



## **ENVIRONMENTAL LABORATORY SECTOR**

### **VOLUME 1**

# **MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS**

## **Module 6: Quality Systems for Radiochemical Testing**

### **TNI Standard**

**Adopted by the NELAP Board, September 8, 2009  
Effective for use in NELAP after September 1, 2010**

**P.O. Box 2439  
Weatherford, TX 76086  
817-598-1624  
[www.nelac-institute.org](http://www.nelac-institute.org)**

## **PREFACE**

---

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

Section 1.7.1 c) of this document has been processed in accordance with the TNI requirement for a Tentative Interim Amendment. The same or similar amendment will undergo the consensus standards development process within the time-frame specified in SOP 2-100.

---

**VOLUME 1, MODULE 6****Quality Systems for Radiochemical Testing**

---

**Table of Contents**

---

<b>1.0</b>	<b>RADIOCHEMICAL TESTING .....</b>	<b>1</b>
1.1	Introduction .....	1
1.2	Scope .....	1
1.3	Terms and Definitions .....	1
1.3.1	Additional Terms and Definitions .....	1
1.3.2	Exclusions and Exceptions .....	1
1.4	Method Selection .....	1
1.5	Method Validation .....	2
1.5.1	Validation of Methods .....	2
1.5.2	Detectable Activity .....	2
1.5.3	Evaluation of Precision and Bias .....	3
1.5.4	Measurement Uncertainty .....	3
1.5.5	Evaluation of Selectivity .....	3
1.6	Demonstration of Capability (DOC) .....	4
1.6.1	General .....	4
1.6.2	Initial DOC .....	4
1.6.3	Ongoing DOC .....	5
1.7	Technical Requirements .....	6
1.7.1	Instrument Calibration .....	6
1.7.2	Quality Control for Radiochemistry .....	8
1.7.3	Data Acceptance / Rejection Criteria .....	13
1.7.4	Sample Handling .....	14

---

## VOLUME 1, MODULE 6

### Quality Systems for Radiochemical Testing

---

#### 1.0 RADIOCHEMICAL TESTING

##### 1.1 Introduction

This Standard contains detailed 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

These requirements apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. Procedures for radiochemical analysis may involve some form of chemical separation followed by detection of the radioactive emissions of the analyte (or indicative daughters) and tracer isotopes where used. Procedures for the determination of radioactive isotopes by mass spectrometry (e.g., ICP-MS or TIMS) or optical (e.g., KPA) techniques are outside the scope of this document.

The essential quality control procedures applicable to radiochemistry measurements are included in this Standard. Additional quality control requirements that are specified by method, regulation or project shall be 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

Reserved

###### 1.3.2 Exclusions and Exceptions

Reserved

##### 1.4 Method Selection

A reference method is a method issued by an organization generally recognized as competent to do so. (When ISO refers to a standard method, that term is equivalent to reference method). When a laboratory is required to analyze a parameter by a specific method due to a regulatory requirement, the parameter/method combination is recognized as a reference method. If there is not a regulatory requirement for the parameter/method combination, the parameter/method combination is recognized as a reference method if it can be analyzed by another similar reference method of the same matrix and technology, and the inclusion of the parameter in the method meets all required calibration requirements of the method and the quality control requirements of the method to which the parameter is being added. If no QC exists in the method, the laboratory shall adhere to the requirements outlined in the similar method.

When it is necessary to use methods not covered by reference methods, these shall be subject to agreement with the client and shall include a clear specification of the client's requirements and the purpose of the environmental test. The method developed shall have been validated appropriately before use.

## **1.5 Method Validation**

### **1.5.1 Validation of Methods**

- a) Validation is the confirmation by examination and the objective evidence that the particular requirements for a specific intended use are fulfilled.
- b) For reference methods, the minimum detectable activity (Section 1.5.2.1) applies. Evaluating precision and bias is covered in Section 1.5.3.
- c) The laboratory shall validate non-reference methods, laboratory-designed/developed methods, reference methods used outside their published scope, and amplifications and modifications of reference methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use. The minimum requirements for method validation are given in Sections 1.5.2 – 1.5.5.

### **1.5.2 Detectable Activity**

All procedures used shall be documented. Documentation shall include the quality system matrix type. All supporting data shall be retained.

#### **1.5.2.1 Minimum Detectable Activity (MDA)**

The laboratory shall utilize a method that provides an MDA that is appropriate and relevant for the intended use of the data. MDAs shall be determined by the protocol in the mandated method. If the protocol for determining the MDA is not specified, the selection of the procedure shall reflect instrument limitations and the intended application of the method.

- a) The laboratory shall determine the MDA for the method for each target analyte of concern in the quality system sample matrices. All sample-processing steps of the analytical method shall be included in the determination of the MDA.
- b) The MDA shall be initially determined for the analytes of interest in each method in a quality system matrix in which there are no target analytes and no interferences at levels that would impact the results.
- c) The MDA shall be determined each time there is a change in the method that affects how the test is performed, or when a change in instrumentation occurs that affects the analytical detection capability.
- d) The MDA is an estimate of the smallest true activity (or activity concentration) of analyte in a sample that ensures a 95% probability of detection, given a detection criterion that ensures only a 5% probability of detection in analyte-free samples.

#### **1.5.2.2 Required Detection Limit for Drinking Water Compliance**

Laboratories that analyze drinking-water samples for Safe Drinking Water Act (SDWA) compliance monitoring shall use methods whose detection limits meet the requirements of 40 CFR 141. The

SDWA detection limit is defined in 40 CFR 141.25(c) as equal to the analyte 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). The SDWA detection limit is equivalent to the concentration at which the relative standard deviation of the measurement due to counting statistics is 1/1.96.

### 1.5.3 Evaluation of Precision and Bias

- a) Reference Methods. The laboratory shall evaluate the precision and bias of a reference method for each analyte of concern for each quality system matrix according to Section 1.6 or alternate documented procedure when the analyte cannot be spiked into the sample matrix and QC samples are not commercially available.
- b) Non-Reference Methods. For laboratory-developed methods or non-reference methods that were not in use by the laboratory before July 2003, the laboratory shall have a documented procedure to evaluate precision and bias. The laboratory shall also compare results of the precision and bias measurements with criteria established by the client, given in the reference method, or established by the laboratory.
- c) The laboratory shall also evaluate precision and bias in the relevant quality system matrices and shall process the samples through the entire measurement system for each analyte of interest.
- d) An example of a systematic approach to evaluate precision and bias could be the following:

Analyze QC samples in triplicate containing the analytes of concern at or near the MDA, at a level near ten (10) times the MDA, and at a mid-range concentration. Process these samples on different days as three (3) sets of samples through the entire measurement system for each analyte of interest. Each day one QC sample at each concentration is analyzed. A separate method blank shall be subjected to the analytical method along with the QC samples on each of the three (3) days. For each analyte, calculate the mean recovery for each day, for each level over days, and for all nine (9) samples. Calculate the relative standard deviation for each of the separate means obtained.

### 1.5.4 Measurement Uncertainty

All radiochemical measurements shall provide the uncertainty of each quantitative measurement result. 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 precision at each 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.

The combined standard uncertainty, when used, is the uncertainty of a measured value expressed as an estimated standard deviation. It is calculated by combining the standard uncertainties of the input estimates.

### 1.5.5 Evaluation of Selectivity

The laboratory shall evaluate selectivity, if applicable, by following the checks established within the method.

## 1.6 Demonstration of Capability (DOC)

### 1.6.1 General

Prior to acceptance and institution of any method for data reporting, satisfactory initial DOC is required (see Section 1.6.2).

Thereafter, ongoing DOC (Section 1.6.3), as per the quality control requirements in Section 1.7.3 (such as laboratory control samples) is required.

In cases where a laboratory analyzes 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, personnel 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.

For the initial DOC, appropriate records as discussed in Section 1.6.2 shall be completed.

An initial DOC shall be completed each time there is a change in instrument type, personnel, or method.

All demonstrations shall be documented. All data applicable to the demonstration 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.c).

#### 1.6.2.2 If the method or regulation 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 concentrations that will impact the results of a specific method) sufficient to prepare four (4) aliquots at a laboratory specified concentration. 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.,  $^{241}\text{Am}$ ), medium (e.g.,  $^{137}\text{Cs}$ ) and high (e.g.,  $^{60}\text{Co}$ ) 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.

- b) At least four (4) aliquots shall be prepared and analyzed according to the method either concurrently or over a period of days.
- c) Using all of the results, calculate the mean recovery 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.
- d) Compare the information from (c) above to the corresponding acceptance criteria for precision and accuracy in the method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters does not meet the acceptance criteria, the performance is unacceptable for that parameter.
- e) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst shall proceed according to i) or ii) below.
  - i) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with b) above.
  - ii) Beginning with b) above, repeat the test for all parameters that failed to meet criteria.
- f) Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with b).
- 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 and quantified this is not required.

### 1.6.3 Ongoing DOC

1.6.3.1 The laboratory shall have a documented procedure describing ongoing DOC. The analyst(s) shall demonstrate ongoing capability by meeting the quality control requirements of the method, laboratory SOP, client specifications, and/or this Standard. 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 a blind sample (single blind to the analyst);

Note: Successful analysis of a blind performance sample on a similar method using the same technology.

- b) another initial DOC;
- c) at least four (4) consecutive laboratory control samples with acceptable levels of precision and accuracy. The laboratory shall determine the acceptable limits for precision and accuracy

prior to analysis. The laboratory shall tabulate or be able to readily retrieve four (4) consecutive passing LCS for each method for each analyst each year;

- d) a documented process of analyst review using QC samples. QC samples can be reviewed to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary;
- 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 Calibration

#### a) Initial Calibration

This Section addresses those practices that are necessary for proper calibration of radiation counting instruments for environmental testing involving radioanalytical measurements.

This Section specifies the essential elements that shall define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data shall be of known quality and be appropriate for a given regulation or decision. This Standard does not specify detailed procedural steps (“how to”) for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated method or regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which standard is more stringent, then the requirements of the mandated method or regulation are to be followed.

Given that radiation detection efficiency is essentially independent of sample activity at all but high activity levels (where dead time becomes significant), the requirements for calibration ranges of standards, of data reporting in calibration range, and the number of calibration standards are not applicable to radiochemical method calibrations except for mass attenuation in gas-proportional counting and sample quench in liquid scintillation counting. Nuclear counting instruments are subject to calibration prior to initial use, when the instrument is placed back into service after major repairs and the instrument’s response has changed as determined by a performance check, when the instrument’s response exceeds predetermined acceptance criteria for the instrument quality control. Instruments may also be recalibrated on a regular frequency even in the absence of these conditions.

The frequency of calibration shall be described in the laboratory method SOP if not specified in the method. A specific frequency (e.g., annually) or calibrations based on observations from the associated control or tolerance chart, shall be specified in the laboratory method SOP.

Instrument calibration shall be performed with reference standards as defined in Section 1.7.2.5.c). The standards shall have the same general characteristics (i.e., geometry, homogeneity, density, etc.) as the associated samples.

The following items are essential elements of initial instrument calibration:

- i) The details of the initial instrument calibration procedures including calculations, acceptance criteria and associated statistics shall be included or referenced in the

method SOP. When initial instrument calibration procedures are referenced in the method, then the referenced material shall be retained by the laboratory and be available for review.

- ii) Sufficient raw data records shall be retained to permit reconstruction of the initial instrument calibration (e.g., calibration date, method, instrument, analysis date, each analyte name, analyst's initials or signature; activity and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to activity or concentration).
  - iii) Sample results shall be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method, or program.
  - iv) All initial instrument calibrations shall be verified with a standard obtained from a second manufacturer or lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Traceability shall be to a national standard, when commercially available.
  - v) Criteria for the acceptance of an initial instrument calibration shall be established (e.g., correlation coefficient or relative percent difference). The criteria used shall be appropriate to the calibration technique employed.
  - vi) If the initial instrument calibration results are outside established acceptance criteria, corrective actions shall be performed and all associated samples re-analyzed. If re-analysis of the samples is not possible, data associated with an unacceptable initial instrument calibration shall be reported with appropriate data qualifiers.
  - vii) If a reference or mandated method does not specify the number of calibration standards, the laboratory shall have a written procedure for determining the number of points for establishing the initial instrument calibration.
- b) Instrument Calibration Verification (Performance Checks)

Performance checks shall be performed using appropriate check sources and monitored with control charts or tolerance charts to ensure that the instrument is operating properly, the detector response has not significantly changed, and therefore the instrument calibration has not changed. The same check source used in the preparation of the tolerance chart or control chart at the time of calibration shall be used in the calibration verification of the instrument (performance checks). The check sources shall provide adequate counting statistics for a relatively short count time and the source should be sealed or encapsulated to prevent loss of activity and contamination of the instrument and laboratory personnel.

- i) For gamma-ray spectroscopy systems, performance checks for detection efficiency, energy calibration, and peak resolution shall be performed on a day-of-use basis.
- ii) For alpha-particle spectroscopy systems, the performance check for energy calibration shall be performed on a weekly basis and the performance check for detection efficiency shall be performed on at least a monthly basis.
- iii) For gas-proportional and liquid scintillation counters, the performance check for detection efficiency shall be performed on a day-of-use basis. For batches of samples that uninterruptedly count for more than a day, a performance check may be performed instead at the beginning and end of the batch as long as this time interval is no greater than one week.

- iv) For scintillation counters the calibration verification for detection efficiency shall be performed on a day-of-use basis.

- c) Background Measurement

Background measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required measurement quality objectives. (This background measurement is not the short term check for contamination that is addressed in 1.7.1 d). These values must be subtracted from the total measured activity in the determination of the sample activity.

- i) For gamma-ray spectroscopy systems, background measurements shall be performed on at least a monthly basis.
- ii) For alpha-particle spectroscopy systems, background measurements shall be performed on at least a monthly basis.
- iii) For gas-proportional counters background measurements shall be performed on at least a weekly basis.
- iv) For scintillation counters, background measurements shall be performed each day of use.

- d) Instrument Contamination Monitoring

The laboratory shall have a written procedure for monitoring radiation measurement instrumentation for radioactive contamination. The procedure shall indicate the frequency of the monitoring and shall indicate criteria, which initiates corrective action.

## 1.7.2 Quality Control for Radiochemistry

The laboratory shall have quality control procedures for monitoring the validity of environmental tests undertaken as specified in this Section. This monitoring shall be planned and reviewed.

The failure of any QC sample analysis and the corrective actions taken shall be noted in the laboratory report for the associated samples.

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

- a) The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps or for other low-level bias. The method blank shall be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure. Procedures shall be in place to determine if a method blank result is significantly different from zero. Any affected samples associated with a failed method blank shall be reprocessed for analysis or the results reported with appropriate data-qualifying codes.
- b) The method blank shall be analyzed at a minimum of one (1) per preparation batch, which shall be a maximum of twenty (20) field samples, for all radiochemical methods except gross alpha/beta in solid matrices and gamma-ray spectrometry.
- c) The method blank shall consist of a quality system matrix that is similar to the associated samples and is known to be as free of the analytes of interest as possible.

There shall be no subtraction of the method blank result from the sample results in the associated preparation or analytical batch unless permitted by method or program. This requirement does not preclude corrections for background radiation (e.g., instrument background, analyte in the tracer or carrier, reagent impurities, peak overlap, etc.) to all analyzed samples, both program/project submitted and internal quality control samples. However, these corrections shall not depend on the result of the method blank analysis, whose purpose is to check for uncorrected contamination or other low-level bias.

The method blank sample shall be prepared with aliquot size similar to that of the routine samples for analysis.

#### 1.7.2.2 Positive Control – Method Performance: Laboratory Control Sample (LCS)

- a) The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria may indicate that the analytical system is “out of control.” Any affected samples associated with an out-of-control LCS shall be reprocessed for reanalysis or the results reported with appropriate data qualifying codes.
- b) The LCS shall be analyzed at a minimum of one per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available.
- c) The LCS is a quality system matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes.

NOTE: The matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS.

- d) Alternatively the LCS may consist of a medium containing known and verified concentrations of analytes or as Certified Reference Material (CRM). The components to be spiked shall be as specified by the mandated method or regulation or as requested by the client.
- e) The activity of the laboratory control sample shall be: (1) at least ten (10) times the MDA, and (2) at a level comparable to that of routine samples when such information is available if the sample activities are expected to exceed ten times the MDA.
- f) The laboratory standards used to prepare the laboratory control sample shall be from a source independent of the laboratory standards used for instrument calibration and shall meet the requirements for reference standards provided in Section 1.7.5.2.c).
- g) Where a radiochemical method, other than gamma-ray spectroscopy, has more than one reportable analyte isotope (e.g. plutonium,  $^{238}\text{Pu}$  and  $^{239}\text{Pu}$ , using alpha-particle spectrometry), only one of the analyte isotopes need be included in the laboratory control sample at the indicated activity level. However, where more than one analyte is detectable, each shall be assessed against the specified acceptance criteria.
- h) 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.,  $^{241}\text{Am}$ ), medium (e.g.,  $^{137}\text{Cs}$ ) and high (e.g.,  $^{60}\text{Co}$ ) 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.
- i) The laboratory control sample shall be prepared with similar aliquot size to that of the routine samples for analyses.

### 1.7.2.3 Sample-Specific Controls

The laboratory shall document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of quality system matrix specific quality control (QC) samples and are designed as data quality indicators for a specific sample using the designated method.

Examples of matrix-specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD); and replicates. The laboratory shall have procedures in place for tracking, managing, and handling matrix-specific QC criteria including spiking appropriate components at appropriate concentrations, calculating recoveries and relative percent difference, evaluating and reporting results based on performance of the QC samples.

#### a) Matrix Spike

- i) Matrix spikes indicate the effect of the sample matrix on the accuracy of the results generated using the selected method. The results of this analysis shall be one of the quality control measures used to assess the batch.
- ii) The frequency of the analysis of matrix spikes are as specified by the method or may be determined as part of the contract review process.
- iii) The components to be spiked shall be as specified by the mandated method. Any permit specified analytes, as specified by regulation or client requested analytes shall also be included.
- iv) The lack of sufficient sample aliquot size to perform a matrix spike shall be noted in the laboratory report.
- v) The activity of the matrix spike analytes(s) shall be greater than five times the MDA.
- vi) The laboratory standards used to prepare the matrix spike shall be from a source independent of the laboratory standards used for instrument calibration and shall meet the requirements for reference standards of Section 1.7.2.5.c).
- vii) The matrix spike shall be prepared by adding a known activity of target analyte after sub-sampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.). Where a radiochemical method, other than gamma-ray spectroscopy, has more than one reportable analyte isotope (e.g. plutonium,  $^{238}\text{Pu}$  and  $^{239}\text{Pu}$ , using alpha-particle spectrometry), only one of the analyte isotopes need be included in the matrix spike sample at the indicated activity level. However, where more than one analyte is detectable, each shall be assessed against the specified acceptance criteria.

#### b) Replicates / Matrix Spike Duplicates / Laboratory Control Sample Duplicates

- i. Replicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. Replicates provide the most useful measure of precision when target analytes are found in the sample chosen for replication.
- ii. The frequency of the analysis of matrix replicates and duplicates are as specified by the method or may be determined as part of the contract review process.
- iii. Replicates are performed on replicate aliquots of actual samples.

- iv. For low-level samples (less than approximately three times the MDA) the laboratory may analyze a laboratory control samples duplicate or a replicate matrix spike (matrix spike and a matrix spike duplicate) to determine reproducibility within a preparation batch in place of a sample replicate. In addition based on project or program requirements, the laboratory may analyze a laboratory control sample duplicate or a matrix spike duplicate in place of a sample replicate.

c) Tracer

For those methods that employ a tracer for yield determination, each sample result shall have an associated tracer yield calculated and reported. The tracer shall be added to the sample after subsampling, if required, but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The tracer yield for each sample result shall be one of the quality control measures to be used to assess the associated sample result acceptance. The tracer yield shall be assessed against the specific acceptance criteria specified in the laboratory method SOP. When the specified tracer yield acceptance criteria are not met, the specified corrective action and contingencies shall be followed. The occurrence of a failed tracer yield and the actions taken shall be noted in the laboratory report to the client.

d) Carrier

For those methods that utilize a carrier for yield determination, each sample shall have an associated carrier yield calculated and reported. The carrier shall be added to the sample after subsampling, if required, but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The carrier yield for each sample shall be one of the quality control measures to be used to assess the associated sample result acceptance. The carrier yield shall be assessed against the specific acceptance criteria specified in the laboratory method SOP. When the specified carrier yield acceptance criteria are not met, the specified corrective action and contingencies shall be followed. The occurrence of a failed carrier yield and the actions taken shall be noted in the laboratory report to the client.

#### 1.7.2.4 Data Reduction

- a) The procedures for data reduction, such as use of linear regression, shall be documented.
- b) Measurement Uncertainties. Each result shall be reported with its measurement uncertainty. The report should clearly explain the uncertainty. At a minimum the report shall:
  - i) indicate whether the uncertainty is the combined standard uncertainty (“one sigma”) or an expanded uncertainty; and
  - ii) for expanded uncertainties, indicate the coverage factor (k) and optionally the approximate level of confidence.
- c) The procedures for determining the measurement uncertainty shall be documented and shall be consistent with the ISO Guide 98: 1995, Guide to the Expression of Uncertainty in Measurement (GUM) and with the recommendations of Chapter 19 of the Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP) Volume I (EPA 402-B-04-001A), Volume II (EPA 402-B-04-001B), Volume III (EPA 402-B-04-001C), July 2004.

### 1.7.2.5 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 that are used in a radiochemical laboratory shall be obtained from NIST or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.
  - ii) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.
  - iii) Laboratories should consult with the supplier if the lab's verification of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory shall use only the decay-corrected certified value. The laboratory shall have a written procedure for handling, storing, and establishing expiration dates for reference standards.

### 1.7.2.6 Selectivity

The laboratory shall evaluate selectivity by following the checks established within the method.

### 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.
- b) Glassware Cleaning. Glassware 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 laboratory records and SOPs. 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 Acceptance/Rejection Criteria

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

- a) While the goal is to have no statistically significant difference from zero, each method blank shall be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. The source of contamination or other bias shall be investigated and measures taken to minimize or eliminate the problem and affected samples reprocessed or data shall be appropriately qualified if:
  - i) the absolute value of the activity of a targeted analyte in the blank exceeds three times its combined standard uncertainty, AND is greater than 1/10 of the activity measured in any sample; or
  - ii) the method blank result otherwise affects the sample results as per the method requirements or the project-specific measurement quality objectives.
- b) The acceptance criteria for samples associated with a failed method blank shall be calculated in a manner that compensates for sample results based on differing aliquot sizes.
- c) When a blank result is determined to be significantly different from zero, the cause shall be investigated and measures taken to minimize or eliminate the problem. Samples associated with a failed blank shall be evaluated as to the best corrective action for the samples (e.g., reprocessing or data qualifying codes).
- d) The occurrence of a failed method blank and any associated corrective action shall be noted in the laboratory report to the client.

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

- a) The results of the individual batch LCS are calculated in percent recovery or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation.
- b) The individual LCS is compared to the acceptance criteria as published in the mandated method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria.
- c) An LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with an LCS determined to be “out of control” shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.
- d) The occurrence of a failed LCS and any associated actions shall be noted in the laboratory report to the client.

#### 1.7.3.3 Sample-Specific Controls

- a) Matrix Spike; Matrix Spike Duplicates
  - i) The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), relative percent difference (RPD), or other appropriate

statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation for %R, RPD or other statistical treatment used.

- ii) The results are compared to the acceptance criteria as published in the mandated method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix spike results outside established criteria, corrective action shall be documented or the data reported with appropriate data qualifying codes.
  - iii) The occurrence of a failed matrix spike and any associated actions shall be noted in the laboratory report to the client.
- b) Replicates
- i) The results from replicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e.g., normalized differences).
  - ii) The laboratory shall document the calculation for relative percent difference or other statistical treatments.
  - iii) Results are compared to the acceptance criteria as published in the mandated method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For replicate results outside established criteria, corrective action shall be documented or the data reported with appropriate data qualifying codes.
  - iv) The occurrence of a failed replicate and any associated actions shall be noted in the laboratory report to the client.

#### 1.7.4 Sample Handling

- a) All samples that require thermal preservation shall be considered acceptable if the arrival temperature of a representative sample container is either within 2°C of the required temperature or the method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable.
  - i) Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 1.7.4.a. In these cases, the samples shall be considered acceptable if the samples were received on ice.
  - ii) If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.
- b) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or chlorine, prior to or during sample preparation or analysis.