decay progeny) and tracer isotopes, often following their chemical separation from the sample matrix.

This Standard employs terms, definitions, and requirements from other documents, such as the Safe Drinking Water Act (SDWA)[1], Clean Water Act[2], or the Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual[3]. Additional QA and QC requirements (e.g., Measurement Quality Objectives (MQOs) as indicated in a method, regulation, or contract, or as established in the laboratory's quality system (if there are no established mandatory criteria), shall also be applicable and met by laboratories.

1.3 Terms and Definitions

The relevant definitions from TNI, Volume 1, Module 2, Section 3.0 apply. Definitions related to this document, which are used differently or do not exist in the above references, are defined below.

1.3.1 Additional Terms and Definitions

Activity, Absolute: Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time.

NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.

Activity, Areic: Quotient of the activity of a body of material and its associated area.

Activity, Massic: Quotient of the activity of a body of material and its mass; also called specific activity.

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[1] 42 U.S.C. §300f et seq. (1974), see http://www2.epa.gov/laws-regulations/summary-safe-drinking-water-act.
[2] 33 U.S.C. §1251 et seq. (1972), see http://www2.epa.gov/laws-regulations/summary-clean-water-act.
[3] EPA 402-B-04-001A (July 2004), *Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)*. Available at: https://www.epa.gov/radiation/marlap-manual-and-supporting-documents.

Activity, Volumic: Quotient of the activity of a body of material and its volume; also called activity concentration.

NOTE: In this module, unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.

Activity Reference Date: The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated.

NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.

Batch, Preparation: A Preparation Batch is composed of one (1) to twenty (20) environmental samples of the same quality system matrix that are prepared together with the same process and personnel, using the same lot(s) of reagents, with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours.

NOTE: Preparation Batches are only applicable for tests that require physical or chemical preparation that affects the outcome of the test.

Batch, Radiation Measurements (RMB): A Radiation Measurements Batch is composed of one (1) to twenty (20) environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameters, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last sample in an RMB is fourteen (14) calendar days.

Critical Value: Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level).

NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence ($1 - \alpha$) that the radionuclide is actually present in the material analyzed. For radiometric methods α is often set at 0.05.

Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance: Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25(c) as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).

Measurement Quality Objective (MQO): The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. Measurement Quality Objectives are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.

Minimum Detectable Activity (MDA): Estimate of the smallest true activity that ensures a specified high confidence, $1 - \beta$, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods β is often set at 0.05.

NOTE 1: The MDA is a measure of the detection capability of a measurement process and as such, it is an *a priori* concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample-specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold.

NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.

Test Source: A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.

Uncertainty, Counting: The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as Standard Uncertainty by means of the square root of observed counts) (MARLAP[3]). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).

Uncertainty, Expanded: The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty).

NOTE: Radiochemical results are generally reported in association with the Total Uncertainty or the Counting Uncertainty. Either of these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or an Expanded Uncertainty (k-sigma, where k > 1).

Uncertainty, Measurement: Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand[4].

Uncertainty, Standard: An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).

Uncertainty, Total: An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar terms (c.f., Counting Uncertainty).

1.3.2 Exclusions and Exceptions

The elements of this module apply to techniques used for the purpose of measuring or monitoring radioactivity, or techniques used to demonstrate compliance with regulations pertaining to radioactivity. The laboratory shall comply with the requirements of Volume 1, Module 4 in cases where technique-specific QA/QC is not defined in Module 6 (e.g., Mass Spectrometry [ICP-MS, TIMS] or Kinetic Phosphorimetry) or by the respective reference method (e.g., calibrations, calibration verifications, determinations of detection statistics, or method-specific QCs). The laboratory shall identify in its Quality System how and when it is complying with the requirements and elements of Volume 1, Module 4 and Module 6, as applicable.

[4] BIPM, JCGM 100:2008, *Evaluation of measurement data - Guide to the expression of uncertainty in measurement GUM* (1995 with minor corrections), Joint Committee for Guides in Metrology. Available at: http://www.bipm.org/en/publications/guides/gum.html.

1.4 Method Selection

Refer to Volume 1, Module 2, Sections 5.4.2, 5.4.3, and 5.4.4.

1.5 Method Validation

1.5.1 Validation of Methods

a) Prior to their acceptance and institution, methods for which data will be reported shall be validated across the range of physical and chemical parameters (e.g., density, Test Source composition, and analytical configurations) and activities that will be encountered in samples. Where applicable, the activity range shall include zero activity.

b) The laboratory shall validate the method in each quality system matrix for which it is applicable by demonstrating the method's detection capability, precision, bias, Measurement Uncertainty, and selectivity using the procedures specified in Sections 1.5.2 through 1.5.5.

c) The laboratory shall perform validation for each method for which documented data are not available to demonstrate that the above requirements are met. For reference methods, published data, if available, may be used to satisfy these requirements. For existing methods, QC data produced at the laboratory may be used to comply with validation requirements.

d) The laboratory shall record the quality system matrix used in the initial method validation and retain all supporting documentation for the initial study in a readily retrievable format for the lifetime of the method.

e) For all methods, the validation must comply with Volume 1, Module 2, Sections 5.4.5.1 through 5.4.5.3.

f) The laboratory shall document the results obtained, the procedure used for the validation, and a statement as to whether the method is suitable for the intended use.

g) The laboratory shall analyze for all methods, whenever available, externally-produced QC samples from a nationally- or internationally-recognized source (i.e., a national metrology institute, accredited TNI Proficiency Test (PT) Provider, an accredited ISO 17043:2010[5] PT Provider, an accredited ISO/IEC 17034:2016[6] reference material provider, or from an ANSI N42.22[7] compliant PT manufacturer). The laboratory shall evaluate the results of these analyses to determine its ability to produce acceptable data.

NOTE: The use of non-TNI accredited PT Providers is strictly for method validation purposes, and not for laboratory accreditation.

[5] ISO/IEC 17043:2010, *Conformity assessment - General requirements for proficiency testing;* International Organization for Standardization and International Electrotechnical Commission. Available from: http://www.iso.org/.
[6] ISO 17034:2016, *General requirements for the competence of reference material producers;* International Organization for Standardization and International Electrotechnical Commission. Available from: http://www.iso.org/.
[7] ANSI N42.22-1995, *American National Standard - Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control,* Institute of Electrical and Electronics Engineers, 1995 (2005). Available at: http://ieeexplore.ieee.org/.

1.5.2 Detection Capability

a) The laboratory shall establish the detection capability for each method/matrix combination. Detection Capability may refer to the Critical Value, MDA, or SDWA DL (terms defined in Section 1.3.1).

b) The laboratory shall document the procedure used to determine the detection capability.

c) The procedure a laboratory uses to determine the detection capability of a method must comply with the specific requirements of Volume 1, Module 6, Sections 1.5.2.1 and 1.5.2.2.

d) Method validation documentation shall include identification of software used for detection capability calculations and the software must conform to the requirements in Volume 1, Module 2, Section 5.4.7.2.

1.5.2.1 Minimum Detectable Activity (MDA) (see definition in Volume 1, Module 6, Section 1.3.1)

The laboratory shall utilize a method that is capable of providing an MDA that is appropriate and relevant for the intended use of the data (see Volume 1, Module 2, Section 4.4). The laboratory shall determine MDAs using the protocol specified in mandated methods. If no protocol is specified, the laboratory shall select a procedure that reflects instrument limitations and the intended application of the method.

a) Unless specified otherwise in the mandated method protocols, the laboratory shall include all sample-processing steps of the analytical method in the determination of detection capability.

b) The laboratory shall initially determine the detection capability of each method for the analytes of interest in a quality system matrix free of target analytes and interferences at levels that would impact the results.

c) The laboratory shall determine the detection capability each time there is a change in the test method or when there is a change in instrumentation that affects the analytical detection capability.

1.5.2.2 Required Detection Limit for Drinking Water Compliance (see definition in Section 1.3.1)

Laboratories performing radiochemical testing of drinking-water samples for SDWA compliance monitoring shall meet the requirements of 40 CFR 141.25(c). These laboratories shall use only approved methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141.25(c). The detection capability shall be expressed in terms of the DL as defined in Section 1.3.1 instead of Method Detection Limit (MDL) as defined in 40 CFR Part 136, Appendix B.

1.5.3 Evaluation of Precision and Bias

a) The laboratory shall compare results of precision and bias measurements determined during validation with criteria established by method, regulation, or contract, or as established in the laboratory's quality system (if there are no established mandatory criteria).

b) The laboratory shall utilize a method that provides precision and bias data for each of the analytes of interest that is appropriate and relevant for the intended use of the data (see Volume 1, Module 2, Section 4.4). Precision and bias shall be characterized across the range of activities that brackets those applicable in samples, including zero activity.

c) The laboratory shall process the validation samples through the entire measurement system for each analyte of interest and shall evaluate precision and bias in each relevant quality system matrix.

d) The laboratory shall determine the precision and bias of a method each time there is a change in the test method that affects the performance of the method or when a change in instrumentation occurs that affects the precision and bias.

e) Where there are no established criteria, the laboratory shall develop acceptance criteria for precision and bias based on one or more of the following:

i. intended use of the data;

ii. applicable regulations;

iii. guidelines in publications such as MARLAP[3], The Forum on Environmental Measurements Validation and Peer Review of U.S. Environmental Protection Agency Radiochemical Methods of Analysis[8], and/or The Fitness for Purpose of Analytical Methods, A Laboratory Guide to Method Validation and Related Topics[9].

1.5.4 Measurement Uncertainty

a) All radiochemical measurement results shall be reported with an estimate of Total Uncertainty expressed either as a standard deviation (i.e., a Standard Uncertainty) or a multiple thereof (i.e., an Expanded Uncertainty).

i. Total Uncertainty shall be documented by the laboratory's quality system consistent with the GUM[4], the recommendations in the MARLAP[3] Volume II Chapter 19, or other equivalent approaches.

ii. For purposes of compliance with the SDWA, or in order to comply with specific requirements established by method, regulation, or contract, or as established by the laboratory's quality system (if there are no established mandatory criteria), laboratories may report the Counting Uncertainty in lieu of the Total Uncertainty as specified in the appropriate method, regulation or contract, and as documented in the laboratory's Quality System.

b) The report shall clearly specify the type of uncertainty reported. The report shall:

i. express the uncertainty in the same unit of measurement as the measurement result unless the report clearly states otherwise;

ii. indicate whether the uncertainty is a Total Uncertainty or Counting Uncertainty;

iii. indicate whether the uncertainty is the Standard Uncertainty (i.e., "one-sigma") or an Expanded Uncertainty (e.g., "k-sigma"); and

iv. for Expanded Uncertainties, indicate the coverage factor (k) or the level of confidence.

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[8] *Validation and Peer Review of U.S. Environmental Protection Agency Radiochemical Methods of Analysis*; EPA Forum on Environmental Measurements (FEM), FEM Method Validation Team, FEM Document Number 2006-01, September 29, 2006. Available at: https://www.epa.gov/measurements/method-validation-and-peer-review-policies-and-guidelines.

[9] EURACHEM Guide. 2014. *The Fitness for Purpose of Analytical Methods, A Laboratory Guide to Method Validation and Related Topics*. Available at: http://www.eurachem.org/.

c) The results of the precision evaluation in Section 1.5.3 shall be compared to the uncertainty estimates as a check on the validity of the uncertainty evaluation procedures.

i. The experimentally-observed standard deviation from the initial precision evaluation at any testing level shall not be statistically greater than the maximum Standard Uncertainty of the measurement results at that level, although it may be somewhat less. If the experimentally-observed standard deviation at each testing level statistically exceeds the Standard Uncertainty, then the uncertainty estimate should be re-evaluated.

ii. A comparison of the experimentally-observed precision evaluation need not be performed for measurements that are required to be reported only with Counting Uncertainty per Section 1.5.4.a.ii except as directed by program/project-specific requirements or regulations.

1.5.5 Evaluation of Selectivity

a) The laboratory shall qualitatively evaluate selectivity, if applicable, by addressing the following sample and matrix characteristics:

i. the effect of matrix composition on the ability of the method to detect analyte;

ii. the ability of the method to chemically separate the analyte from the interfering analytes; and

iii. spectral and instrumental interferences.

b) The evaluation of selectivity may be accomplished by testing matrix blanks, spiked matrix blanks, worst-case samples, or certified reference materials. If applicable, a qualitative selectivity statement shall be included in the SOP.

1.6 Demonstration of Capability (DOC)

1.6.1 General

a) An individual who prepares and/or analyzes samples must have constant, close supervision until a satisfactory initial DOC is completed (see Section 1.6.2).

b) Thereafter, an ongoing DOC (Section 1.6.3) is required.

c) In cases where an individual has prepared and/or analyzed samples using a method that has been in use by the laboratory for at least one (1) year prior to applying for accreditation, and there have been no significant changes in instrument type or method, the ongoing DOC shall be acceptable as an initial DOC. The laboratory shall have records on file to demonstrate that an initial DOC is not required.

d) All demonstrations of capability shall be documented. All data applicable to the demonstrations shall be retained and readily available at the laboratory.

1.6.2 Initial DOC

An initial DOC shall be made prior to using any method and at any time there is a change in instrument type, personnel or method; or any time that a method has not been performed by the laboratory or analyst in a twelve (12) month period.

1.6.2.1 The laboratory shall document each initial DOC in a manner such that the following information is readily available for each affected employee:

a) analyst(s) involved in preparation and/or analysis;

b) matrix;

c) analyte(s), class of analyte(s), or measured parameter(s);

d) identification of method(s) performed;

e) identification of laboratory-specific SOP used for analysis, including revision number;

f) date(s) of analysis;

g) summary of analyses, including information outlined in Section 1.6.2.2.

1.6.2.2 If the method, regulation or contract does not specify an initial DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to initial DOC are adequate.

a) Prepare four (4) Test Samples consistent with Section 1.7.2.3. The analyst shall also prepare four (4) blank samples of clean quality system matrix in which no target analytes or interferences are present at activities that will impact the results of a specific method.

b) Where gamma-ray spectrometry is used to identify and quantify more than one analyte, the Test Sample shall contain gamma-emitting radionuclides that represent the low (e.g., ^{241}Am) and high (e.g., ^{60}Co) energy range of the analyzed gamma-ray spectra. As indicated by these examples, the nuclides need not exactly bracket the calibrated energy range or the range over which nuclides are identified and quantified.

c) The samples shall be prepared and analyzed according to the method.

d) Using all of the results, calculate the mean recovery of the spiked samples and the mean of the blank results in the appropriate reporting units and the standard deviations of the population sample (in the same units) for each parameter of interest. When it is not possible to determine means and standard deviation, the laboratory shall assess performance against established and documented criteria.

e) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy specified by method, regulation, or contract, or as established by the laboratory’s quality system (if there are no established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of field samples may begin.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, repeat the test for the parameters that exceed acceptance criteria. If test results fall outside acceptance criteria again, this confirms there is a general problem with the method and or measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all parameters of interest.

g) When an analyte not currently found on the laboratory’s list of accredited analytes is added to an existing accredited method, an initial DOC shall be performed for that analyte. When analytes are added to gamma-ray spectrometry, this is not required.

1.6.3 Ongoing DOC

1.6.3.1 The laboratory shall have a documented procedure describing ongoing DOC that includes procedures for how the laboratory will identify data associated with ongoing DOCs. The analyst(s) shall demonstrate ongoing capability by routinely meeting the QC requirements specified by the method, regulation, or contract, or as established by this Standard and by the laboratory's quality system (if there are no established mandatory criteria). If the method has not been performed by the analyst in a twelve (12) month period, an initial DOC (Section 1.6.2) shall be performed. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

1.6.3.2 This on-going demonstration may include one of the following:

a) Acceptable performance of blank(s) and sample(s) that have known, accepted values, single blind to the analyst.

b) Another initial DOC.

c) At least four (4) consecutive spiked samples (e.g., batch laboratory control samples) each with levels of precision and accuracy consistent with those specified in the method scope; and four (4) consecutive blank samples, each containing activity consistent with method performance specified in the method scope (e.g., generally activity less than Critical Value). The laboratory shall tabulate or be able to readily retrieve four (4) consecutive passing Laboratory Control Samples (LCS) and four (4) consecutive blank samples for each method for each analyst each year. The laboratory shall specify acceptable limits for precision and accuracy prior to analysis.

d) A documented process of reviewing ongoing QC samples by an analyst or a predefined group of analysts relative to the QC requirements specified by the method, regulation, or contract, or as established by this Standard, or by the laboratory's quality system (if there are no established mandatory criteria). This review should be used to identify patterns for individuals or groups of analysts and identify the need for corrective action or retraining as necessary; or

e) If a) through d) are not technically feasible, then analysis of real-world samples with results within predefined acceptance criteria (as defined by the laboratory or method) shall be performed.

1.7 Technical Requirements

1.7.1 Instrument Set-up, Calibration, Performance Checks, and Background Measurements[10]

This section addresses requirements for the proper set-up, calibration, calibration verification, and instrument performance checks of radiation measurement systems, as well as the requirements for subtraction background measurements and short-term background checks.

These requirements ensure that the measurements will be of known and appropriate quality for meeting regulatory and contractual requirements and for supporting decision making. This section does not specify detailed procedural steps for these operations, but establishes essential elements for selection of the appropriate technique(s). This allows flexibility and permits employment of a wide variety of analytical procedures and statistical approaches.

[10] One approach that addresses in detail all elements of this section is presented by ASTM International Standard Practice D7282-14, *Standard Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements*, ASTM, West Conshohocken, PA, 2014. Available at: http://www.astm.org.

a) At a minimum, the instrument QC program shall incorporate requirements imposed by the method, regulation, contract, or this Standard. Where imposed regulations are more stringent than this Standard, the imposed regulations take precedence (see Volume I, Module 2, Section 5.9.3.c. If it is not apparent which Standard is more stringent, the laboratory shall follow the requirements of the regulation or the method in that order. Where there are no established mandatory requirements, the laboratory shall incorporate guidelines consistent with MARLAP[3] or other consensus standard organizations.

1.7.1.1 Initial Set-Up of Instrumentation

a) The laboratory shall maintain the required radiation measurement systems for each method it performs. The laboratory shall set up radiation measurement systems to produce consistent, comparable results across multiple detectors used for a common method. The laboratory shall establish the configuration and operating parameters for each radiation measurement system used consistent with the method requirements.

b) The laboratory shall document radiation measurement system configuration and maintainable values for hardware- and software-related operational parameters prior to initial calibration. If a specific method or application requires that system configuration or operational parameters deviate from the manufacturer recommended specifications, the laboratory shall identify the modifications and document the rationale for such changes.

c) The laboratory shall periodically verify user-maintainable values for operational parameters to ensure their consistency with values recorded at the time of initial calibration to ensure the continued integrity of system configuration. If system configuration or operating parameters have changed, the laboratory shall perform corrective actions to determine and ameliorate any potential impact of the changes.

1.7.1.2 Initial Calibration

This section specifies the essential elements that define the procedures and documentation for initial calibration of radiation measurement systems.

a) Radiation measurement systems are subject to calibration prior to initial use and any time the following conditions occur:

   i. following replacement of a key detector element (e.g., a photomultiplier tube, silicon barrier detector, gas proportional detector chamber, germanium crystal, etc.);

   ii. after a repair, modification of system parameters, or other event (possibly unknown) when subsequent performance checks exceed predetermined acceptance criteria (i.e., limit of a statistical or tolerance control char or other QC parameters) indicating a change in performance since the initial calibration;

   iii. when indicated by corrective actions;

   iv. when calibration is due according to a predetermined frequency.

   The laboratory shall document the criteria that initiate (re)calibration in its SOPs.

b) Given that the instrument detection efficiency is linear with respect to count rate at all but the highest activity levels (i.e., where detection system dead time becomes significant), calibration curves with standards of varying activity need not be performed for radiometric techniques. Some techniques require multiple-point calibration curves to correlate a number of parameters other than activity. For example:

i. channel-energy calibration of alpha or gamma spectrometers;
ii. energy-efficiency calibration of gamma spectrometers;
iii. mass-efficiency (mass-attenuation) calibration of gas-flow proportional or x-ray detectors;
iv. quench-efficiency calibration of liquid scintillation detectors;
v. mass-crosstalk calibration of gas-flow proportional; and
vi. quench-crosstalk calibration of liquid scintillation detectors.

c) The laboratory shall base instrument calibrations on physical measurement of reference standards as defined in Section 1.7.2.6.c. These standards shall have general physical characteristics (i.e., geometry, density, composition, nuclear decay properties, etc.) that match as closely as possible those of the samples to which the calibration will be applied, except as noted in Section 1.7.1.2.d.

d) In some cases, calibration standard characteristics do not exactly match sample characteristics. The laboratory may use empirical techniques (e.g., gamma transmission) and/or computational techniques (e.g., Monte Carlo or efficiency modeling techniques) to generate corrections that are applied to calibrations performed with reference standards to account for minor differences between the physical characteristics of the calibration standard (i.e., geometry, density, coincidence-summing, etc.) and the samples to which the correction is to be applied, if:

i. the laboratory has performed a documented validation of the correction method or model by physical measurement of reference standards as defined in Section 1.7.2.6.c. The validation shall span the entire range of physical characteristics observed in samples to which the correction shall be applied (i.e., geometry, density, etc.);

ii. the applied correction consistently minimizes measurement bias across the range of physical characteristics; and

iii. the laboratory has estimated and validated the uncertainty associated with the correction (see Section 1.5.4) and included it in the uncertainty reported with each associated sample result.

e) The following items are essential elements of initial instrument calibration:

i. The laboratory shall establish and document, in written procedures and in records, the details of the initial instrument calibration. Details shall, at minimum, include:

a. the type of calibrations to be performed;
b. the number of calibration points required;
c. a description of the calibration standards required;
d. the preparation of the calibration standards;
e. the counting of the calibration standards;
f. the maximum permissible uncertainty for calibration measurements (e.g., a maximum relative combined uncertainty of the calibration parameter or a minimum number of counts collected); and
g. all calculations.

ii. Except in technically justifiable instances (e.g. prepared standard is dropped, physically marred, inconsistent distribution on the planchet, etc.), it is NOT acceptable to remove points from a calibration curve to meet established criteria. There must be some demonstrable reason to remove a point, and such removal must be approved by the Technical Manager or designee and documented.

iii. The laboratory shall establish criteria, appropriate to the calibration technique, for the acceptance of an initial instrument calibration in written procedures.

iv. If the initial instrument calibration results are outside established acceptance criteria, the laboratory shall perform corrective actions.

v. The laboratory shall retain sufficient raw data records to permit reconstruction of the initial instrument calibration.

f) The laboratory shall quantitate sample results only from the initial instrument calibrations unless otherwise allowed by regulation, method, or contract.

1.7.1.3 Calibration Verification

a) Prior to use of an initial calibration for analysis of samples, the laboratory shall verify the initial instrument calibration with a reference standard as defined in Section 1.7.2.6.c. The laboratory shall obtain the standard from a source or a lot independent of the reference standard used in the initial calibration, if available (or vendor certified different lot if a second source is not available). For unique situations where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples. The calibration verification may take two (2) forms:

i. performing a second set of calibration measurements to be compared to the initial calibration;

ii. quantifying a set of prepared standards using the initial calibration.

b) The laboratory shall specify the maximum permissible uncertainty for calibration verification measurements (e.g., the minimum number of counts collected for each measurement) in their SOPs.

c) The laboratory shall specify calibration verification acceptance criteria in their SOPs (e.g., for the relative combined uncertainty of the prepared standard recovery). If the criteria for the calibration verification are not met, the laboratory shall perform corrective action.

1.7.1.4 Instrument Performance Checks

Instrument performance checks measure and track the stability of key detector response-related parameters over time. The continuing validity of initial calibrations is established by demonstrating the stability of the detection system from the point of initial calibration to the time of the Test Source measurement.

a) The following are essential elements of instrument performance checks:

i. The check source used for instrument performance checks need not be a reference standard as defined in Section 1.7.2.6.c.

ii. The laboratory shall use the same check source for ongoing performance checks as the one in the preparation of the tolerance or control chart limits at the point of the initial calibration.

iii. The laboratory shall prepare, handle, seal and/or encapsulate check sources to prevent damage, loss of activity and contamination.

a. If the instrument performance check source becomes compromised (e.g. dropped and becomes damaged), in lieu of performing a new initial calibration the laboratory may confirm that the check source was actually compromised and document the investigation showing this. The current calibration must then be verified using an independent standard (e.g. Calibration Verification). If the veracity of the calibration is substantiated, the laboratory may employ a new check source with newly generated limits.

iv. The laboratory shall minimize the uncertainty of the check source count to allow detection of small changes in detector response relative to the acceptance criteria. The count duration and check source activity should be sufficient to provide adequate counting statistics over the life of the source.

v. Where significant, the radioactive decay in the check source shall be taken into account when evaluating count-rate sensitive parameters such as efficiency.

vi. The laboratory shall monitor the results of instrument performance checks using control or tolerance charts to ensure that instrument performance does not change significantly relative to the point of the initial calibration.

vii. The laboratory procedure shall specify what corrective actions are to be taken when performance check acceptance criteria are not met.

NOTE: If a performance check result exceeds established limits, instrument performance may have changed since the initial calibration. The laboratory should verify that the change is not attributable to normal statistical variability of the check measurement prior to taking corrective action.

b) The laboratory shall establish the minimum frequency for performance checks for specified calibration parameters as follows:

i. Gamma-ray spectrometry systems

Detection efficiency, energy calibration, and peak resolution:

a. Semiconductor detectors: At least twice weekly, but not on consecutive days, for a continuously operating detector; day of use for a non-continuously operating detector.

b. Scintillation detectors (e.g., sodium iodide): Day of use.

ii. Alpha-particle spectrometry systems

a. Energy calibration: Weekly.
b. Detection efficiency: Monthly.

iii. Gas-proportional and semiconductor alpha/beta detectors

Alpha and beta efficiency: Day of use.

iv. Liquid scintillation detectors

a. Manufacturer system calibration: At the frequency recommended by the manufacturer.

b. Efficiency with unquenched ^{3}H and ^{14}C standards: Day of use.

v. Solid-state scintillation detectors (e.g., zinc sulfide) used for non-spectrometric measurements

Efficiency: Day of use.

c) Exceptions to minimum frequencies for performance checks:

i. An individual Test Source may be uninterruptedly measured for a time longer than the required interval between performance checks to allow completion of the count of a Test Source as long as instrument performance checks performed at the beginning and end of the measurement period meet all applicable acceptance criteria.

ii. Test Sources may be uninterruptedly measured for a time longer than the required interval between performance checks to allow for completion of a Preparation Batch or RMB (Section 1.3.1) measured on an instrument with an automated sample changer (e.g., a liquid scintillation or gas proportional counter), as long as the period between the checks does not exceed seven (7) calendar days, and checks are done at the beginning and end of the measurement in question and meet all applicable acceptance criteria.

d) If the detection system is powered off between performance checks, a new performance check shall be performed prior to the next Test Source measurement.

1.7.1.5 Subtraction Background Measurements

Subtraction background measurements are performed to assess and correct for contributions due to cosmic radiation, naturally-occurring radioactivity, electronic noise, impurities in the detector, shielding, and source mounting material, or other sources that are not affected by the analytical processes. Contributions from impurities in the reagents, reference standards, or other sources introduced during the analytical processes are assessed with the use of method blanks (Section 1.7.2.2).

Numerous counting configurations may be used to determine subtraction background, depending on the detector and the method, including: counting an empty detector; counting an empty container or blank Test Source in a detector; or counting a container filled with a surrogate matrix material free of measureable levels of radioactivity.

a) The subtraction background shall be specific to each detector and appropriate to the method.

b) The subtraction background counting time shall be at least as long as the longest associated sample counting time and shall ensure a representative determination of the background rate.

c) The subtraction background measurement shall be accomplished in one of the following ways:

i. Paired measurements in which the subtraction background measurement is counted before or after the Test Source measurement or batch of Test Source measurements.

ii. Measurements performed at a fixed frequency, in which Test Sources may be measured between successive background subtraction measurements. In this case, the laboratory shall perform background subtraction measurements at the following minimum frequencies:

a. Gamma-ray spectrometry systems: Monthly.

b. Alpha-particle spectrometry systems: Monthly.

c. Gas-proportional and semiconductor alpha/beta detectors: Quarterly.

d. Liquid scintillation detectors.

- Individual quenched background: Once per Preparation Batch.
- Quenched background curve: According to frequency specified in laboratory procedures.

e. Solid-state scintillation detectors (e.g., zinc sulfide) used for non-spectrometric measurements: Prior to use.

NOTE: The frequency of subtraction background measurements may be increased from the above requirements when there is a low tolerance for unacceptable data due to failure of a subtraction background measurement.

iii. Composite measurements, in which the subtraction background is determined by combining background measurements collected in a manner that results in a representative determination of the background with a combined counting time at least as long as the longest associated Test Source count time. (See also Section 1.7.2.2.f)

d) The laboratory shall have written procedures for performing and evaluating subtraction background measurements. These procedures shall:

i. indicate the frequency and length of subtraction background measurements;

ii. establish control or tolerance charts and acceptance criteria of subtraction background measurements;

iii. ensure that the subtraction background measurement counts or count rate of a detector or an analytical region of interest is monitored for significant changes that introduce bias significant enough that could compromise the use of these measurements.

e) When the subtraction background has changed since the previous determination such that significant bias is imparted to intervening Test Source measurements, the laboratory shall initiate a corrective action. If the bias cannot be resolved, the laboratory shall qualify affected results.

1.7.1.6 Short-Term Background Checks

Short-term background checks, performed between subtraction background measurements, are QC measures used to verify the integrity of subtraction background measurements, check for possible detector contamination, electronics noise and to monitor each detector for trends and deviations from Poisson statistics. These background checks may be shorter in duration, yet more frequent than the subtraction background measurements, and therefore they may not always effectively identify every discrepancy that could compromise Test Source measurements (e.g., low-level contamination).

a) The laboratory shall have written procedures for performing and evaluating short-term background checks. These procedures shall:

i. Indicate the frequency and length of checks.

NOTE: Short-term background checks are performed after a predetermined number of samples, after a hot sample, or at a predetermined frequency. The frequency for the checks should be based on an evaluation of the laboratory instrument system and an acceptable rate for unacceptable data should short-term background check result fails. The frequency of these checks may be decreased if the laboratory is able to document that doing so does not result in an unacceptable rate of lost data. Conversely, the frequency should be increased when there is a high probability of the checks failing or there is a low tolerance for lost data due to failure of short-term background check.

ii. Establish control or tolerance charts and acceptance criteria of short-term background checks.

iii. Ensure that the short-term background counts or count rate of a detector or an analytical region of interest is monitored for significant changes that would indicate background bias significant enough that could compromise Test Source results.

b) Exceptions to minimum frequencies for short-term background checks:

i. An individual Test Source may be uninterruptedly measured for a time longer than the required interval between short-term background checks to allow completion of the count of a Test Source as long as short-term background checks performed at the beginning and end of the measurement period meet all applicable acceptance criteria.

ii. Test Sources may be uninterruptedly measured for a time longer than the required interval between short-term background checks to allow for completion of a Preparation Batch or RMB measured on an instrument with an automated sample changer (e.g., a liquid scintillation or gas proportional counter), as long as the period between the checks does not exceed seven (7) calendar days and the checks are done at the beginning and end of the measurement period and meet all applicable acceptance criteria.

c) When short-term background has changed since the previous determination, such that significant background bias is imparted to intervening Test Source measurements, the laboratory shall initiate a corrective action. If the bias cannot be resolved, the laboratory shall qualify affected results.

d) If subtraction background measurements are performed with sufficient frequency for a given method or detector type, such that they ensure background integrity and are capable of identifying detector contamination, the subtraction background measurements may be substituted for short-term background checks, in which case the short-term background checks shall not be required.

e) For liquid scintillation detectors, the laboratory shall check short-term unquenched background each day of use.

1.7.1.7 Contamination Monitoring

The laboratory shall have written procedures that address cases where radiation detectors have been contaminated, as determined by the subtraction background measurements, short-term background checks, or method blanks (Section 1.7.2.2). Detectors may not be brought back into service until corrective actions are completed.

1.7.2 Quality Control for Radiochemistry

1.7.2.1 General

a) The laboratory shall follow a documented QC program that monitors and assesses the performance of the laboratory's analytical systems. At a minimum, the QC program shall incorporate requirements imposed by regulation, methods and this Standard. Where imposed regulations are more stringent than this Standard, the imposed regulations take precedence (see Module 2, Section 5.9.3.c). If it is not apparent which requirement is more stringent, the laboratory shall follow the requirements of the regulation or the mandated method. Where there are no established requirements, the laboratory may reference guidelines consistent with MARLAP[3] or other consensus standard organizations in its quality system.

b) The laboratory shall process batch and sample-specific QCs to provide empirical evidence that demonstrates that the analytical system is in control. Results for these controls may be used to assess the data quality of sample results produced by the analytical system.

c) The laboratory shall employ either a sample Preparation Batch or an RMB to determine the grouping of samples and assignment of batch QC.

   i. A sample Preparation Batch shall be initiated where sample testing is performed that involves physical or chemical processing which affects the outcome of the test. Samples and associated QC assigned to a Preparation Batch shall be prepared together using the same processes, personnel, and lot(s) of reagents.

   ii. Where testing is performed that does not involve physical or chemical processing which affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors), an RMB may be initiated in lieu of a Preparation Batch. The samples and associated QC in the RMB shall share similar physical and chemical parameters, and analytical configurations (e.g., analytes, geometry, calibration, and background correction).

   iii. Samples may be added to the RMB for fourteen (14) calendar days from the start of the first sample count, or until twenty (20) environmental samples have been counted, whichever occurs first.

   iv. The laboratory may combine samples and associated QC within an RMB that share a range of physical and chemical parameters, and analytical configurations (e.g., analytes, geometry, calibration, density) that conform to the ranges of physical and chemical parameters, and analytical configurations demonstrated by method validation studies (see Section 1.5). Laboratory procedures shall document how method validation is performed, and laboratory records shall document any corrections (e.g., for efficiency, density, cascade summing, and background) applied to physical calibrations.

d) The laboratory's QC program shall document the frequency required for QCs. Minimum QC requirements are specified below.

e) The laboratory shall process all batch QC samples together with and under the same conditions as the associated samples, and shall use the same processes and procedures for preparation, analysis, data reduction and reporting of results.

   NOTE: Although samples in a Preparation Batch must be prepared together, they need not be analyzed concurrently on a single detection system, rather they may be analyzed on different detection systems as long as the detection systems are calibrated for the technique in question and instrument QCs indicate that the systems are in control.

f) The laboratory shall not systematically or preferentially use specific detectors, equipment or glassware for the analysis of QC samples. This should not preclude laboratories from segregating detectors, equipment, or glassware to minimize the risk of cross-contamination of samples or equipment as long as the criteria for segregation applies equally to batch QC samples and samples.

g) The laboratory's QC program shall document acceptance criteria for batch QC samples, sample-specific QCs, and for the evaluation of long-term trends and the methods used to establish these criteria.

h) The laboratory shall assess the results of the QC samples against acceptance criteria documented in the QC program. Where there are no established criteria in regulations, the method, or contract, the laboratory shall develop its acceptance criteria consistent with guidelines in MARLAP[3] or other consensus standards, or other criteria such as statistical control charts developed by the laboratory.

i) The laboratory shall track and trend the results of batch QC samples using statistical or tolerance control charts.

j) The laboratory shall investigate the cause when results do not meet acceptance criteria and take corrective actions to eliminate the source or minimize the magnitude of the problem. The laboratory shall consider samples associated with a failed QC parameter as suspect and shall, wherever possible, reprocess such samples. Where reprocessing is not possible, the laboratory shall report results with appropriate data qualifiers. The laboratory shall note the occurrence of a failed QC sample and any associated actions in the laboratory report.

1.7.2.2 Negative Control – Method Performance: Method Blank (MB)

The MB assesses the process of handling, preparation and analysis for cross-contamination and for low-level analytical bias. For methods with minimal physical treatment or no chemical processing (e.g., drying, grinding and homogenization of solid samples, or preparation of sample Test Sources for swipe or air filter samples for non-destructive gamma spectrometry or alpha-beta counting), the MB assesses sample handling and the analytical process.

a) The laboratory shall analyze a method blank at a minimum of one (1) per Preparation Batch or RMB.

b) The MB sample Test Source shall simulate quality system matrix characteristics that significantly affect results, such as geometry, size, and other factors, as appropriate.

i. The laboratory shall prepare the MB using materials that are free of analytes of interest at levels that will interfere with the evaluation of the results. If an analyte-free matrix is not available, the laboratory shall use a surrogate matrix to simulate the quality system matrix. For a RMB, the MB should be handled along with other samples during sample management (e.g. aliquotting, handling/transporting) when there is significant potential for contamination.

ii. The sample aliquot used for the MB shall be similar to that of routine samples. If the sample aliquot in a Preparation Batch varies (e.g., due to differences in sample density or restrictions on the activity or mass residue that may be processed), the laboratory shall use acceptance criteria that compensate for differing aliquot sizes (e.g., z-score per MARLAP[3], Vol. III, Chapter 18, Section 18.4.1).

c) The laboratory shall have procedures in place to determine if a MB result is significantly different from zero or impacts the analytical results. For example:

i. The MB exceeds the pre-established upper or lower bounds for the measurement, where the upper and lower bounds are plus x times the Standard Uncertainty and negative y times the Standard Uncertainty and x and y are the coverage factors for the confidence interval as established by the laboratory's quality system. The upper and lower bounds are not necessarily symmetrical.

ii. When applicable, the sample-specific MDA for the MB is greater than the required MDA.

d) Corrective actions shall be taken if it is determined that a MB result is significantly different from zero and associated sample results are less than five (5) times the MB activity, or if a MB result may impact the analytical results.

e) The laboratory shall evaluate results of MBs for long term trends, absolute bias, possible contamination, or interferences that may affect sample results.

f) The laboratory shall not subtract the batch MB from sample results in the associated Preparation Batch or RMB. The laboratory may subtract the average historical activity of MB measurements to address a demonstrated bias. The laboratory shall account for the uncertainty of the subtracted value in its estimate of uncertainty for the final result.

1.7.2.3 Positive Control – Method Performance: Laboratory Control Sample (LCS)

The LCS is used to evaluate the performance of the analytical system, including all preparation and analysis steps. For methods with minimal physical treatment and no chemical processing (e.g., drying, grinding and homogenization of solid samples, or preparation of sample Test Sources for swipe or air filter samples for non-destructive gamma spectrometry or alpha-beta counting), the LCS assesses the analytical process for bias.

a) The laboratory shall analyze a LCS at a minimum of one (1) per Preparation Batch or RMB. For RMBs, a calibration verification standard may be analyzed in lieu of the LCS.

b) The LCS Test Source shall simulate quality system matrix characteristics that significantly affect results, such as geometry, size or other factors.

i. The material used to create the LCS should be free of analytes of interest at levels that will interfere with the evaluation of the results. If an analyte-free matrix is not available, the laboratory may use a surrogate matrix to simulate the sample matrix. If analyte-free materials are not available for the LCS, the materials must be characterized and documented for the analyte(s) of concern and accounted for in the evaluation of the LCS.

ii. The aliquot used for the LCS shall be similar to that of routine samples. If the aliquot in a Preparation Batch varies (e.g., due to restrictions on the activity or mass residue that may be processed), the laboratory shall use acceptance criteria for samples that compensate for differing aliquot sizes (e.g., z-score per MARLAP[3], Vol. III, Chapter 18, Section 18.4.3).

c) For methods with minimal physical treatment and no chemical processing, the laboratory may prepare the LCS a single time and reuse the standard with subsequent batches of samples.

d) The laboratory shall spike the LCS at a level such that the uncertainty of the analytical result is less than one-third (1/3) of the acceptance criteria. For example, if it is required that the LCS result be within +/- 30% of the known value, the laboratory shall spike the LCS at a level such that the uncertainty of the analytical result is less than or equal to 10%. When practical, the LCS should be spiked at a level comparable to the action level if known; or at approximately ten (10) times the MDA; or that of routine samples if the activities are expected to exceed ten (10) times the MDA.

e) When available, the standard used to prepare the LCS shall meet the requirements for reference standards provided in Section 1.7.2.6.c. The final prepared LCS needs to have the activity and its uncertainty known; however, it need not be strictly traceable to a national standard organization. The LCS shall include all of the radionuclide(s) being determined with the following exceptions:

i. For methods that measure gross activity (e.g., gross alpha, gross beta), an appropriate surrogate analyte shall be used. This will generally be the radionuclide(s) used to calibrate the detector.

ii. For alpha spectrometry measurements, when multiple individual radionuclides with similar chemical characteristics are determined simultaneously with a single measurement and calibration, only one of the analytes/isotopes needs to be included in the LCS at the activity level indicated in Section 1.7.2.3.d.

iii. Where a non-destructive gamma-ray spectrometry measurement is made using a multi-point energy/efficiency calibration curve which covers the energy range of the analyte(s) of interest:

a. a radionuclide with similar gamma energies as those of the analyte(s) of interest may be used (e.g., ^{133}Ba may be used in place of ^{131}I); or

b. the LCS shall contain gamma-emitting radionuclides that, at a minimum, represent the low (e.g., ^{241}Am) and high (e.g., ^{60}Co) energy range of the analyzed gamma-ray spectra. As indicated by these examples, the nuclides need not exactly bracket the calibration energy range or the range over which radionuclides are identified and quantified.

f) The laboratory shall evaluate results of the batch LCS using a statistical technique such as the percent recovery or z-score that allows comparison to acceptance criteria documented in the laboratory QC program.

g) Where more than one (1) analyte is spiked at a level that meets the LCS requirements (see Section 1.7.2.3.d above), each shall be assessed against the specified acceptance criteria.

1.7.2.4 Sample-Specific QC Measures

a) The laboratory shall document procedures for determining the effect of the sample matrix on the analytical results. These procedures relate to the analyses of specific QC samples and are designed as data quality indicators for a specific sample using the designated method. Examples of sample-specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD), Matrix Duplicate (MD), Tracers, and Carriers.

b) The laboratory shall have procedures in place for tracking, managing, and handling sample-specific QC criteria including spiking radionuclides at appropriate activities, calculating recoveries, determining variability (e.g., relative percent difference and/or z-score), and evaluating and reporting results based on the performance of the QC samples.

c) Matrix Spike

i. MS recoveries are an indication of effects of the matrix on sample result accuracy using the selected method. The MS results are employed by the data user to determine if an MS issue has any impact on their related batch samples. MSs are not typically employed for non-destructive methods (e.g., gamma spectrometry or direct counting of samples for alpha or beta radioactivity), or for methods that employ a chemical yield tracer or carrier for each sample.

ii. The frequency of the analysis of MSs is specified by the method, a regulation or determined as part of the contract review process.

iii. The radionuclides spiked shall be as specified by the mandated method, regulation or as determined as part of the contract review process. At minimum, they will be consistent with those specified for the LCS in Sections 1.7.2.3.d and 1.7.2.3.e.

iv. The quantity of the aliquot used for MS shall be similar to that of routine samples analyzed in the Preparation Batch. If the sample size in the Preparation Batch varies (e.g., due to restriction on the activity or mass residue that may be processed), the laboratory shall apply appropriate corrections to compensate for differing aliquot sizes when applying the acceptance criteria for the batch.

v. When an MS is required, the lack of sufficient sample aliquot to perform an MS shall be noted in the laboratory report.

vi. The activity of the MS analyte(s) shall be greater than five (5) times the MDA.

vii. Acceptance criteria for MS recoveries shall be established as specified by the method, regulation or contract. Where there are no mandatory acceptance criteria established in the method, regulation or contract, the laboratory shall develop acceptance criteria based on industry practices and guidelines, or consistent with the guidelines of MARLAP[3] or other consensus standards. These criteria shall be documented or referenced in the laboratory's quality manual.

viii. When available, the standard used to prepare the MS shall meet the requirements for reference standard provided in Section 1.7.2.6.c. The final prepared MS needs to have the activity and its uncertainty known; however, it need not be strictly traceable to a national standard organization.

ix. The MS shall be prepared by adding a known activity of target analyte prior to performing any processes that affect the analyte of interest (e.g., chemical digestion, dissolution, ashing, separation, etc.).

d) Matrix Duplicates/Matrix Spike Duplicates/LCS Duplicates

i. A duplicate is defined as a second aliquot of the same sample taken through the entire analytical procedure. The results of this analysis provide indications of the measurement precision of the analyte for the specific sample using the selected method. Duplicate analyses provide a measure of precision when the target analyte is present in the sample chosen for duplication.

ii. Acceptance criteria for duplicates shall be established as specified by the method, regulation or contract. Where there are no mandatory acceptance criteria established in the method, regulation or contract, the laboratory shall develop acceptance criteria based on industry practices and guidelines, such as control charting developed by the laboratory, or consistent with the guidelines of MARLAP3 or other consensus standards. These criteria shall be documented or referenced in the laboratory's quality manual.

iii. At a minimum, the laboratory shall analyze one (1) MD per Preparation Batch or RMB. For RMBs, the MD may consist of a second measurement of one sample test source if insufficient sample is available for a second test source. The laboratory should prepare a second aliquot for samples with sufficient volume. If the batch is counted on more than one (1) detector, the MD shall be performed on a second detector.

iv. When samples have low-levels of activity (less than approximately three (3) times the MDA) the laboratory, at its discretion, may analyze MS/MSD to determine reproducibility within a Preparation Batch in place of a MD.

v. Based on specific project or program requirements or when there is insufficient sample available, the laboratory may choose to analyze a LCS in duplicate in place of a MD. The LCS and its duplicate will provide a measure of analytical precision. However, they will not provide information on matrix effects.

e) Chemical Yield Tracers and Carriers

i. For those methods that employ a radioactive Tracer or a stable Carrier as a chemical yield monitor in the analysis, each sample shall have an associated chemical yield calculated and reported. The chemical yield is one of the QC measures to be used to assess the associated sample result acceptance.

ii. The selection of a Tracer or Carrier shall not significantly interfere with the analyte(s) of interest nor cause bias in its measurements. When such a Tracer or Carrier is unavailable, the interference or bias caused shall be quantifiable and appropriate correction applied to the sample results.

iii. The Tracer or Carrier used to monitor chemical yield shall be added to the sample prior to performing any processes that affect the analyte of interest (e.g., chemical digestion, dissolution, ashing, separation, etc.) unless otherwise specified by the method.

iv. The chemical yield shall be assessed against acceptance criteria specified in the method, regulation, contract or laboratory SOP. The laboratory shall develop its criteria for data acceptance based on guidelines established in the MARLAP[3] or other criteria such as control charting developed by the laboratory. This assessment shall meet established project or program MQOs (Section 1.3.1).

v. When the established chemical yield acceptance criteria are not met, the specified corrective action and contingencies shall be followed. The occurrence of a failed chemical yield and the actions taken shall be noted in the laboratory report.

1.7.2.5 Data Reduction

a) The procedures for data reduction shall be documented.

b) Detection capability (e.g., MDA or Critical Level) shall be calculated as described in Section 1.5.2.

c) Measurement uncertainties shall be calculated and reported as described in Section 1.5.4.

1.7.2.6 Reagent Quality, Water Quality, and Checks

a) In methods where the purity of reagents is not specified, reagents shall be analytical reagent grade or better. Reagents of lesser purity than those specified by the method shall not be used.

b) The quality of water sources shall be monitored and documented and shall meet method specified requirements.

c) The QC program shall establish and maintain provisions for radionuclide standards.

i. Radionuclide standards shall be fit for purpose; they shall be of appropriate chemical and physical form and stability, and be traceable to a national metrology institute (NMI), with levels of uncertainty and purity that exceed those required to meet measurement quality objectives

ii. Reference standards used for instrument calibration shall be obtained from a national metrology institute (NMI), e.g. NIST in the USA or NPL in Great Britain, or from suppliers of NMI reference standards. Alternatively, reference standards may be obtained from reference material providers that conform to ANSI N42.22[7] or other standards or programs that require inter-comparison with a NMI similar to that specified in ANSI N42.22.

iii. **Reference materials used for quality control purposes may be obtained from provides described in 1.7.2.6.c.i above or from providers that are accredited to ISO Guides 17034:2016[6]** or 17043:2010**[5]**.Reference standards shall be accompanied with a certificate of calibration that meets the requirements of either ISO Guide 31:2015[11], or ANSI N42.22[7], Section 8, Certificates, and shall include at least the following information: manufacturer, radionuclides calibrated, identification number, calibration method, activities or emission rates with associated uncertainties and the confidence limits, standard quantity, activity reference time (date or time as appropriate to the half-life of the radionuclide), physical and/or chemical description of the source, and radionuclide impurities.

iv. Standards prepared or derived from externally-obtained reference materials shall be verified against an independent standard obtained from a second manufacturer prior to use for analysis of samples. The use of a standard from a second lot obtained from the same manufacturer is acceptable for use as a second source standard. Discrepancies between observed and expected values shall be investigated and appropriate measures taken that document the validity of standards prior to use.

v. The laboratory shall account for radioactive decay/ingrowth whenever decay/ingrowth has occurred between the Activity Reference Date and use that could impact use of the results.

vi. The laboratory shall have written procedures for handling, storing, and establishing expiration dates for reference standards.

vii. If there is no known provider of a particular standard (e.g., non-routine radionuclide or non-standard matrix) that is traceable to the International System of Units (SI), the laboratory may have no alternative but to use a standard with less rigorously established traceability. In this event, the laboratory shall obtain from the provider the minimum information described in Section 1.7.2.6.c.ii. The laboratory shall independently verify the activity of such standards prior to use and document the verification.

viii. If the laboratory's verification indicates a significant deviation from the original information from the provider, the standard should not be used unless the discrepancy can be resolved. If the standard is used for analysis of sample unknowns, the source and any other known limitations of the standard shall be disclosed in the final report.

1.7.2.7 Constant and Consistent Test Conditions

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[11] ISO Guide 31:2015, *Reference materials - Contents of certificates and labels*; International Organization for Standardization, 2015. Available from: http://www.iso.org/.

a) The laboratory shall assure that test instruments consistently operate within the specifications required of the application for which the equipment is used, according to Section 1.7.1.

b) Labware shall be cleaned to meet the sensitivity requirements of the method. Any cleaning and storage procedures that are not specified by the method shall be documented in the laboratory's quality system and records. Note that some applications may require single-use glassware.

c) The laboratory shall maintain a radiological control program that addresses analytical radiological control. The radiological control program shall explicitly define how low-level and high-level samples will be identified, segregated and processed to identify and minimize sample cross-contamination. The radiological control program shall include the measures taken to monitor and evaluate background activity or contamination on an ongoing basis.

1.7.3 Data Evaluation and Reporting

1.7.3.1 Negative Control – Method Performance: Method Blank (MB)

a) MB results shall be evaluated for long term trends, absolute bias, possible contamination or interferences that may affect results for samples in the batch.

b) MB acceptance criteria are discussed in Section 1.7.2.2 above. If acceptance limits are not met, corrective actions shall be taken to investigate the source of contamination or other bias. If sample activity levels are greater than five times the activity found in the MB, lacking other requirements, it is acceptable to report qualified results for the samples associated with the blank. Otherwise, reprocessing and reanalysis of the associated samples shall be required.

c) When sample results associated with a failed MB are reported, the failure and associated corrective actions, or inability to complete corrective actions, shall be noted in the laboratory report.

1.7.3.2 Positive Control – Method Performance: Laboratory Control Sample (LCS)

a) LCS recoveries shall be evaluated to assess the performance of the entire analytical system independent of the sample matrix. LCS results shall be calculated in percent recovery or other appropriate statistical measure that allows comparison to established acceptance criteria. The laboratory shall document the calculation.

b) LCS acceptance criteria are discussed in Section 1.7.2.3 above. An LCS that is determined to be within established acceptance limits effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch. Samples associated with an LCS that fails to meet acceptance limits are considered suspect and the samples shall be reprocessed and reanalyzed. If samples cannot be reprocessed and reanalyzed, the failure and associated corrective actions or inability to complete corrective actions shall be noted in the laboratory report.

1.7.3.3 Sample-Specific Controls

a) Matrix Spike, Matrix Duplicates, and Matrix Spike Duplicates

i. MSs and MDs allow evaluation of the effect of matrix on the accuracy and precision of results. Results from MSs shall be calculated as percent recovery or other appropriate statistical measure that allows comparison to established acceptance criteria. Results from MD and MSD precision shall be calculated as relative percent difference, z_{Rep} (see MARLAP[3], Vol. III, Chapter 18, Section 18.4.2), or other appropriate statistical measure that allows comparison to established acceptance criteria. The laboratory shall document the calculation of QC results.

ii. Acceptance criteria are discussed in Section 1.7.2.4 above. For results outside established criteria, corrective action shall be documented and the data reported with appropriate data qualifying codes. QC results outside acceptance limits shall be noted in the laboratory report.

b) Tracers and Carriers

i. For those methods that employ radioactive Tracers or stable Carriers as chemical yield monitors in each sample, results shall be expressed as percent yield or other appropriate statistical measure that allows comparison to established acceptance criteria.

ii. For alpha spectrometry, evaluation of Tracer acceptability shall include evaluation of chemical yield (e.g., uncertainty, variability) and peak resolution.

iii. Acceptance criteria are discussed in Section 1.7.2.4 above. Samples associated with Tracers or Carriers that fail to meet acceptance limits are considered suspect, and the samples shall be reprocessed and/or reanalyzed. If samples cannot be reprocessed and/or reanalyzed, the failure and associated corrective actions or inability to complete corrective actions shall be noted in the laboratory report.

1.7.3.4 Evaluation of Sample Results

d) Instrument raw data from energy spectral analysis shall be evaluated to ensure that the target radionuclides are quantified consistent with laboratory procedures and applicable MQOs, and that target radionuclides in the spectra are evaluated for possible interferences.

e) Results shall be reviewed for internal consistency, such as the presence of radionuclides consistent with known parent-progeny relationships and expected or likely decay series.

f) Sample-specific estimates of uncertainty and MDA shall be evaluated to ensure that MQOs have been met.

g) Sample-specific QC requirements (e.g. FWHM, centroid (energy), quench value or mass within calibration range, etc) shall be defined in the laboratory SOPs and/or client requirements and evaluated to ensure that samples meet method quality objectives (MQOs).

h) If these objectives have not been met, then samples shall be reprocessed and/or reanalyzed. If samples cannot be reprocessed and/or reanalyzed, the failure and associated corrective actions, or inability to complete corrective actions, shall be noted in the laboratory report.

1.7.3.5 Reporting Results

a) Reports delivered to the laboratory's client shall be consistent with the requirements of this Standard (Volume 1, Module 2, Section 5.10).

b) Following evaluation according to Section 1.7.3.4, results shall be reported directly as obtained, with appropriate units, even if the results are negative.

c) Results shall be expressed with an appropriate number of significant figures.

d) All radiochemical results shall be reported with an estimate of uncertainty, as discussed in Section 1.5.4.

e) Laboratories shall report the Activity Reference Date in association with all radiochemical measurement results.

f) Project- or client-specified reporting requirements can take precedence as described in Volume 1, Module 2, Section 5.10.

1.7.4 Sample Handling

1.7.4.1 While it may not be possible to physically verify all methods of preservation (e.g., addition of oxidizing or reducing agents), wherever practicable the laboratory shall verify that samples have been preserved in compliance with all applicable requirements specified by regulation, method, or contract, or as established in the laboratory's quality system (if there are no established mandatory criteria).

1.7.4.2 The laboratory shall document the required timing, methods for performing measurements to verify preservation, the acceptance range, or any other conditions indicating acceptable preservation.

a) Where thermal preservation of samples is required, the laboratory shall verify the temperature of samples upon receipt.

b) Where chemical preservation of samples is required, the laboratory shall verify that samples have been preserved using readily available techniques such as pH measurement prior to sample preparation or analysis.

1.7.4.3 If the results of the preservation verification do not satisfy established criteria, the laboratory shall initiate corrective actions (i.e., notification of the client, preservation of the sample at the time of discovery), and qualify all impacted test results in the report to the client.