ENVIRONMENTAL LABORATORY SECTOR

MODIFIED WORKING DRAFT STANDARD (MWDS)

This MWDS is a proposed revision of the 2012 Standard (EL-V1M6-2012). It has been prepared by the Radiochemistry Expert Committee. It will be presented to the membership and the public for discussion and input.

Note: The track changes in this document are the changes made since the publication of the Working Draft Standard on 5-30-14. There were numerous changes and additions to this Standard so a clean copy is presented to improve readability. Contact Ilona Taunton (ilon.a.taunton@nelac-institute.org) if you want a copy where tracking shows proposed changes from the 2012 Standard (EL-V1M6-2012) to the WDS.

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 6: Quality Systems for Radiochemical Testing

TNI Standard

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PREFACE

This Standard is the result of many hours of effort by those volunteers on The NELAC Institute (TNI) Quality Systems Committee and 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 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

<table>
<thead>
<tr>
<th>Action</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Working Draft Standard Published</td>
<td>January 14, 2007</td>
</tr>
<tr>
<td>Voting Draft Standard Published</td>
<td>June 15, 2007</td>
</tr>
<tr>
<td>Draft Interim Standard Published</td>
<td>December 15, 2007</td>
</tr>
<tr>
<td>Approved by Quality Systems Committee</td>
<td>December 22, 2007</td>
</tr>
<tr>
<td>Modified by Editorial Changes</td>
<td>March 12, 2009</td>
</tr>
<tr>
<td>Modified by Tentative Interim Amendments</td>
<td>June 15, 2009</td>
</tr>
<tr>
<td>Adopted by NELAP Board</td>
<td>September 8, 2009</td>
</tr>
<tr>
<td>Scheduled for Implementation by NELAP</td>
<td>July 1, 2011</td>
</tr>
<tr>
<td>Working Draft Standard Published</td>
<td>May 30, 2014</td>
</tr>
<tr>
<td>Modified Working Draft Standard Published</td>
<td>November 1, 2014</td>
</tr>
</tbody>
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VOLUME 1, MODULE 6

Quality Systems for Radiochemical Testing

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1.0 RADIOCHEMICAL TESTING

1.1 Introduction

This Standard contains detailed quality assurance and quality control requirements for environmental testing activities 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 quality control procedures specified in this module are being followed.

1.2 Scope

Essential quality assurance and quality control 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\(^1\), Clean Water Act\(^2\), or the Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual\(^3\). Additional quality assurance and quality control requirements (e.g., Measurement Quality Objectives (MQOs)) as indicated in a method, regulation, or contract, or as established in the laboratory’s quality management plan (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), or 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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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 Time: 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 time 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 systems matrix that are prepared and/or analyzed 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: A Radiation Measurements Batch (RMB) 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) and the maximum time between the start of processing of the first and last samples in an RMB is fourteen (14) days.

Batch, Analytical: For Module 6, Radiochemical Testing, the analytical batch is reserved for processes that do not involve physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, or alpha/beta counting of air filters or swipes on gas proportional detectors). The analytical batch is composed of one (1) to twenty (20) environmental samples that share similar characteristics and analytical configurations (e.g., analytes, geometry, calibration, and background corrections) and/or analyzed together using the same process. The maximum time between the start of processing of the first and last sample in the batch is fourteen (14) 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 $\alpha$ 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 $\alpha$ 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 ($1.96\sigma$ where $\sigma$ is the standard deviation of the net counting rate of the sample).

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 $\beta$ of false negatives below the critical value. For radiometric methods $\beta$ 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 \textit{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.

**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.

**Measurement Uncertainty:** Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand (GUM, JCGM 100:2008).

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

**Expanded Uncertainty:** 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 $k > 1$).

**Counting Uncertainty:** The component of measurement uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts) \textit{(after MARLAP)}. Older references sometimes refer to this parameter as \textit{Counting Error} or \textit{Count Error}. (c.f., Total Uncertainty).

**Total Uncertainty:** 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 \textit{Combined Standard Uncertainty} or \textit{Total Propagated Uncertainty}, and in some older references as the \textit{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 may choose to shall comply with corresponding sections of Module 4 in cases where technique-specific Quality Assurance/Quality Control (QA/QC) is not defined by 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 quality controls). The laboratory must identify in their quality management plan how and when they are complying with the requirements and elements of Module 4 and Module 6, as applicable.

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, and 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 is not available to demonstrate that the above requirements are met. For reference methods, published data, if available, may be used to satisfy these requirements.

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

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

f) The laboratory shall analyze for all methods, whenever available, externally-produced quality control samples from a nationally- or internationally-recognized source (i.e., a national metrology institute, accredited TNI proficiency test (PT) provider, an accredited ISO 17043 PT provider, or from an ANSI N42.22 or an accredited or ISO/IEC Guide 34 provider, or from an ANSI N42.22 compliant PT commercial vendor provider). The laboratory shall evaluate the results of these analyses on an ongoing basis to determine its ability to produce acceptable data.

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, Minimum Detectable Activity (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 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.

d) 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.
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 each method 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 Safe Drinking Water Act (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 detection limit (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

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 management plan (if there are no established mandatory criteria).

a) 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.

b) 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.

c) 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.

d) 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
1.5.4 Measurement Uncertainty

a) Each radiochemical measurement result shall be reported with an estimate of its total uncertainty expressed either as an estimated standard deviation (i.e., a standard uncertainty) or a multiple thereof (i.e., an expanded uncertainty).

i) Although the reported uncertainty should generally be an estimate of the total uncertainty of the measurement, for purposes of compliance with the Safe Drinking Water Act, or in order to comply with specific requirements established by method, regulation, or contract, or as established in the laboratory’s quality management plan (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 SOP. All other radiochemical measurements shall be reported with an estimate of the total uncertainty of the measured result.

ii) Total uncertainty shall be documented in the laboratory’s procedures or quality management program consistent with BIPM JCGM 100:2008: Guide to the Expression of Uncertainty in Measurement (GUM), the recommendations in the Multi-Agency Radiological Laboratory Analytical Protocols Manual Chapter 19 (MARLAP, Volume II, EPA 402-B-04-001B, July 2004), or other equivalent approaches.

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.

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. The experimentally-observed standard deviation at any testing level shall not be statistically greater than the maximum combined 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 combined standard uncertainty, then the uncertainty estimate should be re-evaluated.

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.

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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 performs any activity involved with preparation and/or analysis of 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 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) The analyte(s) shall be diluted in a volume of clean quality system matrix (a sample in which no target analytes or interferences are present at activities that will impact the results of a specific method) sufficient to prepare four (4) aliquots at a laboratory specified activity consistent with Section 1.7.2.3. The analyst shall also prepare four (4) blank samples of clean quality system matrix.
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 laboratory control sample shall contain gamma-emitting radionuclides that represent the low (e.g., $^{241}$Am), medium (e.g., $^{137}$Cs), 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 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 mean and standard deviations, such as for presence/absence and logarithmic values, 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 in the laboratory’s quality management plan (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 quality control requirements specified by the method, regulation, or contract, or as established this Standard and the laboratory’s quality management plan (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 (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 samples 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 with activity consistent 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 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 quality control requirements specified by the method, regulation, or
contract, or as established this Standard and the laboratory’s quality management plan (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

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.

At a minimum, the instrument quality control 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
requirements the laboratory shall incorporate guidelines established in MARLAP 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.

5One approach that addresses in detail all elements of this section is presented by ASTM International Standard
Practice D7282, Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements.
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 when subsequent performance checks indicate a change in performance;
   iii) after modification of system parameters that affect instrument response;
   iv) when instrument performance checks exceed predetermined acceptance criteria (i.e., limit of a statistical or tolerance control chart or other QC parameters) indicating a change in instrument response since the initial calibration;
   v) when indicated by corrective actions;
   vi) 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. Multiple-point calibration curves correlating other parameters (e.g., mass-efficiency, or channel-energy) may be required for some methods. Several, for examples include:

   i) energy-efficiency calibration of gamma spectrometers;
   ii) mass-efficiency (mass-attenuation) calibration of gas-flow proportional or x-ray detectors;
   iii) quench-efficiency calibration of liquid scintillation detectors;
   iv) mass-crosstalk calibration of gas-flow proportional and 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.); and
   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.c and 1.5.4.d) and included it in the uncertainty reported with each associated sample result.

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

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

1. the type of calibrations to be performed;
2. the number of calibration points required;
3. a description of the calibration standards required;
4. the preparation of the calibration standards;
5. the counting of the calibration standards;
6. 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
7. all calculations.

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

iii) If the initial instrument calibration results are outside established acceptance criteria, the laboratory shall perform corrective actions. The laboratory shall re-analyze any samples processed using this calibration, or, if not possible, report the results with qualifiers.

iv) 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. The calibration verification may take two forms:

i) performing a second set 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., the relative combined uncertainty or 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.
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. If a performance check result exceeds control 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.
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:
616  
617 i) Gamma-ray spectrometry systems.
618 Detection efficiency, energy calibration, and peak resolution:
619  1. Semiconductor detectors: At least twice weekly, but not on consecutive days, for a
620  continuously operating detector; day of use for a non-continuously operating detector.
621  2. Scintillation detectors (e.g., sodium iodide): Day of use.
622 ii) Alpha-particle spectrometry systems.
624 Detection efficiency: Monthly.
625 iii) Gas-proportional and semiconductor alpha/beta detectors.
626 Alpha and beta efficiency: Day of use.
627 iv) Liquid scintillation detectors.
628  1. Manufacturer system calibration: At the frequency recommended by the manufacturer.
629  2. Efficiency with unquenched $^3$H and $^{14}$C standards: Day of use.
630 v) Solid-state scintillation detectors (e.g., zinc sulfide) used for non-spectrometric
631 measurements.
632 Efficiency: Day of use.
633
634 c) Exceptions to minimum frequencies for performance checks:
635
636 i) An individual test source may be uninterruptedly measured for a time longer than the
637 required interval between performance checks to allow completion of the count of a test
638 source as long as instrument performance checks performed at the beginning and end of
639 the measurement period meet all applicable acceptance criteria.
640 ii) Test sources may be uninterruptedly measured for a time longer than the required interval
641 between performance checks to allow for completion of a preparation or analytical-radiation
642 measurements batch measured on an instrument with an automated sample changer (e.g.,
643 a liquid scintillation or gas proportional counter), as long as the period between the checks
644 does not exceed seven (7) days, and checks are done at the beginning and end of the
645 measurement in question and meet all applicable acceptance criteria.
646
d) If the detection system is powered off between performance checks, a new performance check
647 shall be performed prior to the next test source measurement.
648
650 1.7.1.5 Subtraction Background Measurements
651
652 Subtraction background measurements are performed to assess and correct for contributions due
653 to cosmic radiation, naturally-occurring radioactivity, electronic noise, impurities in the detector,
654 shielding, and source mounting material, or other sources that are not affected by the analytical
655 processes. Contributions from impurities in the reagents, reference standards, or other sources
656 introduced during the analytical processes are assessed with the use of method blanks (Section
657 1.7.2.2).
658
659 Numerous counting configurations may be used to determine subtraction background, depending
660 on the detector and the method, including: Counting an empty detector; counting an empty
661 container or blank test source in a detector; or counting a container filled with a surrogate matrix
662 material free of measurable levels of radioactivity.
663
664 a) The subtraction background shall be specific to each detector and the method.
665
666 b) The subtraction background counting time shall be at least as long as the longest associated
667 sample counting time and shall ensure a representative determination of the background rate.
668
669 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:

4. Liquid scintillation detectors.
   - Individual quenched background: Once per preparation batch.
   - Quenched background curve: According to frequency specified in laboratory procedures.
5. Solid-state scintillation detectors (e.g., zinc sulfide) used for non-spectrometric measurements: Day of use.

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

iv) 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 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 quality control measures used to verify the integrity of subtraction background measurements, check for possible detector contamination, electronics noise, as well as 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 predetermined frequency. The frequency for the checks should be based on an evaluation of the laboratory instrument system and an acceptable rate for lost data should short-term background check result fails. The frequency for 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 or analytical batch 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) 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 1.7.2.3). 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 quality control program that monitors and assesses the performance of the laboratory’s analytical systems. At a minimum, the quality control 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 standard is more stringent, the laboratory shall follow the requirements of the regulation or the mandated method. Where there are no established requirements, the laboratory shall incorporate guidelines established in MARLAP or other consensus standard organizations into its quality management system.

b) The laboratory shall process batch and sample-specific quality controls 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) Where sample preparation is performed that involves physical or chemical processing which affects the outcome of the test, the laboratory shall initiate a preparation batch.

d) Where sample testing is performed that does not involve physical or chemical processing which affects the outcome of the test (e.g., non-destructive gamma spectrometry or alpha/beta counting of air filters or swipes on gas proportional detectors), an analytical batch may be initiated in lieu of the preparation batch. The analytical batch, when initiated, shall have the following requirements:

i) Up to twenty (20) environmental samples may be combined into a single analytical batch. All samples and QC samples in the analytical batch shall have characteristics and analytical configurations similar to those used for calibration of the method (e.g., analytes, geometry, calibration, and background corrections).

ii) Samples may be added to the analytical batch until twenty (20) environmental samples have been counted or until the time period for the analytical batch is reached, whichever occurs first. The maximum time for processing an analytical batch (analytical batch period) shall not extend beyond fourteen (14) days from the start of the first sample count.

[iii]) The laboratory shall employ either a sample preparation batch or a radiation measurement batch (RMB, Section 1.3.1) to determine the grouping of samples and assignment of batch QC.

ij) 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) 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.

**j)** The laboratory’s quality control program shall document the minimum required frequency required for quality controls. Minimum quality control requirements are specified below.

**e)** The laboratory shall process all batch quality control 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 quality controls indicate that the systems are in control.

**k)** The laboratory shall not systematically or preferentially use specific detectors, equipment or glassware for the analysis of quality control 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 quality control samples and samples.

**h)** The laboratory shall assess the results of the quality controls against acceptance criteria documented in the quality control program. Where there are no established criteria in regulations, the method, or contract, the laboratory shall develop its acceptance criteria based on guidelines established in MARLAP, other consensus standards or other criteria such as statistical control charts developed by the laboratory.

**m)** The laboratory shall track and trend the results of batch quality control samples using statistical or tolerance control charts.

**n)** The laboratory’s quality control program shall document acceptance criteria for batch quality control samples, sample-specific quality controls, and for the evaluation of long-term trends and the methods used to establish these criteria.

**o)** 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 quality control 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 quality control sample and any associated actions in the laboratory report.

### 1.7.2.2 Negative Control – Method Performance: Method Blank

The method blank 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 method blank assesses sample handling and the analytical process.

**a)** The laboratory shall analyze a method blank at a minimum of one (1) per preparation or analytical-radiation measurements batch.
b) The method blank 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 method blank using materials that conform to the range of physical or chemical parameters applicable to the associated test sources of the same quality system matrix as samples in the batch. The material used for the method blank shall 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 shall use a surrogate matrix to simulate the quality system matrix.

ii) The size of the aliquot used for calculation of the method blank result shall be similar to that of routine samples for analyses. If the size of samples 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, 18.4.1).

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

i) The method blank exceeds the pre-established upper or lower bounds for the measurement, where the upper and lower bounds are plus x times the CSU-standard uncertainty and negative y times the CSU-standard uncertainty and x and y are the coverage factors for the established confidence interval as established by the laboratory’s quality assurance program. The upper and lower bounds are not necessarily symmetrical.

ii) When applicable, the sample-specific MDA for the method blank is greater than the required MDA.

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

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

f) The laboratory shall not subtract the batch method blank from sample results in the associated preparation or radiation measurements analytical batch. The laboratory may subtract the average historical activity of method blank 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 or analytical radiation measurements batch. For radiation measurements analytical batches, 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 laboratory shall prepare the positive controls LCS using materials that conform to the range of physical and chemical parameters applicable to the associated test sources of the same quality system matrix as samples in the batch.

ii) 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 surrogate 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.

iii) The size of the aliquot used for calculation of the LCS result shall be similar to that of routine samples for analyses. If the size of samples 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, 18.4.1).

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. The laboratory may use a calibration source for the LCS if the source is independent of the source used for calibration of the measurement system (see 1.7.2.2.e) below).

d) The laboratory shall spike the LCS at a level such that the uncertainty of the analytical result is less than one-third 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 that of routine samples if the activities are expected to exceed ten (10) times the Decision Level (Critical Value).

e) When available, the standard used to prepare the LCS shall be from a source independent of the laboratory standard used for instrument calibration and shall meet the requirements for reference standards provided in Section 1.7.56.2.c). If an independent source is not available, a second source shall be procured and prepared independently of the calibration source. The final prepared LCS need not be traceable to a national standard organization.

f) 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 indicated activity level (see Section 1.7.2.2.d above).

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

- the LCS shall contain gamma-emitting radionuclides that, at a minimum, represent the low (e.g., $^{241}\text{Am}$) and high (e.g., $^{60}\text{Co}$) energy range of the analyzed gamma-ray spectra. Commonly a medium energy radionuclide is also included in the LCS (e.g., $^{137}\text{Cs}$). 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.

g) 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 established acceptance criteria documented in the laboratory quality control program.

h) Where more than one 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

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 quality control (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. The laboratory shall have procedures in place for tracking, managing, and handling sample-specific QC criteria including spiking components 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.

#### a) Matrix Spike

- **i)** Matrix Spikes ($\text{Spikes}$) 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. Matrix Spikes 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 Matrix Spikes is specified by the method, a regulation or determined as part of the contract review process.

- **iii)** The components 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.e and 1.7.2.3.f.

- **iv)** The size and aliquot used for a Matrix Spike 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)** The lack of sufficient sample aliquot to perform a Matrix Spike shall be noted in the laboratory report.

- **vi)** The activity of the Matrix Spike analyte(s) shall be greater than five (5) times the MDA.
vii) Acceptance criteria for matrix spike recoveries shall be as documented in the method, regulation or in contract. Where there are no established criteria in the method, a regulation or contract, the laboratory shall develop its criteria for matrix spike recoveries based on industry practices and guidelines such as MARLAP.

viii) When available, the standard used to prepare the matrix spike shall be from a source independent of the laboratory standard used for instrument calibration and shall meet the requirements for reference standard provided in Section 1.7.2.6.c1.7.5.2.c (2). If an independent standard is not available, a second source shall be procured and prepared independently of the calibration source. The final prepared matrix spike need not be traceable to a national standards organization.

ix) The matrix spike 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.).

b) 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) Matrix Duplicate (MD) criteria are as specified by the method, regulation or determined as part of the contract review process. Where there are no established criteria in the method, a regulation or contract, the laboratory shall develop its criteria for duplicate acceptance based on guidelines established in the MARLAP or other criteria such control charting developed by the laboratory. This shall be documented in the method SOPs written procedures.

iii) At a minimum, the laboratory shall analyze one MD per preparation or analytical radiation measurements batch. For analytical batches (RMBs), the MD shall consist of a second measurement of one sample. If the batch is counted on more than one detector, the MD shall be performed on a second detector.

iv) When samples have low-levels of activity (less than approximately three times the MDA) the laboratory, at its discretion, may analyze matrix spike/matrix spike duplicate to determine reproducibility within a preparation batch in place of a MD.

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.

c) 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 quality control 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
iii) The chemical yield (tracer or carrier) 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 specific 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 or other criteria such control charting developed by the laboratory. This assessment shall meet established project or program measurement quality objectives (MQO).

v) When the specified 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 levels capability (e.g., MDA or Critical Level, or as appropriate) 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. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular method. Such information shall be available.

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

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

i) Reference standards 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 an ISO/IEC Guide 34 or ANSI N42.22 accredited reference material provider. Reference standards that are used in a radiochemical laboratory shall be obtained from NIST or from suppliers of NIST standards or NIST traceable radionuclides. Alternatively, reference standards may be obtained from suppliers outside the United States, provided that the standards are traceable back to each country’s national standards laboratory.

ii) Reference standards shall be accompanied with a certificate of calibration that meets the requirements of either ISO Guide 31, or ANSI N42.22 - 1995, Section 8, Certificates and shall includes 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, calibration or activity reference date and time (date or time if as appropriate for to the half-life of the radionuclide), physical and/or chemical description of the source, and radionuclide impurities (reference ANSI N42.22 - 1995, Section 8, Certificates).
iii) Standards prepared or derived from externally-obtained reference materials shall be verified against an independent standard obtained from a second manufacturer prior to initial use. 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.

iv) The laboratory shall account for radioactive decay/ingrowth whenever decay/ingrowth has occurred between the reference date and use that could impact use of the results.

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

vi) 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

a) The laboratory shall assure that the 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 Cleaning. 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 management system and records. Note that some applications may require single-use glassware.

c) Radiological Control Program. The laboratory shall maintain a radiological control program that addresses analytical radiological control. The program shall address the procedures for segregating samples with potentially widely varying levels of radioactivity. The radiological control program shall explicitly define how low-level and high-level samples will be identified, segregated and processed in order to prevent 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
a) Method blank results shall be evaluated for long term trends, absolute bias, possible contamination or interferences that may affect results for samples in the batch.

b) Method blank acceptance criteria are discussed in Section 1.7.2.1 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 method blank, 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 method blank 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 are evaluated to assess the performance of the entire analytical system independent of the sample matrix. LCS results are calculated in percent recovery (%R) 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 1.7.2.2 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) Matrix spikes and matrix duplicates allow evaluation of the effect of matrix on the accuracy and precision of results. Results from matrix spikes are calculated as percent recovery (%R), matrix replicates-duplicates, and matrix spike duplicate precision are calculated as relative percent difference (RPD), $Z_{RP}$ (see MARLAP, 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 or 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 are 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

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

b) 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.

c) Sample-specific estimates of uncertainty and minimum detectable activity (MDA) shall be evaluated to ensure that MQOs have been met.

d) 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 1.6.5.4. above.

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 over the requirements of this Standard.

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 management plan (if there are no established mandatory criteria).

1.7.4.2 The laboratory shall document the required timing, methods for performing measurements, 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 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.