ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

TNI Standard

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ENVIRONMENTAL LABORATORY SECTOR
Management and Technical Requirements for Laboratories Performing Environmental Analysis

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ENVIRONMENTAL LABORATORY SECTOR

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MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 1: Proficiency Testing

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PREFACE

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

This Standard supersedes and replaces preceding documents in whole or in part. It may be used by any organization that wishes to implement a program for the accreditation of environmental laboratories.

Standard Revision History

Action	Date
Working Draft Standard Published	January 6, 2012
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VOLUME 1, MODULE 1
Proficiency Testing

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VOLUME 1, MODULE 1
Proficiency Testing

1.0 Introduction, Scope, and Applicability

1.1 Introduction

Volume 1, Module 1 provides the requirements for laboratory participation in the TNI Proficiency Testing (PT) program.

1.2 Scope

The purpose of the TNI PT program is to provide a means for an Accreditation Body (AB) to evaluate a laboratory's performance, under specified conditions relative to a given set of criteria in a specific area of testing, through analysis of PT samples provided by an external source.

1.3 Applicability

1.3.1 Volume 1, Module 1 is applicable to any laboratory attempting to gain or maintain accreditation from a Primary AB that uses this Standard as the basis for accreditation regardless of the number of personnel working in the laboratory or the scope of testing performed by the laboratory.

1.3.2 This Standard applies only to fields of accreditation (FOA) that are also designated as fields of proficiency testing (FoPT) by the TNI Proficiency Testing Program Executive Committee (PTPEC).

2.0 Normative References

Not Applicable.

3.0 Terms and Definitions

For the purpose of this Standard, the relevant terms and definitions conform to *ISO/IEC 17011:2004* and *ISO/IEC 17025:2005*. Additional relevant terms are defined below.

3.1 Field of Accreditation (FOA): Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

3.2 Field of Proficiency Testing (**FoPT**)**:** Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges, and acceptance criteria have been established by the PTPEC.

3.3 Primary Accreditation Body (Primary AB): The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.

3.4 Proficiency Testing (PT): A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.

3.5 **Proficiency Testing Program (PT Program):** The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of results and the collective demographics and results summary of all participating laboratories.

3.6 **Proficiency Testing Provider (PT Provider):** A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT program.

3.7 **Proficiency Testing Provider Accreditor (PTPA):** An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.

3.8 **Proficiency Testing Reporting Limit (PTRL):** A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.

3.9 **Proficiency Testing Sample (PT Sample):** A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.

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3.10 **PT Study Closing Date:**

a) **Scheduled PT Study:** The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider.

b) **Supplemental PT Study:** The calendar date a laboratory submits the results for a PT sample to the PT Provider.

3.11 **PT Study Opening Date:**

a) **Scheduled PT Study:** The calendar date that a PT sample is first made available to all participants of the study by a PT provider.

b) **Supplemental PT Study:** The calendar date the PT Provider ships the sample to a laboratory.

3.12 **Revocation:** The total or partial withdrawal of a laboratory's accreditation by an accreditation body.

3.13 **Study (or PT Study):** This term refers to a Scheduled PT Study or a Supplemental PT Study.

a) **Scheduled PT Study:** A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants.

b) **Supplemental PT Study:** A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard, but that does not have a pre-determined opening date and closing date.

3.14 **Suspension:** The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed six (6) months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.

4.0 Requirements for Accreditation

4.1 General Requirements

4.1.1 TNI publishes lists of FoPTs on the TNI website for which PT studies are required, called TNI FoPT Tables. These FoPT tables may be updated, as needed, by publishing revised FoPT tables on the TNI website.

4.1.2 The laboratory shall participate in PT studies for each field of accreditation where corresponding FoPTs exist in the TNI FoPT tables and for which the laboratory seeks to obtain or maintain accreditation.

4.1.3 The laboratory shall obtain scheduled PT studies or supplemental studies for the individual fields of proficiency testing from a PT Provider accredited to Volume 3 of this Standard by a TNI-approved PTPA.

4.1.4 The laboratory shall analyze unique, single-blind, single-concentration PT samples, when required as stated in the TNI FoPT tables described in Section 4.1.1, to determine compliance for each field of accreditation for which the laboratory seeks to obtain or maintain accreditation.

NOTE: PT results are required by Federal Regulations, 40 CFR 141, per test method, rather than technology, for potable water PTs.

4.1.5 Prior to the closing date of a study, laboratory personnel, including corporate personnel, shall not:

a) send a PT study, or a portion of a PT study, in which it is participating, to another laboratory for the analysis of a field of accreditation for which it seeks accreditation or is accredited;

b) knowingly receive and analyze any PT sample or portion of a PT sample from another laboratory for which the results of the PT sample are intended for use for initial or continued accreditation of that laboratory;

c) communicate with any individual at another laboratory, including other laboratories under common ownership, concerning the analysis of the PT sample;

d) attempt to obtain the assigned value of any portion of the PT study from the PT Provider.

4.1.6 Participation in any of the above activities listed in 4.1.5 is cause for revocation of accreditation.

4.1.7 When a regulatory program has additional PT requirements for FoPTs not covered by this Standard, then the laboratory shall follow those requirements.

4.2 Sample Handling, Preparation, and Analysis Requirements

4.2.1 The laboratory shall handle and prepare the PT study samples in accordance with the instructions provided by the PT Provider.

4.2.2 PT samples shall be analyzed in accordance with the laboratory's routine standard operating procedures (SOPs) using the same quality control (QC), acceptance criteria and staff as used for the analysis of routine environmental samples.

4.2.3 The laboratory shall evaluate the analytical result for each chemistry and radiochemistry field of accreditation to the PTRL as established by the TNI FoPT Tables.

4.2.4 For chemistry analyses, if the laboratory's Limit of Quantitation (LOQ) is below the PTRL, they may evaluate results to their normal LOQ.

4.2.5 For chemistry PT results where the concentrations are below the calibration range established by the initial calibration curve, the following actions are acceptable:

a) the laboratory may re-scale its initial calibration curve to bracket the concentration of the PT sample result; or

b) the laboratory may report the results, as measured with the initial calibration curve, without qualification to the PT Provider, provided the laboratory adheres to the requirements of Section 4.3.7.

4.3 Reporting Requirements

4.3.1 The laboratory shall report PT study results to the PT Provider on or before the closing date of the study using the reporting format offered by the PT Provider.

4.3.2 The laboratory shall, on or before the closing date of the study, direct the PT Provider to report the PT study performance results directly to the AB(s) designated by the laboratory. For initial accreditation(s), the laboratory shall direct the PT Provider to provide all relevant PT study results to the AB to support their accreditation application.

4.3.3 The laboratory shall report results in such a way that there is a specific match between the analytical result for the FoPT and the corresponding Field of Accreditation for which the PT sample was analyzed.

4.3.4 Except for drinking water analytes referenced in 40 CFR 141, a laboratory may choose to analyze and report a single method to represent a technology in a single PT study for a particular analyte. If the laboratory analyzes and reports PT studies by "technology," the score obtained for the reported method will be applied to all methods in that technology for which the laboratory seeks to obtain or maintain accreditation in that matrix.

NOTE: If a laboratory reports PT results for multiple methods using the same analytical technology, an evaluation of "not acceptable" for one method will be applied to all methods reported with that technology.

4.3.5 The laboratory shall report chemistry PT study results to the PTRL as established by the TNI FoPT tables, or if the laboratory LOQ is below the PTRL, the laboratory may report results down to their normal LOQ, and as specified in Section 4.2.4.

4.3.6 Radiochemistry results shall be reported as measured, including zero, negative, and positive results, and shall not be censored or reported as "less than" values. All radiochemistry PT study results shall be reported in association with the measurement uncertainty, as appropriate to the program.

4.3.7 The laboratory shall evaluate and report each chemistry FoPT result to the PT Provider as follows:

a) If the analytical result is a numeric value above or equal to the PTRL, the laboratory shall report the value. If the PTRL is less than the laboratory's LOQ, the laboratory shall report the result without the qualification of result required in Volume 1, Module 4 of this Standard.

b) If the analytical result is a numeric value below the PTRL, the laboratory shall report one of the following:

i. <PTRL or,

ii. the obtained analytical result, if the result is between the LOQ and the PTRL, or,

iii. <LOQ, if the analytical result is below the LOQ and the PTRL.

c) If the analytical result is a "non-detect", the laboratory shall report one of the following:

i. <PTRL, or

ii. <LOQ**

NOTE: In the case where the laboratory LOQ is greater than the PTRL: If the laboratory chooses to report a value of <LOQ and the analyte is present above the PTRL, the result will be scored as "Not Acceptable" by the PT Provider.

4.3.8 The PTRL value shall not be adjusted for sample amount used or percent moisture.

4.4 Record Retention

4.4.1 The laboratory shall retain all records necessary to facilitate reconstruction of the preparation, processing, and reporting of analytical results for PT samples for a minimum of five (5) years. The laboratory shall make these records available for review upon request by the Primary AB.

5.0 PT Study Frequency Requirements for Accreditation

5.1 Initial Accreditation

5.1.1 Chemical Testing, Radiochemical Testing, Asbestos, and Microbiology

a) The laboratory shall achieve a history of two (2) successful (acceptable scores) PT studies out of the most recent three (3) attempts for each field of accreditation specified in Section 4.1.1 for which the laboratory seeks accreditation.

NOTE: If the laboratory has two (2) consecutive acceptable PT scores, a third study is not needed.

b) The two (2) PT studies identified in Section 5.1.1 a) must be performed no more than eighteen (18) months prior to obtaining initial accreditation from an AB.

c) The opening date of the second study must be at least seven (7) calendar days after the closing date of the first study.

d) The closing date of the most recent successful PT study for an FoPT must be no more than six (6) months prior to the application for initial accreditation, and the laboratory shall continue to participate in PT studies at least semi-annually (no more than seven (7) months apart between consecutive attempts) from that point on.

5.1.2 For Whole Effluent Toxicity (WET) testing, the laboratory shall demonstrate to the Primary AB that it has received an acceptable evaluation for at least one (1) PT study to obtain initial accreditation. The study closing date of the most recent successful PT study shall be no more than twelve (12) months prior to obtaining initial accreditation from an AB, and the laboratory shall continue to participate in PT studies annually from that point on.

NOTE: "Acceptable" PT study scores from a PT Provider do not automatically result in a successful evaluation of a PT study by an AB. For example, failure to report an analytical method or reporting of an incorrect method, failure to provide the PT Provider with a release of results to the AB before the close of the study, failure to report results to the PT Provider before the closing date, failure to handle PT study samples in the same manner as routine environmental samples, etc., may be cause for an unsuccessful evaluation by an AB.

5.2 Continued Accreditation

5.2.1 Chemical Testing, Radiochemical Testing, Asbestos, and Microbiology

5.2.1.1 The laboratory shall maintain a history of two (2) successful (acceptable scores) PT studies out of the most recent three (3) attempts for each field of accreditation specified in Section 4.1.1 for which the laboratory holds accreditation. Failure to do so may result in suspension of the affected field of accreditation. The laboratory's accreditation for a field of accreditation may be revoked for failure of three (3) consecutive PT studies, either by failure to participate in the required PT study or due to failure to obtain acceptable results.

5.2.1.2 The laboratory shall analyze and report a PT study at least twice per year for each accreditation FoPT for which it seeks to maintain accreditation, in accordance with the following criteria:

a) The closing dates of subsequent PT study samples for a particular accreditation FoPT shall be no more than seven (7) months apart.

b) The opening date of PT study samples for a particular field of accreditation must be at least seven (7) calendar days after the closing date of a PT study for the same field of accreditation.

c) A laboratory that analyzes and reports PT study results with an opening date of subsequent PT studies for the same field of accreditation that are closer than seven (7) days from the closing date of the previous PT study are invalid for the purposes of compliance with this Standard and are not counted toward the laboratory's PT history of the most recent three (3) attempts.

5.2.2 For WET testing: To maintain accreditation, the laboratory shall participate in one (1) WET PT study per calendar year for each accreditation FoPT that correspond to the fields of accreditation for which the laboratory is accredited.

a) This requirement can be met by annual participation in the Environmental Protection Agency (EPA) Discharge Monitoring Report-Quality Assurance (DMRQA) studies for WET, or

b) If the laboratory is not participating in an EPA DMRQA study for WET, the closing dates of subsequent PT study samples for WET testing PT studies must be no more than fourteen (14) months apart.

5.2.3 A laboratory that fails to analyze and report PT studies for a particular field of accreditation with the frequency specified in Sections 5.2.1 or 5.2.2 for which it seeks to maintain accreditation is charged with a failed PT study.

NOTE 1: A laboratory may withdraw from a PT study, but withdrawal from a PT study does not exempt the laboratory from analyzing and reporting a PT study as specified in Sections 5.2.1 and 5.2.2.

NOTE 2: "Acceptable" PT study scores from a PT Provider do not automatically result in a successful evaluation of a PT study by an AB. For example, failure to report an analytical method or reporting of an incorrect method, failure to provide the PT Provider with a release of results to the AB before the close of the study, failure to report results to the PT Provider before the closing date, failure to handle PT study samples in the same manner as routine environmental samples, etc., may be cause for an unsuccessful evaluation by an AB.

6.0 Requirements for Corrective Action

6.1 A laboratory that fails to successfully analyze a PT study for a particular FOA shall determine the root cause of the failure and take corrective action.

6.2 The laboratory shall document the root cause investigation and subsequent corrective action.

NOTE: The requirements for corrective action are described in Volume 1, Module 2 of this Standard.

6.3 The laboratory shall provide the root cause investigation and corrective action documentation to the Primary AB within thirty (30) calendar days of a request from the AB.

6.4 Failure to submit documentation of the root cause investigation or corrective action records, or both, to the AB within thirty (30) calendar days of the request from the Primary AB is due cause for suspension of accreditation for a particular FOA.

6.5 Documentation for WET corrective actions shall include:

a) a copy of the raw data used for the study;

b) a copy of the current Standard Reference Toxicant (SRT) control chart relevant to the PT study.

7.0 Requirements for Complaint Resolution

7.1 The laboratory shall submit questions about PT samples or performance evaluations made by the PT Provider to the PT Provider. If the PT Provider is not able or is unwilling to resolve the question to the satisfaction of the laboratory, the laboratory shall refer those questions to the PT Provider's PTPA.

7.2 The laboratory shall submit questions to its AB in regards to the AB's PT evaluation, if necessary.

8.0 Requirements for Reinstatement of Accreditation after Suspension or Revocation

8.1 A laboratory seeking to have its accreditation reinstated for an FoPT after suspension shall meet the requirements for continued accreditation as described in Section 5.2 of this module.

8.2 A laboratory seeking to have its accreditation reinstated for an FoPT after revocation shall meet the requirements for initial accreditation as described in Section 5.1 of this module.

8.3 A laboratory seeking to have its accreditation reinstated for an FoPT after suspension due to not supplying a requested corrective action report shall meet the requirements for continued accreditation as described in Section 5.2 of this module.

ENVIRONMENTAL LABORATORY SECTOR

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Module 2: Quality Systems General Requirements

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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. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

This Standard supersedes and replaces preceding documents in whole or in part. It is conformant with the requirements of ISO/IEC 17025:2005, and includes applicable clauses from that international standard. The ISO clauses are provided *in italics.* Additional TNI text is provided in a normal font.

This Standard may be used by any organization that wishes to implement a program for the accreditation of environmental laboratories.

Standard Revision History

All Sections

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Working Draft Standard Published	June 26, 2011
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Section 5.5.13.1

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VOLUME 1, MODULE 2
Quality Systems General Requirements

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VOLUME 1, MODULE 2
Quality Systems General Requirements

1.0 Introduction, Scope, and Applicability

1.1 Introduction

Each laboratory shall have a quality system. The laboratory's quality system is the means by which an organization ensures the quality of the products or services it provides and includes a variety of management, technical, and administrative elements such as:

a) policies and objectives,
b) procedures and practices,
c) organizational authority,
d) responsibilities, and
e) performance measures.

The quality system provides the framework for planning, implementing, assessing, and improving work performed by an organization so as to provide the client with data of known and documented quality, sufficient to evaluate the usability of the data to the clients needs. The quality system shall be documented in the laboratory's quality manual and related quality documentation, and shall be referenced in the quality manual.

This Standard contains detailed quality system requirements for consistent and uniform implementation by the laboratories conducting testing and the consistent and uniform evaluation of those laboratories by accreditation bodies. Each laboratory seeking accreditation under this Standard shall ensure that they are implementing their quality system and that all Quality Control (QC) procedures specified in this module are being followed. The Quality Assurance (QA) policies, which establish QC procedures, are applicable to environmental laboratories regardless of size and complexity.

This Standard is consistent with ISO/IEC 17025:2005 requirements that are relevant to the scope of environmental testing services.

All items identified in this document shall be available for an on-site assessment.

1.2 Scope

The requirements in this document give the basis for a laboratory's quality system in order to carry out environmental tests. It covers testing performed using reference methods, non-reference methods, and laboratory-developed methods. This document contains the essential elements required to establish a quality system that produces data of known and documented quality, and demonstrates proficiency through the use of proficiency testing and employee training.

The general requirements of this document apply to all organizations performing environmental tests, regardless of the number of personnel or the degree of environmental testing activities. When the use of the data requires compliance with the Standards, these Standards shall be followed.

This document is for use by laboratories, clients, regulatory authorities, and accreditation bodies to ensure the laboratory has appropriate management and technical quality systems to perform environmental testing. This document specifies technical, managerial, and documentation requirements needed for assessment by organizations or accreditation bodies to grant approval. This document provides the requirements needed for laboratory accreditation. If the requirements of this document are met, the laboratory operates a quality system in conformance with the

applicable clauses of ISO/IEC 17025:2005. The ISO/IEC 17025:2005 language is incorporated verbatim into this Standard, and appears as italicized text.

The notes given provide clarification of the text, examples and/or guidance. They do not contain requirements and do not form an integral part of this Standard.

2.0 Normative References *(ISO/IEC 17025:2005, Clause 2)*

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/IEC 17000, Conformity assessment -- Vocabulary and general principles.

VIM, International vocabulary of basic and general terms in metrology, issued by BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML.

NOTE: Further related standards, guides, etc., on subjects included in this International Standard are given in the Bibliography, Section 6.0.

3.0 Terms and Definitions

The relevant definitions listed in the referenced ISO/IEC documents apply when using those documents. Definitions related to this document, which are used differently or do not exist in the above references, are defined below.

3.1 Additional Terms and Definitions

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.

Analyst: The designated individual who performs the “hands-on” analytical methods and associated techniques, and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analyte: A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation).

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to

twenty (20) environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples.

Bias: The systematic or persistent distortion of a measurement process which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. Blanks include:

> **Method Blank:** A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).
2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference material used for calibration.

Certified Reference Material (CRM): Reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.

Chain of Custody Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. See also Legal Chain of Custody Protocols.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation, Alternate wavelength, Derivatization, Mass spectral interpretation, Alternative detectors, or Additional cleanup procedures.

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more useful form.

Demonstration of Capability: A procedure to establish the ability of the analyst to perform analyses with acceptable accuracy and precision.

Detection Limit: See Limit of Detection

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Finding: An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.

Holding Times: The maximum time that can elapse between two (2) specified activities.

In-depth Data Monitoring: When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Legal Chain of Custody Protocols: Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Limit(s) of Detection (LOD): The minimum result, which can be reliably discriminated from a blank with a predetermined confidence level. Also used is Detection Limit.

Limit(s) of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

Lot: A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.

Matrix: The substrate of a test sample.

Matrix Duplicate: A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Measurement System: A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

Method Detection Limit (MDL): One way to establish a Limit of Detection.

Mobile Laboratory: A portable, enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include, but are not limited to: trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (NMI).

Physical Parameter: A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical or biological components.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis.

Procedure: A specified way to carry out an activity or process. Procedures can be documented or not.

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.

Protocol: A detailed, written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Quality Assurance (QA): An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Control (QC): The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities.

Quality System Matrix: These matrix definitions are to be used for purposes of batch and QC requirements:

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device.

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Reference Material: Material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: (To be used to determine the extent of method validation in Modules 3-7.) A reference method is a published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a “standard method”, that term is equivalent to “reference method”). When a laboratory is required to analyze an analyte by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is not a regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte from another component that may be a potential interferent or that may behave similarly to the target analyte within the measurement system.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.

Standard Operating Procedures (SOPs): A written document that details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

Verification: Confirmation by examination and objective evidence that specified requirements have been met.

In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

3.2 Sources

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American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991

ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for Radiobioassay Laboratories

International Vocabulary of Basic and General Terms in Metrology (VIM): 1984. Issued by Bureau International des Poids et Mesures (BIPM), International Electrotechnical Commission (IEC), International Organization for Standardization (ISO)/IEC and International Organization of Legal Metrology (OIML)

National Institute of Standards and Technology (NIST)

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United States Environmental Protection Agency (US EPA) Quality Assurance Management Section (QAMS), Glossary of Terms of Quality Assurance Terms, 8/31/92 and 12/6/95

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Uniform Federal Policy for Quality Assurance Project Plans (UFP QAPP) March 2005

VIM – Draft edition October 2005

TNI Technical Modules, as follows:

Volume 1, Module 3 Quality Systems for Asbestos Testing

Volume 1, Module 4 Quality Systems for Chemical Testing

Volume 1, Module 5 Quality Systems for Microbiological Testing

Volume 1, Module 6 Quality Systems for Radiochemical Testing

Volume 1, Module 7 Quality Systems for Toxicity Testing

3.3 Exclusions and Exceptions

Reserved

4.0 Management Requirements

4.1 Organization *(ISO/IEC 17025:2005, Clause 4.1)*

4.1.1 The laboratory or the organization of which it is part shall be an entity that can be held legally responsible.

4.1.2 It is the responsibility of the laboratory to carry out its testing and calibration activities in such a way as to meet the requirements of this International Standard and to satisfy the needs of the customer, the regulatory authorities or organizations providing recognition.

4.1.3 The management system shall cover work carried out in the laboratory's permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

4.1.4 If the laboratory is part of an organization performing activities other than testing and/or calibration, the responsibilities of key personnel in the organization that have an involvement or influence on the testing and/or calibration activities of the laboratory shall be defined in order to identify potential conflicts of interest.

NOTE 1: Where a laboratory is part of a larger organization, the organizational arrangements should be such that departments having conflicting interests, such as production, commercial marketing or financing do not adversely influence the laboratory's compliance with the requirements of this International Standard.

NOTE 2: If the laboratory wishes to be recognized as a third-party laboratory, it should be able to demonstrate that it is impartial and that it and its personnel are free from any undue commercial, financial and other pressures which might influence their technical judgment. The third-party testing or calibration laboratory should not engage in any activities that may endanger the trust in its independence of judgment and integrity in relation to its testing or calibration activities.

4.1.5 The laboratory shall:

a) have managerial and technical personnel who, irrespective of other responsibilities, have the authority and resources needed to carry out their duties, including the implementation, maintenance and improvement of the management system, and to identify the occurrence of departures from the management system or from the procedures for performing tests and/or calibrations, and to initiate actions to prevent or minimize such departures (see also Section 5.2);

b) have arrangements to ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work;

c) have policies and procedures to ensure the protection of its customers' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results;

d) have policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment or operational integrity;

e) define the organization and management structure of the laboratory, its place in any parent organization, and the relationships between quality management, technical operations and support services;

f) specify the responsibility, authority and interrelationships of all personnel who manage, perform or verify work affecting the quality of the tests and/or calibrations;

g) provide adequate supervision of testing and calibration staff, including trainees, by persons familiar with methods and procedures, purpose of each test and/or calibration, and with the assessment of the test or calibration results;

h) have technical management which has overall responsibility for the technical operations and the provision of the resources needed to ensure the required quality of laboratory operations;

i) appoint a member of staff as quality manager (however named) who, irrespective of other duties and responsibilities, shall have defined responsibility and authority for ensuring that the management system related to quality is implemented and followed at all times; the quality manager shall have direct access to the highest level of management at which decisions are made on laboratory policy or resources;

j) appoint deputies for key managerial personnel (see Note);

k) ensure that its personnel are aware of the relevance and importance of their activities and how they contribute to the achievement of the objectives of the management system.

NOTE: Individuals may have more than one function and it may be impractical to appoint deputies for every function.

4.1.6 Top management shall ensure that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.

4.1.7 Additional Requirements for Laboratories

4.1.7.1 Where staffing is limited, the technical manager and the quality manager may be the same person. The laboratory's quality manager and/or his/her designee(s) shall:

a) serve as the focal point for QA/QC and be responsible for the oversight and/or review of QC data;

b) have functions independent from laboratory operations for which they have QA oversight;

c) be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

d) have documented training and/or experience in QA/QC procedures and the laboratory's quality system;

e) have a general knowledge of the analytical methods for which data review is performed;

f) arrange for or conduct internal audits as per Section 4.14 annually;

g) notify laboratory management of deficiencies in the quality system; and

h) monitor corrective actions.

4.1.7.2 The laboratory's technical manager(s), however named, and/or his/her designee(s) shall:

a) be a member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results;

b) be experienced in the fields of accreditation for which the laboratory is seeking accreditation;

c) have duties that include:

i. monitoring standards of performance in QC and QA, and

ii. monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data.

d) not be the technical manager(s) of more than one accredited environmental laboratory without authorization from the primary Accreditation Body. Circumstances to be considered in the decision to grant such authorization shall include:

i. the extent to which operating hours of the laboratories to be directed overlap,

ii adequacy of supervision in each laboratory, and

iii the availability of environmental laboratory services in the area served.

e) if absent for a period of time exceeding fifteen (15) consecutive calendar days shall designate another staff member meeting the qualifications of the technical manager(s) to temporarily perform this function. If this absence exceeds thirty-five (35) consecutive calendar days, the primary accreditation body shall be notified in writing; and

f) meet qualification requirements as specified in Section 5.2.6.1.

4.2 Management *(ISO/IEC 17025:2005, Clause 4.2)*

4.2.1 The laboratory shall establish, implement and maintain a management system appropriate to the scope of its activities. The laboratory shall document its policies, systems, programmes, procedures and instructions to the extent necessary to assure the quality of the test and/or calibration results. The system's documentation shall be communicated to, understood by, available to, and implemented by the appropriate personnel.

4.2.2 The laboratory's management system policies related to quality, including a quality policy statement, shall be defined in a quality manual (however named). The overall objectives shall be established, and shall be reviewed during management review. The quality policy statement shall be issued under the authority of top management. It shall include at least the following:

a) the laboratory management's commitment to good professional practice and to the quality of its testing and calibration in servicing its customers;

b) the management's statement of the laboratory's standard of service;

c) the purpose of the management system related to quality;

d) a requirement that all personnel concerned with testing and calibration activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work; and

e) the laboratory management's commitment to comply with this International Standard and to continually improve the effectiveness of the management system.

NOTE: The quality policy statement should be concise and may include the requirement that tests and/or calibrations shall always be carried out in accordance with stated methods and customers' requirements. When the test and/or calibration laboratory is part of a larger organization, some quality policy elements may be in other documents.

4.2.3 Top management shall provide evidence of commitment to the development and implementation of the management system and to continually improving its effectiveness.

4.2.4 Top management shall communicate to the organization the importance of meeting customer requirements as well as statutory and regulatory requirements.

4.2.5 The quality manual shall include or make reference to the supporting procedures including technical procedures. It shall outline the structure of the documentation used in the management system.

4.2.6 The roles and responsibilities of technical management and the quality manager, including their responsibility for ensuring compliance with this International Standard, shall be defined in the quality manual.

4.2.7 Top management shall ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented.

4.2.8 Additional Management System Requirements

4.2.8.1 The laboratory shall establish and maintain a documented data integrity system. There are four (4) required elements within a data integrity system. These are 1) data integrity training, 2) signed data integrity documentation for all laboratory employees, 3) periodic in-depth data monitoring, and 4) data integrity procedure documentation. The data integrity procedures shall be signed and dated by top management. The requirements for data integrity investigation are listed in Section 4.16. The requirements for data integrity training and documentation are listed in Section 5.2.7. Management shall annually review data integrity procedures and update as needed.

a) Laboratory management shall provide a procedure for confidential reporting of data integrity issues in their laboratory. A primary element of the procedure is to assure confidentiality and a receptive environment in which all employees may privately discuss ethical issues or report items of ethical concern.

b) In instances of ethical concern, the procedure shall include a process whereby laboratory management is to be informed of the need for any further detailed investigation.

4.2.8.2 The quality manager shall be responsible for maintaining the currency of the quality manual.

4.2.8.3 The quality manual shall contain:

a) document title;

b) laboratory's full name and address;

c) name, address (if different from above), and telephone number of individual(s) responsible for the laboratory;

d) identification of all major organizational units that are to be covered by this quality manual and the effective date of the version;

e) identification of the laboratory's approved signatories;

f) the signed and dated concurrence (with appropriate names and titles), of all responsible parties including the quality manager(s), technical manager(s), and the agent who is in charge of all laboratory activities, such as the laboratory director or laboratory manager;

g) the objectives of the quality system and contain or reference the laboratory's policies and procedures;

h) the laboratory's official quality policy statement, which shall include quality system objectives and management's commitment to ethical laboratory practices and to upholding the requirements of this Standard; and

i) a table of contents, and applicable lists of references, glossaries and appendices.

4.2.8.4 The quality manual shall contain or reference:

a) all maintenance, calibration and verification procedures used by the laboratory in conducting tests;

b) major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;

c) verification practices, which may include inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC schemes;

d) procedures for reporting analytical results;

e) the organization and management structure of the laboratory, its place in any parent organization, and relevant organizational charts;

f) procedures to ensure that all records required under this Standard are retained, as well as procedures for control and maintenance of documentation through a document control system that ensures that all standard operating procedures (SOPs), manuals, or documents clearly indicate the time period during which the procedure or document was in force;

g) job descriptions of key staff and reference to the job descriptions of other laboratory staff;

h) procedures for achieving traceability of measurements;

i) a list of all methods under which the laboratory performs its accredited testing;

j) procedures for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

k) procedures for handling samples;

l) procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

m) policy for permitting departures from documented policies and procedures or from standard specifications;

n) procedures for dealing with complaints;

o) procedures for protecting confidentiality (including national security concerns), and proprietary rights;

p) procedures for audits and data review;

q) procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training; and

r) policy addressing the use of unique electronic signatures, where applicable.

4.2.8.5 Laboratories shall maintain SOPs that accurately reflect all phases of current laboratory activities, such as assessing data integrity, corrective actions, handling customer complaints, and all methods.

a) These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents with adequate detail to allow someone similarly qualified, other than the analyst, to reproduce the procedures used to generate the test result.

b) The relevant SOPs shall be readily accessible to all personnel.

c) Each SOP shall clearly indicate the effective date of the document, the revision number, and the signature(s) of the approving authority.

d) Documents that contain sufficient information to perform the tests, do not need to be supplemented or rewritten as internal procedures if the documents are written in a way that they can be used as written. Any changes, including the use of a selected option, shall be documented and included in the laboratory's records.

e) The laboratory shall have and maintain an SOP for each accredited analyte or method.

f) The SOP may be a copy of a published or referenced method or may be written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described. Each method shall include or reference the following topics where applicable:

i. identification of the method;
ii. applicable matrix or matrices;
iii. limits of detection and quantitation;
iv. scope and application, including analytes to be analyzed;
v. summary of the method;
vi. definitions;
vii. interferences;
viii. safety;
ix. equipment and supplies;
x. reagents and standards;
xi. sample collection, preservation, shipment and storage;
xii. quality control;
xiii. calibration and standardization;
xiv. procedure;
xv. data analysis and calculations;
xvi. method performance;
xvii. pollution prevention;
xviii. data assessment and acceptance criteria for QC measures;
xix. corrective actions for out-of-control data;
xx. contingencies for handling out-of-control or unacceptable data;
xxi. waste management;
xxii. references; and
xxiii. any tables, diagrams, flowcharts and validation data.

4.3 Document Control *(ISO/IEC 17025:2005, Clause 4.3)*

4.3.1 General

The laboratory shall establish and maintain procedures to control all documents that form part of its management system (internally generated or from external sources), such as regulations, standards, other normative documents, test and/or calibration methods, as well as drawings, software, specifications, instructions and manuals.

NOTE 1: In this context "document" could be policy statements, procedures, specifications, calibration tables, charts, text books, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic or written.

NOTE 2: The control of data related to testing and calibration is covered in 5.4.7. The control of records is covered in 4.13.

4.3.2 Document Approval and Issue

4.3.2.1 All documents issued to personnel in the laboratory as part of the management system shall be reviewed and approved for use by authorized personnel prior to issue. A master list or an equivalent document control procedure identifying the current revision status and distribution of documents in the management system shall be established and shall be readily available to preclude the use of invalid and/or obsolete documents.

4.3.2.2 The procedure(s) adopted shall ensure that:

a) authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed;

b) documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements;

c) invalid or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use;

d) obsolete documents retained for either legal or knowledge preservation purposes are suitably marked.

4.3.2.3 Management system documents generated by the laboratory shall be uniquely identified. Such identification shall include the date of issue and/or revision identification, page numbering, and the total number of pages or a mark to signify the end of the document, and the issuing authority(ies).

4.3.3 Document Changes

4.3.3.1 Changes to documents shall be reviewed and approved by the same function that performed the original review unless specifically designated otherwise. The designated personnel shall have access to pertinent background information upon which to base their review and approval.

4.3.3.2 Where practicable, the altered or new text shall be identified in the document or the appropriate attachments.

4.3.3.3 If the laboratory's document control system allows for the amendment of documents by hand pending the re-issue of the documents, the procedures and authorities for such amendments shall be defined. Amendments shall be clearly marked, initialled and dated. A revised document shall be formally re-issued as soon as practicable.

4.3.3.4 Procedures shall be established to describe how changes in documents maintained in computerized systems are made and controlled.

4.4 Review of Requests, Tenders, and Contracts *(ISO/IEC 17025:2005, Clause 4.4)*

4.4.1 The laboratory shall establish and maintain procedures for the review of requests, tenders and contracts. The policies and procedures for these reviews leading to a contract for testing and/or calibration shall ensure that:

a) the requirements, including the methods to be used, are adequately defined, documented and understood (see 5.4.2);

b) the laboratory has the capability and resources to meet the requirements;

c) the appropriate test and/or calibration method is selected and is capable of meeting the customers' requirements (see 5.4.2).

Any differences between the request or tender and the contract shall be resolved before any work commences. Each contract shall be acceptable both to the laboratory and the customer.

NOTE 1: The request, tender and contract review should be conducted in a practical and efficient manner, and the effect of financial, legal and time schedule aspects should be taken into account. For internal customers, reviews of requests, tenders and contracts can be performed in a simplified way.

NOTE 2: The review of capability should establish that the laboratory possesses the necessary physical, personnel and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the tests and/or calibrations in question. The review may also encompass results of earlier participation in interlaboratory comparisons or proficiency testing and/or the running of trial test or calibration programmes using samples or items of known value in order to determine uncertainties of measurement, limits of detection, confidence limits, etc.

NOTE 3: A contract may be any written or oral agreement to provide a customer with testing and/or calibration services.

4.4.2 Records of reviews, including any significant changes, shall be maintained. Records shall also be maintained of pertinent discussions with a customer relating to the customer's requirements or the results of the work during the period of execution of the contract.

NOTE: For review of routine and other simple tasks, the date and the identification (e.g. the initials) of the person in the laboratory responsible for carrying out the contracted work are considered adequate. For repetitive routine tasks, the review need be made only at the initial enquiry stage or on granting of the contract for on-going routine work performed under a general agreement with the customer, provided that the customer's requirements remain unchanged. For new, complex or advanced testing and/or calibration tasks, a more comprehensive record should be maintained.

4.4.3 The review shall also cover any work that is subcontracted by the laboratory.

4.4.4 The customer shall be informed of any deviation from the contract.

4.4.5 If a contract needs to be amended after work has commenced, the same contract review process shall be repeated and any amendments shall be communicated to all affected personnel.

4.5 Subcontracting of Environmental Tests *(ISO/IEC 17025:2005, Clause 4.5)*

4.5.1 When a laboratory subcontracts work, whether because of unforeseen reasons (e.g. workload, need for further expertise or temporary incapacity) or on a continuing basis (e.g. through permanent subcontracting, agency or franchising arrangements), this work shall be placed with a competent subcontractor. A competent subcontractor is one that, for example, complies with this International Standard for the work in question.

4.5.2 The laboratory shall advise the customer of the arrangement in writing and, when appropriate, gain the approval of the customer, preferably in writing.

4.5.3 The laboratory is responsible to the customer for the subcontractor's work, except in the case where the customer or a regulatory authority specifies which subcontractor is to be used.

4.5.4 The laboratory shall maintain a register of all subcontractors that it uses for tests and/or calibrations and a record of the evidence of compliance with this International Standard for the work in question.

4.5.5 When a laboratory subcontracts work, this work shall be placed with a laboratory accredited to this Standard for the tests to be performed or with a laboratory that meets applicable statutory and regulatory requirements for performing the tests and submitting the results of tests performed. The laboratory performing the subcontracted work shall be indicated in the final report. The laboratory shall make a copy of the subcontractor's report available to the client when requested.

4.6 Purchasing Services and Supplies *(ISO/IEC 17025:2005, Clause 4.6)*

4.6.1 The laboratory shall have a policy and procedure(s) for the selection and purchasing of services and supplies it uses that affect the quality of the tests and/or calibrations. Procedures shall exist for the purchase, reception and storage of reagents and laboratory consumable materials relevant for the tests and calibrations.

4.6.2 The laboratory shall ensure that purchased supplies and reagents and consumable materials that affect the quality of tests and/or calibrations are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the tests and/or calibrations concerned. These services and supplies used shall comply with specified requirements. Records of actions taken to check compliance shall be maintained.

4.6.3 Purchasing documents for items affecting the quality of laboratory output shall contain data describing the services and supplies ordered. These purchasing documents shall be reviewed and approved for technical content prior to release.

NOTE: The description may include type, class, grade, precise identification, specifications, drawings, inspection instructions, other technical data including approval of test results, the quality required and the management system standard under which they were made.

4.6.4 The laboratory shall evaluate suppliers of critical consumables, supplies and services which affect the quality of testing and calibration, and shall maintain records of these evaluations and list those approved.

4.7 Service to the Client *(ISO/IEC 17025:2005, Clause 4.7)*

4.7.1 The laboratory shall be willing to cooperate with customers or their representatives in clarifying the customer's request and in monitoring the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other customers.

NOTE 1: Such cooperation may include:

a) providing the customer or the customer's representative reasonable access to relevant areas of the laboratory for the witnessing of tests and/or calibrations performed for the customer;

b) preparation, packaging, and dispatch of test and/or calibration items needed by the customer for verification purposes.

NOTE 2: Customers value the maintenance of good communication, advice and guidance in technical matters, and opinions and interpretations based on results. Communication with the customer, especially in large assignments, should be maintained throughout the work. The laboratory should inform the customer of any delays or major deviations in the performance of the tests and/or calibrations.

4.7.2 The laboratory shall seek feedback, both positive and negative, from its customers. The feedback shall be used and analysed to improve the management system, testing and calibration activities and customer service.

NOTE: Examples of the types of feedback include customer satisfaction surveys and review of test or calibration reports with customers.

4.8 Complaints *(ISO/IEC 17025:2005, Clause 4.8)*

The laboratory shall have a policy and procedure for the resolution of complaints received from customers or other parties. Records shall be maintained of all complaints and of the investigations and corrective actions taken by the laboratory (see also 4.11).

4.9 Control of Nonconforming Environmental Testing Work *(ISO/IEC 17025:2005, Clause 4.9)*

4.9.1 The laboratory shall have a policy and procedures that shall be implemented when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its own procedures or the agreed requirements of the customer. The policy and procedures shall ensure that:

a) the responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding of test reports and calibration certificates, as necessary) are defined and taken when nonconforming work is identified;

b) an evaluation of the significance of the nonconforming work is made;

c) correction is taken immediately, together with any decision about the acceptability of the nonconforming work;

d) where necessary, the customer is notified and work is recalled;

e) the responsibility for authorizing the resumption of work is defined.

NOTE: Identification of nonconforming work or problems with the management system or with testing and/or calibration activities can occur at various places within the management system and technical operations. Examples are customer complaints, quality control, instrument calibration, checking of consumable materials, staff observations or supervision, test report and calibration certificate checking, management reviews and internal or external audits.

4.9.2 Where the evaluation indicates that the nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the corrective action procedures given in 4.11 shall be promptly followed.

4.10 Improvement *(ISO/IEC 17025:2005, Clause 4.10)*

The laboratory shall continually improve the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review.

4.11 Corrective Action *(ISO/IEC 17025:2005, Clause 4.11)*

4.11.1 General

The laboratory shall establish a policy and a procedure and shall designate appropriate authorities for implementing corrective action when nonconforming work or departures from the policies and procedures in the management system or technical operations have been identified.

NOTE: A problem with the management system or with the technical operations of the laboratory may be identified through a variety of activities, such as control of nonconforming work, internal or external audits, management reviews, and feedback from customers and from staff observations.

4.11.2 Cause Analysis

The procedure for corrective action shall start with an investigation to determine the root cause(s) of the problem.

NOTE: Cause analysis is the key and sometimes the most difficult part in the corrective action procedure. Often the root cause is not obvious and thus a careful analysis of all potential causes of the problem is required. Potential causes could include customer requirements, the samples, sample specifications, methods and procedures, staff skills and training, consumables, or equipment and its calibration.

4.11.3 Selection and Implementation of Corrective Actions

Where corrective action is needed, the laboratory shall identify potential corrective actions. It shall select and implement the action(s) most likely to eliminate the problem and to prevent recurrence.

Corrective actions shall be to a degree appropriate to the magnitude and the risk of the problem.

The laboratory shall document and implement any required changes resulting from corrective action investigations.

4.11.4 Monitoring of Corrective Actions

The laboratory shall monitor the results to ensure that the corrective actions taken have been effective.

4.11.5 Additional Audits

Where the identification of nonconformities or departures casts doubts on the laboratory's compliance with its own policies and procedures, or on its compliance with this International Standard, the laboratory shall ensure that the appropriate areas of activity are audited in accordance with 4.14 as soon as possible.

NOTE: Such additional audits often follow the implementation of the corrective actions to confirm their effectiveness. An additional audit should be necessary only when a serious issue or risk to the business is identified.

4.11.6 The laboratory shall have documented procedure(s) to address Sections 4.11.1 and 4.11.3 through 4.11.5. These procedure(s) shall also include:

a) which individual(s) or positions are responsible for assessing each QC data type; and

b) which individual(s) or positions are responsible for initiating and/or recommending corrective actions.

4.11.7 Cause analysis described in Section 4.11.2 applies to failures that indicate a systematic error.

4.12 Preventive Action *(ISO/IEC 17025:2005, Clause 4.12)*

4.12.1 Needed improvements and potential sources of nonconformities, either technical or concerning the management system, shall be identified. When improvement opportunities are identified or if preventive action is required, action plans shall be developed, implemented and monitored to reduce the likelihood of the occurrence of such nonconformities and to take advantage of the opportunities for improvement.

4.12.2 Procedures for preventive actions shall include the initiation of such actions and the application of controls to ensure that they are effective.

NOTE 1: Preventive action is a pro-active process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

NOTE 2: Apart from the review of the operational procedures, the preventive action might involve analysis of data, including trend and risk analyses and proficiency-testing results.

4.13 Control of Records *(ISO/IEC 17025:2005, Clause 4.13)*

4.13.1 General

4.13.1.1 The laboratory shall establish and maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records shall include reports from internal audits and management reviews as well as records of corrective and preventive actions.

4.13.1.2 All records shall be legible and shall be stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss. Retention times of records shall be established.

NOTE: Records may be in any media, such as hard copy or electronic media.

4.13.1.3 All records shall be held secure and in confidence.

4.13.1.4 The laboratory shall have procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records.

4.13.2 Technical Records

4.13.2.1 The laboratory shall retain records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each test report or calibration certificate issued, for a defined period. The records for each test or calibration shall contain sufficient information to facilitate, if possible, identification of factors affecting the

uncertainty and to enable the test or calibration to be repeated under conditions as close as possible to the original. The records shall include the identity of personnel responsible for the sampling, performance of each test and/or calibration and checking of results.

NOTE 1: In certain fields it may be impossible or impractical to retain records of all original observations.

NOTE 2: Technical records are accumulations of data (see 5.4.7) and information which result from carrying out tests and/or calibrations and which indicate whether specified quality or process parameters are achieved. They may include forms, contracts, work sheets, work books, check sheets, work notes, control graphs, external and internal test reports and calibration certificates, customers' notes, papers and feedback.

4.13.2.2 Observations, data and calculations shall be recorded at the time they are made and shall be identifiable to the specific task.

4.13.2.3 When mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records shall be signed or initialled by the person making the correction. In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data.

4.13.3 Additional Requirements

a) The laboratory shall establish a record keeping system that allows the history of the sample and associated data to be readily understood through the documentation. This system shall produce unequivocal, accurate records that document all laboratory activities such as laboratory facilities, equipment, analytical methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification, and inter-laboratory transfers of samples and/or extracts.

b) The laboratory shall retain all records for a minimum of five (5) years from generation of the last entry in the records.

c) Records shall be available to the accreditation body.

d) Records that are stored only on electronic media shall be supported by the hardware and software necessary for their retrieval.

e) Access to archived information shall be documented with an access log.

f) All information necessary for the historical reconstruction of data shall be maintained by the laboratory.

i. all raw data, whether hard copy or electronic, for calibrations, samples and QC measures, including analysts' worksheets and data output records (chromatograms, strip charts, and other instrument response readout records);

ii. a written description or reference to the specific method used, which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

iii. laboratory sample ID code;

iv. date of analysis;

v. time of analysis is required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., extractions and incubations);

vi. instrumentation identification and instrument operating conditions/parameters (or reference to such data);

vii. all manual calculations;

viii. analyst or operator initials/signature or electronic identification;

ix. sample preparation, including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

x. test results;

xi. standard and reagent origin, receipt, preparation, and use;

xii. calibration criteria, frequency and acceptance criteria;

xiii. data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

xiv. QC protocols and assessment;

xv. electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

xvi. method performance criteria including expected QC requirements;

xvii. proficiency test results;

xviii. records of demonstration of capability for each analyst; and

xix. a record of names, initials, and signatures for all individuals who are responsible for signing or initialing any laboratory record.

g) All generated data, except those that are generated by automated data collection systems, shall be recorded legibly in permanent ink.

i. An individual making corrections to records shall date and initial the correction.

ii. Corrections due to reasons other than transcription errors shall specify the reason for the correction.

h) The laboratory shall have a plan to ensure that the records are maintained or transferred according to the clients' instructions in the event that a laboratory transfers ownership or goes out of business. In addition, appropriate regulatory and state legal requirements concerning laboratory records shall be followed.

4.14 Internal Audits *(ISO/IEC 17025:2005, Clause 4.14)*

4.14.1 The laboratory shall periodically, and in accordance with a predetermined schedule and procedure, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the management system and this International Standard. The internal audit programme shall address all elements of the management system, including the testing and/or calibration activities. It is the responsibility of the quality manager to plan and organize audits as

required by the schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.

NOTE: The cycle for internal auditing should normally be completed in one year.

4.14.2 When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test or calibration results, the laboratory shall take timely corrective action, and shall notify customers in writing if investigations show that the laboratory results may have been affected.

4.14.3 The area of activity audited, the audit findings and corrective actions that arise from them shall be recorded.

4.14.4 Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken.

4.14.5 Additional Items

a) The laboratory shall have a policy that specifies the time frame for notifying a client of events that cast doubt on the validity of the results.

b) The laboratory management shall ensure that these actions are discharged within the agreed time frame.

c) The internal audit schedule shall be completed annually.

4.15 Management Reviews *(ISO/IEC 17025:2005, Clause 4.15)*

4.15.1 In accordance with a predetermined schedule and procedure, the laboratory's top management shall periodically conduct a review of the laboratory's management system and testing and/or calibration activities to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. The review shall take account of:

- *the suitability of policies and procedures;*
- *reports from managerial and supervisory personnel;*
- *the outcome of recent internal audits;*
- *corrective and preventive actions;*
- *assessments by external bodies;*
- *the results of interlaboratory comparisons or proficiency tests;*
- *changes in the volume and type of the work;*
- *customer feedback;*
- *complaints;*
- *recommendations for improvement;*
- *other relevant factors, such as quality control activities, resources, and staff training.*

NOTE 1: A typical period for conducting a management review is once every 12 months.

NOTE 2: Results should feed into the laboratory planning system and should include the goals, objectives and action plans for the coming year.

NOTE 3: A management review includes consideration of related subjects at regular management meetings.

4.15.2 Findings from management reviews and the actions that arise from them shall be recorded. The management shall ensure that those actions are carried out within an appropriate and agreed timescale.

4.15.3 Management review shall be completed on an annual basis.

4.16 Data Integrity Investigations

All investigations resulting from data integrity issues should be conducted in a confidential manner until they are completed. These investigations shall be documented, as well as any notifications made to clients receiving any affected data.

5.0 Technical Requirements

5.1 General *(ISO/IEC 17025:2005, Clause 5.1)*

5.1.1 Many factors determine the correctness and reliability of the tests and/or calibrations performed by a laboratory. These factors include contributions from:

- *human factors (5.2);*
- *accommodation and environmental conditions (5.3);*
- *test and calibration methods and method validation (5.4);*
- *equipment (5.5);*
- *measurement traceability (5.6);*
- *sampling (5.7);*
- *the handling of test and calibration items (5.8).*

5.1.2 The extent to which the factors contribute to the total uncertainty of measurement differs considerably between (types of) tests and between (types of) calibrations. The laboratory shall take account of these factors in developing test and calibration methods and procedures, in the training and qualification of personnel, and in the selection and calibration of the equipment it uses.

5.2 Personnel *(ISO/IEC 17025:2005, Clause 5.2)*

5.2.1 The laboratory management shall ensure the competence of all who operate specific equipment, perform tests and/or calibrations, evaluate results, and sign test reports and calibration certificates. When using staff who are undergoing training, appropriate supervision shall be provided. Personnel performing specific tasks shall be qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

NOTE 1: In some technical areas (e.g. non-destructive testing) it may be required that the personnel performing certain tasks hold personnel certification. The laboratory is responsible for fulfilling specified personnel certification requirements. The requirements for personnel certification might be regulatory, included in the standards for the specific technical field, or required by the customer.

NOTE 2: The personnel responsible for the opinions and interpretation included in test reports should, in addition to the appropriate qualifications, training, experience and satisfactory knowledge of the testing carried out, also have:

- *relevant knowledge of the technology used for the manufacturing of the items, materials, products, etc. tested, or the way they are used or intended to be used, and of the defects or degradations which may occur during or in service;*
- *knowledge of the general requirements expressed in the legislation and standards; and*
- *an understanding of the significance of deviations found with regard to the normal use of the items, materials, products, etc. concerned.*

5.2.2 The management of the laboratory shall formulate the goals with respect to the education, training and skills of the laboratory personnel. The laboratory shall have a policy and procedures for identifying training needs and providing training of personnel. The training programme shall be relevant to the present and anticipated tasks of the laboratory. The effectiveness of the training actions taken shall be evaluated.

5.2.3 The laboratory shall use personnel who are employed by, or under contract to, the laboratory. Where contracted and additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's management system.

5.2.4 The laboratory shall maintain current job descriptions for managerial, technical and key support personnel involved in tests and/or calibrations.

NOTE: Job descriptions can be defined in many ways. As a minimum, the following should be defined:

- *the responsibilities with respect to performing tests and/or calibrations;*
- *the responsibilities with respect to the planning of tests and/or calibrations and evaluation of results;*
- *the responsibilities for reporting opinions and interpretations;*
- *the responsibilities with respect to method modification and development and validation of new methods;*
- *expertise and experience required;*
- *qualifications and training programmes;*
- *managerial duties.*

5.2.5 The management shall authorize specific personnel to perform particular types of sampling, test and/or calibration, to issue test reports and calibration certificates, to give opinions and interpretations and to operate particular types of equipment. The laboratory shall maintain records of the relevant authorization(s), competence, educational and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel. This information shall be readily available and shall include the date on which authorization and/or competence is confirmed.

All references to Calibration Certificates in *ISO/IEC 17025:2005* are not applicable to environmental testing.

5.2.6 Additional Personnel Requirements

5.2.6.1 Technical Manager Qualifications

The applicable requirements for technical managers are given below.

a) Any technical manager of an accredited environmental laboratory engaged in chemical analysis shall be a person with a bachelor’s degree in the chemical, environmental, biological

sciences, physical sciences or engineering, with at least twenty-four (24) college semester credit hours in chemistry and at least two (2) years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one (1) year of experience.

b) Any technical manager of an accredited environmental laboratory limited to inorganic chemical analysis, other than metals analysis, shall be a person with at least an earned associate's degree in the chemical, physical or environmental sciences, or two (2) years of equivalent and successful college education, with a minimum of sixteen (16) college semester credit hours in chemistry. In addition, such a person shall have at least two (2) years of experience performing such analysis.

c) Any technical manager of an accredited environmental laboratory engaged in microbiological or biological analysis shall be a person with a bachelor's degree in microbiology, biology, chemistry, environmental sciences, physical sciences or engineering with a minimum of sixteen (16) college semester credit hours in general microbiology and biology and at least two (2) years of experience in the environmental analysis of representative analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one (1) year of experience.

   A person with an associate's degree in an appropriate field of the sciences or applied sciences, with a minimum of four (4) college semester credit hours in general microbiology may be the technical manager(s) of a laboratory engaged in microbiological analysis limited to fecal coliform, total coliform, E. coli, and standard plate count. Two (2) years of equivalent and successful college education, including the microbiology requirement, may be substituted for the associate's degree. In addition, each person shall have one (1) year of experience in microbiological analyses.

d) Any technical manager of an accredited environmental laboratory engaged in radiological analysis shall be a person with a bachelor's degree in chemistry, environmental, biological sciences, physical sciences or engineering with twenty-four (24) college semester credit hours of chemistry with two (2) or more years of experience in the radiological analysis of environmental samples. A master's or doctoral degree in one of the above disciplines may be substituted for one (1) year experience.

e) The technical manager(s) of an accredited environmental laboratory engaged in microscopic examination of asbestos and/or airborne fibers shall meet the following requirements:

   i. For procedures requiring the use of a transmission electron microscope, a bachelor's degree, successful completion of courses in the use of the instrument, and one (1) year of experience, under supervision, in the use of the instrument. Such experience shall include the identification of minerals.

   ii. For procedures requiring the use of a polarized light microscope, an associate's degree or two (2) years of college study, successful completion of formal coursework in polarized light microscopy, and one (1) year of experience, under supervision, in the use of the instrument. Such experience shall include the identification of minerals.

   iii. For procedures requiring the use of a phase contrast microscope, as in the determination of airborne fibers, an associate's degree or two (2) years of college study, documentation of successful completion of formal coursework in phase contrast microscopy, and one (1) year of experience, under supervision, in the use of the instrument.

f) Any technical manager of an accredited environmental laboratory engaged in the examination of radon in air shall have at least an associate's degree or two (2) years of college and one (1) year of experience in radiation measurements, including at least one (1) year of experience in the measurement of radon and/or radon progeny.

5.2.6.2 Technical Manager Qualification Exceptions

a) Notwithstanding any other provision of this Section, a full-time employee of a drinking water or sewage treatment facility who holds a valid treatment plant operator's certificate appropriate to the nature and size of such facility shall be deemed to meet the educational requirements as the technical manager. A technical manager shall have two (2) year testing experience devoted exclusively to the testing of environmental samples specified in the scope of the facility's regulatory permit. Such accreditation for a water treatment facility and/or a sewage treatment facility shall be limited to the scope of that facility's regulatory permit.

b) A full-time employee of an industrial waste treatment facility with a minimum of two (2) years of experience under supervision in testing of environmental samples taken within such facility for the scope of that facility's regulatory permit shall be deemed to meet the requirements for serving as the technical manager of an accredited laboratory. Such accreditation for an industrial waste treatment facility shall be limited to the scope of that facility's regulatory permit.

c) Persons who do not meet the education credential requirements, but possess the requisite experience of Section 5.2.6.1, shall qualify as technical manager(s) subject to the following conditions.

i. The person shall be a technical manager of the laboratory on the date the laboratory applies for accreditation and/or becomes subject to accreditation under this Standard, and shall have been a technical manager in that laboratory continuously for the previous twelve (12) months or more.

ii. The person will be approved as a technical manager for only those fields of accreditation for which he/she has been technical manager in that laboratory for the previous twelve (12) months or more.

iii. A person who is admitted as a technical manager under these conditions, and leaves the laboratory, will be eligible for hire as a technical manager for the same fields of accreditation in another accredited laboratory.

5.2.7 Data Integrity Training

Data integrity training shall be provided as a formal part of new employee orientation and shall also be provided on an annual basis for all current employees. Employees are required to understand that any infractions of the laboratory data integrity procedures shall result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment or civil/criminal prosecution. The initial data integrity training and the annual refresher training shall have a signature attendance sheet or other form of documentation that demonstrates all staff have participated and understand their obligations related to data integrity.

Data integrity training requires emphasis on the importance of proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially deficient. The topics covered in such training shall be documented in writing (such as an agenda) and provided to all trainees. At a minimum, the following topics and activities shall be included:

a) organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues, and record keeping;

b) training, including discussion regarding all data integrity procedures;

c) data integrity training documentation;

d) in-depth data monitoring and data integrity procedure documentation; and

e) specific examples of breaches of ethical behavior such as improper data manipulations, adjustments of instrument time-clocks, and inappropriate changes in concentrations of standards.

The data integrity procedures may also include written ethics agreements, examples of improper practices, examples of improper chromatographic manipulations, requirements for external ethics program training, and any external resources available to employees.

5.3 Accommodation and Environmental Conditions *(ISO/IEC 17025:2005, Clause 5.3)*

5.3.1 Laboratory facilities for testing and/or calibration, including but not limited to energy sources, lighting and environmental conditions, shall be such as to facilitate correct performance of the tests and/or calibrations. The laboratory shall ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any measurement. Particular care shall be taken when sampling and tests and/or calibrations are undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of tests and calibrations shall be documented.

5.3.2 The laboratory shall monitor, control and record environmental conditions as required by the relevant specifications, methods and procedures or where they influence the quality of the results. Due attention shall be paid, for example, to biological sterility, dust, electromagnetic disturbances, radiation, humidity, electrical supply, temperature, and sound and vibration levels, as appropriate to the technical activities concerned. Tests and calibrations shall be stopped when the environmental conditions jeopardize the results of the tests and/or calibrations.

5.3.3 There shall be effective separation between neighbouring areas in which there are incompatible activities. Measures shall be taken to prevent cross-contamination.

5.3.4 Access to and use of areas affecting the quality of the tests and/or calibrations shall be controlled. The laboratory shall determine the extent of control based on its particular circumstances.

5.3.5 Measures shall be taken to ensure good housekeeping in the laboratory. Special procedures shall be prepared where necessary.

5.4 Environmental Methods and Method Validation

All references to Calibration Laboratories and Calibration Methods in *ISO/IEC 17025:2005* in these Clauses are not applicable to environmental testing.

5.4.1 General (*ISO/IEC 17025:2005, Clause 5.4.1)*

The laboratory shall use appropriate methods and procedures for all tests and/or calibrations within its scope. These include sampling, handling, transport, storage and preparation of items to be tested and/or calibrated, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of test and/or calibration data.

The laboratory shall have instructions on the use and operation of all relevant equipment, and on the handling and preparation of items for testing and/or calibration, or both, where the absence of

such instructions could jeopardize the results of tests and/or calibrations. All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be kept up to date and shall be made readily available to personnel (see 4.3). Deviation from test and calibration methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the customer.

NOTE: International, regional or national standards or other recognized specifications that contain sufficient and concise information on how to perform the tests and/or calibrations do not need to be supplemented or rewritten as internal procedures if these standards are written in a way that they can be used as published by the operating staff in a laboratory. It may be necessary to provide additional documentation for optional steps in the method or additional details.

5.4.2 Selection of Methods (*ISO/IEC 17025:2005, Clause 5.4.2)*

The laboratory shall use test and/or calibration methods, including methods for sampling, which meet the needs of the customer and which are appropriate for the tests and/or calibrations it undertakes. Methods published in international, regional or national standards shall preferably be used. The laboratory shall ensure that it uses the latest valid edition of a standard unless it is not appropriate or possible to do so. When necessary, the standard shall be supplemented with additional details to ensure consistent application.

When the customer does not specify the method to be used, the laboratory shall select appropriate methods that have been published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment. Laboratory-developed methods or methods adopted by the laboratory may also be used if they are appropriate for the intended use and if they are validated. The customer shall be informed as to the method chosen. The laboratory shall confirm that it can properly operate standard methods before introducing the tests or calibrations. If the standard method changes, the confirmation shall be repeated.

The laboratory shall inform the customer when the method proposed by the customer is considered to be inappropriate or out of date.

5.4.3 Laboratory-Developed Methods *(ISO/IEC 17025:2005, Clause 5.4.3)*

The introduction of test and calibration methods developed by the laboratory for its own use shall be a planned activity and shall be assigned to qualified personnel equipped with adequate resources.

Plans shall be updated as development proceeds and effective communication amongst all personnel involved shall be ensured.

5.4.4 Non-Standard Methods *(ISO/IEC 17025:2005, Clause 5.4.4*)

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

NOTE: For new test and/or calibration methods, procedures should be developed prior to the tests and/or calibrations being performed and should contain at least the following information:

a) appropriate identification;

b) scope;

c) description of the type of item to be tested or calibrated;

d) parameters or quantities and ranges to be determined;

e) apparatus and equipment, including technical performance requirements;

f) reference standards and reference materials required;

g) environmental conditions required and any stabilization period needed;

h) description of the procedure, including

- *affixing of identification marks, handling, transporting, storing and preparation of items,*
- *checks to be made before the work is started,*
- *checks that the equipment is working properly and, where required, calibration and adjustment of the equipment before each use,*
- *the method of recording the observations and results,*
- *any safety measures to be observed;*

i) criteria and/or requirements for approval/rejection;

j) data to be recorded and method of analysis and presentation;

k) the uncertainty or the procedure for estimating uncertainty.

5.4.4.1 The note in 5.4.4 above, which includes a – k, shall be considered during the development of the method.

5.4.4.2 The laboratory shall ensure that once the method has been developed, a Standard Operating Procedure as outlined in 4.2.8.5 f shall be written.

5.4.5 Validation of Methods *(ISO/IEC 17025:2005, Clause 5.4.5)*

5.4.5.1 Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

5.4.5.2 The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard 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.

NOTE 1: Validation may include procedures for sampling, handling and transportation.

NOTE 2: The techniques used for the determination of the performance of a method should be one of, or a combination of, the following:

- *calibration using reference standards or reference materials;*
- *comparison of results achieved with other methods;*
- *interlaboratory comparisons;*
- *systematic assessment of the factors influencing the result;*
- *assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.*

NOTE 3: When some changes are made in the validated non-standard methods, the influence of such changes should be documented and, if appropriate, a new validation should be carried out.

5.4.5.3 The range and accuracy of the values obtainable from validated methods (e.g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the customers' needs.

NOTE 1: Validation includes specification of the requirements, determination of the characteristics of the methods, a check that the requirements can be fulfilled by using the method, and a statement on the validity.

NOTE 2: As method-development proceeds, regular review should be carried out to verify that the needs of the customer are still being fulfilled. Any change in requirements requiring modifications to the development plan should be approved and authorized.

NOTE 3: Validation is always a balance between costs, risks and technical possibilities. There are many cases in which the range and uncertainty of the values (e.g. accuracy, detection limit, selectivity, linearity, repeatability, reproducibility, robustness and cross-sensitivity) can only be given in a simplified way due to lack of information.

5.4.5.4 All methods used by the laboratory, whether non-standard or standard (reference) methods shall be validated before use to ensure that the laboratory has the capability of using the method for its intended use. See Section 1.5. of each of the technical modules (Volume 1, Modules 3 through 7) for specific validation requirements. Non-standard methods must comply with 5.4.5.1 – 5.4.5.3 above, in addition to specific requirements in Section 1.5 of the technical modules.

5.4.6 Estimation of Analytical Uncertainty

Clause 5.4.6 of the *ISO/IEC/IEC 17025:2005* concerning calibration testing does not apply. The following requirement replaces the *ISO/IEC Clause*. Environmental testing laboratories shall have a procedure(s) for estimating analytical uncertainty. Quality control measurement data may be used to determine analytical uncertainty.

5.4.6.1 A calibration laboratory, or a testing laboratory performing its own calibrations, shall have and shall apply a procedure to estimate the uncertainty of measurement for all calibrations and types of calibrations.

5.4.6.2 Testing laboratories shall have and shall apply procedures for estimating uncertainty of measurement. In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases the laboratory shall at least attempt to identify all the components of uncertainty and make a reasonable estimation, and shall ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

NOTE 1: The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

- *the requirements of the test method;*
- *the requirements of the customer;*
- *the existence of narrow limits on which decisions on conformity to a specification are based.*

NOTE 2: In those cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions (see 5.10).

5.4.6.3 When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

NOTE 1: Sources contributing to the uncertainty include, but are not necessarily limited to, the reference standards and reference materials used, methods and equipment used, environmental conditions, properties and condition of the item being tested or calibrated, and the operator.

NOTE 2: The predicted long-term behaviour of the tested and/or calibrated item is not normally taken into account when estimating the measurement uncertainty.

NOTE 3: For further information, see ISO 5725 and the Guide to the Expression of Uncertainty in Measurement (see Bibliography).

5.4.7 Control of Data *(ISO/IEC 17025:2005, Clause 5.4.7)*

5.4.7.1 Calculations and data transfers shall be subject to appropriate checks in a systematic manner.

5.4.7.2 When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage or retrieval of test or calibration data, the laboratory shall ensure that:

a) computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use;

b) procedures are established and implemented for protecting the data; such procedures shall include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;

c) computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of test and calibration data.

NOTE: Commercial off-the-shelf software (e.g. word processing, database and statistical programmes) in general use within their designed application range may be considered to be sufficiently validated. However, laboratory software configuration/modifications should be validated as in 5.4.7.2 a).

5.5 Calibration Requirements *(ISO/IEC 17025:2005, Clause 5.5)*

ISO/IEC Clauses 5.5.1 to 5.5.12 apply with respect to equipment in environmental testing laboratories.

5.5.1 The laboratory shall be furnished with all items of sampling, measurement and test equipment required for the correct performance of the tests and/or calibrations (including sampling, preparation of test and/or calibration items, processing and analysis of test and/or calibration data). In those cases where the laboratory needs to use equipment outside its permanent control, it shall ensure that the requirements of this International Standard are met.

5.5.2 Equipment and its software used for testing, calibration and sampling shall be capable of achieving the accuracy required and shall comply with specifications relevant to the tests and/or calibrations concerned. Calibration programmes shall be established for key quantities or values of the

instruments where these properties have a significant effect on the results. Before being placed into service, equipment (including that used for sampling) shall be calibrated or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications. It shall be checked and/or calibrated before use (see 5.6).

5.5.3 Equipment shall be operated by authorized personnel. Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) shall be readily available for use by the appropriate laboratory personnel.

5.5.4 Each item of equipment and its software used for testing and calibration and significant to the result shall, when practicable, be uniquely identified.

5.5.5 Records shall be maintained of each item of equipment and its software significant to the tests and/or calibrations performed. The records shall include at least the following:

a) the identity of the item of equipment and its software;

b) the manufacturer's name, type identification, and serial number or other unique identification;

c) checks that equipment complies with the specification (see 5.5.2);

d) the current location, where appropriate;

e) the manufacturer's instructions, if available, or reference to their location;

f) dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;

g) the maintenance plan, where appropriate, and maintenance carried out to date;

h) any damage, malfunction, modification or repair to the equipment.

5.5.6 The laboratory shall have procedures for safe handling, transport, storage, use and planned maintenance of measuring equipment to ensure proper functioning and in order to prevent contamination or deterioration.

NOTE: Additional procedures may be necessary when measuring equipment is used outside the permanent laboratory for tests, calibrations or sampling.

5.5.7 Equipment that has been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits, shall be taken out of service. It shall be isolated to prevent its use or clearly labelled or marked as being out of service until it has been repaired and shown by calibration or test to perform correctly. The laboratory shall examine the effect of the defect or departure from specified limits on previous tests and/or calibrations and shall institute the "Control of nonconforming work" procedure (see 4.9).

5.5.8 Whenever practicable, all equipment under the control of the laboratory and requiring calibration shall be labelled, coded or otherwise identified to indicate the status of calibration, including the date when last calibrated and the date or expiration criteria when recalibration is due.

5.5.9 When, for whatever reason, equipment goes outside the direct control of the laboratory, the laboratory shall ensure that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

5.5.10 When intermediate checks are needed to maintain confidence in the calibration status of the equipment, these checks shall be carried out according to a defined procedure.

5.5.11 Where calibrations give rise to a set of correction factors, the laboratory shall have procedures to ensure that copies (e.g. in computer software) are correctly updated.

5.5.12 Test and calibration equipment, including both hardware and software, shall be safeguarded from adjustments which would invalidate the test and/or calibration results.

5.5.13 Additional Requirements and Clarifications

Calibration requirements for analytical support equipment are included in this Section while requirements for instrument (testing) calibration are included in technical modules (i.e., Asbestos, Chemistry, Microbiology, Radiochemistry, and Toxicology).

5.5.13.1 Support Equipment

This Standard applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include, but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices, and mechanical volumetric dispensing devices (such as Eppendorf® or automatic dilutor/dispensing devices).

a) The results of any calibration or verification shall be within the specifications required of the application for which this equipment is used. The laboratory shall define the specifications for acceptability if none exist in method or regulation. If any equipment fails to meet the specifications for acceptability:

   i. the equipment shall be removed from service until repaired; or

   ii. the laboratory shall maintain records of established correction factors to correct all measurements.

b) The laboratory shall maintain all support equipment in proper working order. The records of all repair and maintenance activities, including service calls, shall be kept.

c) On each day the equipment is used, balances, ovens, refrigerators, freezers, incubators, and water baths shall be checked and documented. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

d) Temperature measuring devices shall be calibrated or verified at least annually. Calibration or verification shall be performed using a recognized National Metrology Institute traceable reference, such as NIST, when available.

   i. If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable.

   ii. If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

e) If quantitative results are dependent on their accuracy, such as in standard preparation or dispensing or dilution into a specified volume, the laboratory shall verify volumetric measuring devices as follows:

   i. glass microliter syringes and Class A glassware are exempt from any verification requirements beyond what is stated in Section 4.6.2;

ii. disposable or single-use volumetric equipment shall be verified once per lot, prior to or in conjunction with its first use;

iii. mechanical devices shall be verified prior to first use and on a quarterly basis; mechanical devices used at more than one volume shall be verified at volumes bracketing the range of use, and at the mid-point of the volumes used by the device;

iv. all other volumetric support equipment shall be checked for accuracy prior to or in conjunction with its first use.

f) All other support equipment shall be calibrated or verified at least annually, using a recognized National Metrology Institute, such as NIST, traceable reference when available, bracketing the range of use.

g) Raw data records shall be retained to document equipment performance.

5.6 Measurement Traceability

5.6.1 General *(ISO/IEC 17025:2005, Clause 5.6.1)*

All equipment used for tests and/or calibrations, including equipment for subsidiary measurements (e.g. for environmental conditions) having a significant effect on the accuracy or validity of the result of the test, calibration or sampling shall be calibrated before being put into service. The laboratory shall have an established programme and procedure for the calibration of its equipment.

NOTE: Such a programme should include a system for selecting, using, calibrating, checking, controlling and maintaining measurement standards, reference materials used as measurement standards, and measuring and test equipment used to perform tests and calibrations.

5.6.2 Specific Requirements (*ISO/IEC 17025:2005, Clause 5.6.2)*

5.6.2.1 Calibration

5.6.2.1.1 For calibration laboratories, the programme for calibration of equipment shall be designed and operated so as to ensure that calibrations and measurements made by the laboratory are traceable to the International System of Units (SI) (Système international d'unités).

A calibration laboratory establishes traceability of its own measurement standards and measuring instruments to the SI by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. The link to SI units may be achieved by reference to national measurement standards. National measurement standards may be primary standards, which are primary realizations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards which are standards calibrated by another national metrology institute. When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification (see also 5.10.4.2).

NOTE 1: Calibration laboratories fulfilling the requirements of this International Standard are considered to be competent. A calibration certificate bearing an accreditation body logo from a calibration laboratory accredited to this International Standard, for the calibration concerned, is sufficient evidence of traceability of the calibration data reported.

NOTE 2: Traceability to SI units of measurement may be achieved by reference to an appropriate primary standard (see VIM:1993, 6.4) or by reference to a natural constant, the value of which in terms of the relevant SI unit is known and recommended by the General Conference of Weights and Measures (CGPM) and the International Committee for Weights and Measures (CIPM).

NOTE 3: Calibration laboratories that maintain their own primary standard or representation of SI units based on fundamental physical constants can claim traceability to the SI system only after these standards have been compared, directly or indirectly, with other similar standards of a national metrology institute.

NOTE 4: The term "identified metrological specification" means that it must be clear from the calibration certificate which specification the measurements have been compared with, by including the specification or by giving an unambiguous reference to the specification.

NOTE 5: When the terms "international standard" or "national standard" are used in connection with traceability, it is assumed that these standards fulfil the properties of primary standards for the realization of SI units.

NOTE 6: Traceability to national measurement standards does not necessarily require the use of the national metrology institute of the country in which the laboratory is located.

NOTE 7: If a calibration laboratory wishes or needs to obtain traceability from a national metrology institute other than in its own country, this laboratory should select a national metrology institute that actively participates in the activities of BIPM either directly or through regional groups.

NOTE 8: The unbroken chain of calibrations or comparisons may be achieved in several steps carried out by different laboratories that can demonstrate traceability.

5.6.2.1.2 There are certain calibrations that currently cannot be strictly made in SI units. In these cases calibration shall provide confidence in measurements by establishing traceability to appropriate measurement standards such as:

- *the use of certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material;*
- *the use of specified methods and/or consensus standards that are clearly described and agreed by all parties concerned.*

Participation in a suitable programme of interlaboratory comparisons is required where possible.

5.6.2.2 Testing

5.6.2.2.1 For testing laboratories, the requirements given in 5.6.2.1 apply for measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from the calibration contributes little to the total uncertainty of the test result. When this situation arises, the laboratory shall ensure that the equipment used can provide the uncertainty of measurement needed.

NOTE: The extent to which the requirements in 5.6.2.1 should be followed depends on the relative contribution of the calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements should be strictly followed.

5.6.2.2.2 Where traceability of measurements to SI units is not possible and/or not relevant, the same requirements for traceability to, for example, certified reference materials, agreed methods and/or consensus standards, are required as for calibration laboratories (see 5.6.2.1.2).

5.6.3 Reference Standards and Reference Materials *(ISO/IEC 17025:2005, Clause 5.6.3)*

5.6.3.1 Reference Standards

The laboratory shall have a programme and procedure for the calibration of its reference standards. Reference standards shall be calibrated by a body that can provide traceability as described in 5.6.2.1. Such reference standards of measurement held by the laboratory shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated. Reference standards shall be calibrated before and after any adjustment.

5.6.3.2 Reference Materials

Reference materials shall, where possible, be traceable to SI units of measurement, or to certified reference materials. Internal reference materials shall be checked as far as is technically and economically practicable.

5.6.3.3 Intermediate Checks

Checks needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials shall be carried out according to defined procedures and schedules.

5.6.3.4 Transport and Storage

The laboratory shall have procedures for safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity.

NOTE Additional procedures may be necessary when reference standards and reference materials are used outside the permanent laboratory for tests, calibrations or sampling.

5.6.4 Additional Requirements and Clarifications

5.6.4.1 Reference Standards and Reference Materials

The laboratory shall provide satisfactory evidence of correlation of results, for example, by participation in a suitable program of inter-laboratory comparisons, proficiency testing, or independent analysis.

a) Reference Standards

Where commercially available, this traceability shall be to a national standard of measurement.

b) Reference Materials

Where possible, traceability shall be to national or international standards of measurement or to national or international standard reference materials. Internal reference materials shall be checked as far as is technically and economically practicable.

5.6.4.2 Documentation and Labeling of Standards, Reagents, and Reference Materials

Documented procedures shall exist for the purchase, receipt and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory shall retain records for all standards, reagents, reference materials, and media, including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if available), the date of receipt, and recommended storage conditions.

b) For original containers, if an expiration date is provided by the manufacturer or vendor, it shall be recorded on the container. If an expiration date is not provided by the manufacturer or vendor, it is not required.

c) Records shall be maintained on standard, reference material, and reagent preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

d) All containers of prepared standards, reference materials, and reagents shall bear a unique identifier and expiration date.

e) Procedures shall be in place to ensure prepared reagents meet the requirements of the method.

f) Standards, reference materials, and reagents shall not be used after their expiration dates unless their reliability is verified by the laboratory.

5.7 Collection of Samples *(ISO/IEC 17025:2005, Clause 5.7)*

5.7.1 The laboratory shall have a sampling plan and procedures for sampling when it carries out sampling of substances, materials or products for subsequent testing or calibration. The sampling plan as well as the sampling procedure shall be available at the location where sampling is undertaken. Sampling plans shall, whenever reasonable, be based on appropriate statistical methods. The sampling process shall address the factors to be controlled to ensure the validity of the test and calibration results.

NOTE 1: Sampling is a defined procedure whereby a part of a substance, material or product is taken to provide for testing or calibration of a representative sample of the whole. Sampling may also be required by the appropriate specification for which the substance, material or product is to be tested or calibrated. In certain cases (e.g. forensic analysis), the sample may not be representative but is determined by availability.

NOTE 2: Sampling procedures should describe the selection, sampling plan, withdrawal and preparation of a sample or samples from a substance, material or product to yield the required information.

5.7.2 Where the customer requires deviations, additions or exclusions from the documented sampling procedure, these shall be recorded in detail with the appropriate sampling data and shall be included in all documents containing test and/or calibration results, and shall be communicated to the appropriate personnel.

5.7.3 The laboratory shall have procedures for recording relevant data and operations relating to sampling that forms part of the testing or calibration that is undertaken. These records shall include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and diagrams or other equivalent means to identify the sampling location as necessary and, if appropriate, the statistics the sampling procedures are based upon.

5.7.4 Additional Requirements

a) Documentation shall include the date and time of sampling.

b) Any deviations from sampling procedures shall be documented.

5.8 Handling Samples and Test Items *(ISO/IEC 17025:2005, Clause 5.8)*

5.8.1 The laboratory shall have procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of test and/or calibration items, including all provisions necessary to protect the integrity of the test or calibration item, and to protect the interests of the laboratory and the customer.

5.8.2 The laboratory shall have a system for identifying test and/or calibration items. The identification shall be retained throughout the life of the item in the laboratory. The system shall be designed and operated so as to ensure that items cannot be confused physically or when referred to in records or other documents. The system shall, if appropriate, accommodate a sub-division of groups of items and the transfer of items within and from the laboratory.

5.8.3 Upon receipt of the test or calibration item, abnormalities or departures from normal or specified conditions, as described in the test or calibration method, shall be recorded. When there is doubt as to the suitability of an item for test or calibration, or when an item does not conform to the description provided, or the test or calibration required is not specified in sufficient detail, the laboratory shall consult the customer for further instructions before proceeding and shall record the discussion.

5.8.4 The laboratory shall have procedures and appropriate facilities for avoiding deterioration, loss or damage to the test or calibration item during storage, handling and preparation. Handling instructions provided with the item shall be followed. When items have to be stored or conditioned under specified environmental conditions, these conditions shall be maintained, monitored and recorded. Where a test or calibration item or a portion of an item is to be held secure, the laboratory shall have arrangements for storage and security that protect the condition and integrity of the secured items or portions concerned.

NOTE 1: Where test items are to be returned into service after testing, special care is required to ensure that they are not damaged or injured during the handling, testing or storing/waiting processes.

NOTE 2: A sampling procedure and information on storage and transport of samples, including information on sampling factors influencing the test or calibration result, should be provided to those responsible for taking and transporting the samples.

NOTE 3: Reasons for keeping a test or calibration item secure can be for reasons of record, safety or value, or to enable complementary tests and/or calibrations to be performed later.

5.8.5 Additional Requirements – Documentation

The following are essential to ensure the validity of the laboratory's data.

a) The laboratory shall have a documented system for uniquely identifying the sample containers that hold samples to be tested, to ensure that there can be no confusion regarding the identity of such samples at any time. This system shall include identification for all samples, sub-samples, preservations, sample containers, tests, and subsequent extracts and/or digestates.

b) This laboratory code shall maintain an unequivocal link with the unique field ID code assigned to each sample.

c) The laboratory ID code shall be placed as a durable mark on the sample container.

d) The laboratory ID code shall be entered into the laboratory records and shall be the link that associates the sample with related laboratory activities such as sample preparation.

e) In cases where the sample collector and analyst are the same individual, or the laboratory pre-assigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

5.8.6 Additional Requirements – Sample Acceptance Policy

The laboratory shall have a written sample acceptance policy that includes the following:

a) proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

b) proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) use of appropriate sample containers;

d) adherence to specified holding times;

e) sufficient sample volume to perform the necessary tests;

f) procedures to be used when samples show signs of damage, contamination or inadequate preservation; and

g) qualification of any data that do not meet the above requirements.

5.8.7 Additional Requirements – Sample Receipt Protocols

5.8.7.1 The laboratory shall implement procedures for verifying and documenting preservation.

5.8.7.2 If the sample does not meet the sample receipt acceptance criteria listed in this Standard, the laboratory shall either:

a) retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

b) fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

i. The condition of these samples shall be noted on the chain of custody or transmittal form and laboratory receipt documents.

ii. The analysis data shall be appropriately qualified on the final report.

5.8.7.3 The laboratory shall utilize a permanent chronological record such as a logbook or electronic database to document receipt of all sample containers.

a) This sample receipt log shall record the following:

i. client/project name;

ii. date and time of laboratory receipt;

iii. unique laboratory ID code (see Section 5.8.5 a); and

iv. signature or initials of the person making the entries.

b) During the login process, the following information shall be unequivocally linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request and readily available to individuals who will process the sample. The placement of the laboratory ID number on the sample container is not considered a permanent record.

i. The field ID code, which identifies each sample, shall be linked to the laboratory ID code in the sample receipt log.

ii. The date and time of sample collection shall be linked to the sample and to the date and time of receipt in the laboratory.

iii. The requested analyses (including applicable approved method numbers) shall be linked to the laboratory ID code.

iv. Any comments resulting from inspection for sample rejection shall be linked to the laboratory ID code.

5.8.7.4 All documentation, such as memos, chain of custody, or transmittal forms that are transmitted to the laboratory by the sample transmitter, shall be retained.

5.8.7.5 A complete chain of custody record form, if utilized, shall be maintained.

5.8.8 Additional Requirements – Legal Chain of Custody Protocols

Legal chain of custody procedures are used for evidentiary or legal purposes. If a client specifies that a sample is to be used for evidentiary purposes, then a laboratory shall have a written SOP for how that laboratory will carry out legal chain of custody.

5.8.9 Additional Requirements – Sample Storage and Disposal

a) Samples shall be stored according to the conditions specified by preservation protocols.

i. Samples that require thermal preservation shall be stored under refrigeration that is +/-2°C of the specified preservation temperature unless regulatory or method specific criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable.

ii. Samples shall be stored away from all standards, reagents, and food. Samples shall be stored in such a manner to prevent cross contamination.

b) Sample fractions, extracts, leachates and other sample preparation products shall be stored according to Section 5.8.9 a) above or according to specifications in the method.

c) The laboratory shall have SOPs for the disposal of samples, digestates, leachates and extracts or other sample preparation products.

5.9 Quality Assurance for Environmental Testing *(ISO/IEC 17025:2005, Clause 5.9)*

5.9.1 The laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

a) regular use of certified reference materials and/or internal quality control using secondary reference materials;

b) participation in interlaboratory comparison or proficiency-testing programmes;

c) replicate tests or calibrations using the same or different methods;

d) retesting or recalibration of retained items;

e) correlation of results for different characteristics of an item.

NOTE: The selected methods should be appropriate for the type and volume of the work undertaken.

5.9.2 Quality control data shall be analysed and, where they are found to be outside pre-defined criteria, planned action shall be taken to correct the problem and to prevent incorrect results from being reported.

5.9.3 Essential Quality Control Procedures

These general QC principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., asbestos, chemical, microbiological, radiological, toxicity) and are further described in Technical Modules. The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have detailed written protocols in place to monitor the following quality controls:

i. positive and negative controls (see technical modules), chemical or microbiological as applicable to the test type, to monitor tests such as blanks, matrix spikes, reference toxicants;

ii. tests to define the variability and/or repeatability of the laboratory results such as replicates;

iii. measures to assure the accuracy of the method including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

iv. measures to evaluate method capability, such as limit of detection and limit of quantitation or range of applicability such as linearity;

v. selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;

vi. selection and use of reagents and standards of appropriate quality;

vii. measures to assure the selectivity of the test for its intended purpose; and

viii. measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the method such as temperature, humidity, light or specific instrument conditions.

b) All QC measures shall be assessed and evaluated on an on-going basis and QC acceptance criteria shall be used.

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist.

The QC protocols specified by the laboratory's SOP shall be followed (see Section 4.2.8.5 in this Standard). The laboratory shall ensure that the essential standards outlined in Technical Modules or mandated methods or regulations (whichever are more stringent) are incorporated into their method manuals. When it is not apparent which is more stringent, the QC in the mandated method or regulations is to be followed.

5.10 Reporting the Results

All references to Calibration Certificates in *ISO/IEC 17025:2005* are not applicable to environmental testing.

5.10.1 General *(ISO/IEC 17025:2005, Clause 5.10.1)*

The results of each test, calibration, or series of tests or calibrations carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test or calibration methods.

The results shall be reported, usually in a test report or a calibration certificate (see Note 1), and shall include all the information requested by the customer and necessary for the interpretation of the test or calibration results and all information required by the method used. This information is normally that required by 5.10.2, and 5.10.3 or 5.10.4.

In the case of tests or calibrations performed for internal customers, or in the case of a written agreement with the customer, the results may be reported in a simplified way. Any information listed in 5.10.2 to 5.10.4 which is not reported to the customer shall be readily available in the laboratory which carried out the tests and/or calibrations.

NOTE 1: Test reports and calibration certificates are sometimes called test certificates and calibration reports respectively.

NOTE 2: The test reports or calibration certificates may be issued as hard copy or by electronic data transfer provided that the requirements of this International Standard are met.

5.10.2 Test Reports and Calibration Certificates *(ISO/IEC 17025:2005, Clause 5.10.2)*

Each test report or calibration certificate shall include at least the following information, unless the laboratory has valid reasons for not doing so:

a) a title (e.g. "Test Report" or "Calibration Certificate");

b) the name and address of the laboratory, and the location where the tests and/or calibrations were carried out, if different from the address of the laboratory;

c) unique identification of the test report or calibration certificate (such as the serial number), and on each page an identification in order to ensure that the page is recognized as a part of

the test report or calibration certificate, and a clear identification of the end of the test report or calibration certificate;

d) the name and address of the customer;

e) identification of the method used;

f) a description of, the condition of, and unambiguous identification of the item(s) tested or calibrated;

g) the date of receipt of the test or calibration item(s) where this is critical to the validity and application of the results, and the date(s) of performance of the test or calibration;

h) reference to the sampling plan and procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results;

i) the test or calibration results with, where appropriate, the units of measurement;

j) the name(s), function(s) and signature(s) or equivalent identification of person(s) authorizing the test report or calibration certificate;

k) where relevant, a statement to the effect that the results relate only to the items tested or calibrated.

NOTE 1: Hard copies of test reports and calibration certificates should also include the page number and total number of pages.

NOTE 2: It is recommended that laboratories include a statement specifying that the test report or calibration certificate shall not be reproduced except in full, without written approval of the laboratory.

5.10.3 Test Reports *(ISO/IEC 17025:2005, Clause 5.10.3)*

5.10.3.1 In addition to the requirements listed in 5.10.2, test reports shall, where necessary for the interpretation of the test results, include the following:

a) deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions;

b) where relevant, a statement of compliance/non-compliance with requirements and/or specifications;

c) where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit;

d) where appropriate and needed, opinions and interpretations (see 5.10.5);

e) additional information which may be required by specific methods, customers or groups of customers.

5.10.3.2 In addition to the requirements listed in 5.10.2 and 5.10.3.1, test reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

a) the date of sampling;

b) unambiguous identification of the substance, material or product sampled (including the name of the manufacturer, the model or type of designation and serial numbers as appropriate);

c) the location of sampling, including any diagrams, sketches or photographs;

d) a reference to the sampling plan and procedures used;

e) details of any environmental conditions during sampling that may affect the interpretation of the test results;

f) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

5.10.4 Calibration Certificates *(ISO/IEC 17025:2005, Clause 5.10.4)* does not apply to environmental testing activities.

5.10.4.1 In addition to the requirements listed in 5.10.2, calibration certificates shall include the following, where necessary for the interpretation of calibration results:

a) the conditions (e.g. environmental) under which the calibrations were made that have an influence on the measurement results;

b) the uncertainty of measurement and/or a statement of compliance with an identified metrological specification or clauses thereof;

c) evidence that the measurements are traceable (see Note 2 in 5.6.2.1.1).

5.10.4.2 The calibration certificate shall relate only to quantities and the results of functional tests. If a statement of compliance with a specification is made, this shall identify which clauses of the specification are met or not met. When a statement of compliance with a specification is made omitting the measurement results and associated uncertainties, the laboratory shall record those results and maintain them for possible future reference. When statements of compliance are made, the uncertainty of measurement shall be taken into account.

5.10.4.3 When an instrument for calibration has been adjusted or repaired, the calibration results before and after adjustment or repair, if available, shall be reported.

5.10.4.4 A calibration certificate (or calibration label) shall not contain any recommendation on the calibration interval except where this has been agreed with the customer. This requirement may be superseded by legal regulations.

5.10.5 Opinions and interpretations

When opinions and interpretations are included, the laboratory shall document the basis upon which the opinions and interpretations have been made. Opinions and interpretations shall be clearly marked as such in a test report.

NOTE 1: Opinions and interpretations should not be confused with inspections and product certifications as intended in ISO/IEC 17020 and ISO/IEC Guide 65.

NOTE 2: Opinions and interpretations included in a test report may comprise, but not be limited to, the following:

- *an opinion on the statement of compliance/noncompliance of the results with requirements;*
- *fulfilment of contractual requirements;*
- *recommendations on how to use the results;*
- *guidance to be used for improvements.*

NOTE 3: In many cases it might be appropriate to communicate the opinions and interpretations by direct dialogue with the customer. Such dialogue should be written down.

5.10.6 Testing and calibration results obtained from subcontractors

When the test report contains results of tests performed by subcontractors, these results shall be clearly identified. The subcontractor shall report the results in writing or electronically. When a calibration has been subcontracted, the laboratory performing the work shall issue the calibration certificate to the contracting laboratory.

5.10.7 Electronic transmission of results

In the case of transmission of test or calibration results by telephone, telex, facsimile or other electronic or electromagnetic means, the requirements of this International Standard shall be met (see also 5.4.7).

5.10.8 Format of reports and certificates

The format shall be designed to accommodate each type of test or calibration carried out and to minimize the possibility of misunderstanding or misuse.

NOTE 1: Attention should be given to the lay-out of the test report or calibration certificate, especially with regard to the presentation of the test or calibration data and ease of assimilation by the reader.

NOTE 2: The headings should be standardized as far as possible.

5.10.9 Amendments to test reports and calibration certificates

Material amendments to a test report or calibration certificate after issue shall be made only in the form of a further document, or data transfer, which includes the statement: "Supplement to Test Report [or Calibration Certificate], serial number... [or as otherwise identified]", or an equivalent form of wording. Such amendments shall meet all the requirements of this International Standard. When it is necessary to issue a complete new test report or calibration certificate, this shall be uniquely identified and shall contain a reference to the original that it replaces.

5.10.10 Exceptions

Some regulatory reporting requirements or formats, such as monthly operating reports, may not require all items listed in 5.10.2 and 5.10.3 above; however, the laboratory shall provide all the required information to their client for use in preparing such regulatory reports.

Laboratories operated solely to provide data for compliance purposes (in-house or captive laboratories) shall have all applicable information specified in Section 5.10 readily available for review by the accreditation body. However, formal reports detailing the information are not required if:

a) the in-house laboratory is itself responsible for preparing the regulatory reports; or

b) the laboratory provides information to another individual within the organization for preparation of regulatory reports. The facility management shall ensure that the appropriate report items are in the report to the regulatory authority, if such information is required; or

c) see Section 5.10.1, paragraph 3.

5.10.11 Additional Requirements

a) Time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to seventy-two (72) hours.

b) Results that are reported on a basis other than as received (e. g., dry weight).

c) Any non-accredited tests shall be clearly identified as such to the client when claims of accreditation to this Standard are made in the analytical report or in the supporting electronic or hardcopy deliverables.

d) Clear identification of numerical results with values outside the calibration range.

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[24] Information and documents on laboratory accreditation can be found on the ILAC (International Laboratory Accreditation Cooperation): www.ilac.org

1) To be published. (Revision of ISO 9000:2000)

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ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 3: Quality Systems for Asbestos 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. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

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

Standard Revision History

Action	Date
Working Draft Standard Published	June 26, 2011
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1.0 Asbestos Testing

1.1 Introduction

This Standard applies to laboratories undertaking the examination of asbestos samples. This Standard is organized by analytical technique including transmission electron microscopy (TEM) for the analysis of water, wastewater, air, and bulk samples; phase contrast microscopy (PCM) for analysis of workplace air; and polarized light microscopy (PLM) for analysis of bulk samples. These procedures for asbestos analysis involve sample preparation followed by detection of asbestos.

1.2 Scope

The essential quality control (QC) procedures applicable to asbestos measurements are included in this document. Additional QC 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 are the preferred references. 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

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

The inclusion of the analyte in the method shall meet all required calibration requirements of the method and the QC requirements of the method to which the analyte is being added. If no QC exists in the method, the laboratory shall adhere to the requirements outlined in a similar reference method (when available). A method that meets these requirements shall be identified in such a way so that there is no confusion that the method has been modified.

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

Prior to acceptance and institution of any method for which data will be reported, all methods shall be validated. For both reference and non-standard methods, laboratories shall participate in proficiency testing programs. The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data. Non-standard methods must comply with the requirements in Volume 1, Module 2, Section 5.4.5.

1.6 Demonstration of Capability (DOC)

1.6.1 General

a) An individual who performs any activity involved with preparation and/or analysis of samples must have constant, close supervision as defined in the laboratory's training procedure until a satisfactory initial DOC is completed (see Section 1.6.2).

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

c) In cases where an individual has prepared and/or analyzed samples using a method that has been in use by the laboratory for at least one (1) year prior to applying for accreditation, and there have been no significant changes in instrument type or method, the on-going 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 shall be documented. All data applicable to the demonstration shall be retained and readily available at the laboratory.

1.6.2 Initial DOC

An individual must successfully perform an initial DOC prior to using any method (see 1.6.1.a) above), and at any time there is a change in instrument type or method, or any time that a method has not been performed by the 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; and

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

1.6.2.2 For asbestos, if the method or regulation does not specify a DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to 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.

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 analyte 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 analytes meet the acceptance criteria, the analysis of actual samples may begin. If any one of the analytes does not meet the acceptance criteria, the performance is unacceptable for that analyte.

e) When one or more of the tested analytes 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 analytes of interest beginning with c) above.

   ii. Beginning with c) above, repeat the test for all analytes 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).

1.6.3 On-Going 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 on-going capability by routinely meeting the QC requirements of the method, laboratory SOP, client specifications, and/or this Standard. 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 For asbestos, this ongoing DOC may be one of the following:

a) acceptable performance of a blind sample (single blind to the analyst) or successful analysis of a blind performance sample on a similar method using the same technology (e.g., EPA Methods 100.1 and 100.2);

b) another initial DOC;

c) at least four (4) consecutive laboratory control samples (LCS) 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 or reference sample(s) for each method for each analyst each year;

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

Refer to methods referenced in the following Sections for specific equipment requirements. If NIST standard reference materials (SRM) specified below are unavailable, the laboratory may substitute an equivalent reference material with a certificate of analysis.

1.7.1.1 Transmission Electron Microscopy

Refer to methods referenced in the following sections for specific equipment requirements.

1.7.1.1.1 Water and Wastewater

All calibrations listed below (unless otherwise noted) shall be performed under the same analytical conditions used for routine asbestos analysis and shall be recorded in a notebook and include date and analyst's signature. Frequencies stated below may be reduced to "before next use" if no samples are analyzed after the last calibration period has expired. Likewise, frequencies may have to be increased following non-routine maintenance or unacceptable calibration performance.

a) Magnification Calibration. Magnification calibration shall be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000 and 20,000x. A logbook shall be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification. Calibration data shall be displayed on control charts that show trends over time.

b) Camera Constant. The camera length of the TEM in the Selected Area Electron Diffraction (SAED) mode shall be calibrated before SAED patterns of unknown samples are observed. The diffraction specimen shall be at the eucentric position for this calibration. This calibration shall allow accurate (<10% variation) measurement of layer-line spacings on the medium used for routine measurement, i.e., the phosphor screen or camera film. This shall also allow accurate (<5% variation) measurement of zone axis SAED patterns on permanent media (e.g., film). Calibrations shall be performed monthly to establish the stability of the camera constant. Where non-asbestiform minerals may be expected (e.g., winchite, richterite, industrial talc, vermiculite, etc.), an internal camera constant standard such as gold, shall be deposited and measured on each sample to facilitate accurate indexing of zone axis SAED patterns. In such cases, layer line analysis alone shall not be used. Calibration data shall be displayed on control charts that show trends over time.

c) Spot Size. The diameter of the smallest beam spot at crossover shall be less than 250nm as calibrated quarterly. Calibration data shall be displayed on control charts that show trends over time.

d) Beam Dose. The beam dose shall be calibrated so that beam damage to chrysotile is minimized, specifically so that an electron diffraction pattern from a single fibril >1µm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least fifteen (15) seconds.

e) Energy Dispersive X-Ray Analysis (EDXA) System

i. The x-ray energy vs. channel number for the EDXA system shall be calibrated to within 20 eV for at least two peaks between 0.7 keV and 10 keV. One peak shall be from the low end (0.7 keV to 2 keV) and the other peak from the high end (7 keV to 10 keV) of this range. The calibration of the x-ray energy shall be checked prior to each analysis of samples and recalibrated if out of the specified range.

ii. The ability of the system to resolve the Na Kα line from the Cu L line shall be confirmed quarterly by obtaining a spectrum from the NIST SRM 1866 crocidolite sample on a copper grid.

iii. The k-factors for elements found in asbestos (Na, Mg, Al, Si, Ca, and Fe) relative to Si shall be calibrated semiannually, or anytime the detector geometry may be altered. NIST SRM 2063a shall be used for Mg, Si, Ca, Fe, while k-factors for Na and Al may be obtained from suitable materials such as albite, kaersutite, or NIST SRM 99a. The k-factors shall be determined to a precision (2s) within 10% relative to the mean value obtained for Mg, Al, Si, Ca, and Fe, and within 20% relative to the mean value obtained for Na. The k-factor relative to Si for Na shall be between 1.0 and 4.0, for Mg and Fe shall be between 1.0 and 2.0, and for Al and Ca shall be between 1.0 and 1.75. The k-factor for Mg relative to Fe shall be 1.5 or less. Calibration data shall be displayed on control charts that show trends over time.

iv. The detector resolution shall be checked quarterly to ensure a full-width half maximum resolution of <175 eV at Mn Kα (5.90 keV). Calibration data shall be displayed on control charts that show trends over time.

v. The portions of a grid in a specimen holder for which abnormal x-ray spectra are generated under routine asbestos analysis conditions shall be determined and these areas shall be avoided in asbestos analysis.

vi. The sensitivity of the detector for collecting x-rays from small volumes shall be documented quarterly by collecting resolvable Mg and Si peaks from a unit fibril of NIST SRM 1866 chrysotile.

f) Low Temperature Asher. The low temperature asher shall be calibrated quarterly by determining a calibration curve for the weight vs. ashing time of collapsed mixed-celluloseester (MCE) filters. Calibration data shall be displayed on control charts that show trends over time.

g) Grid Openings. The magnification of the grid opening measurement system shall be calibrated using an appropriate standard at a frequency of 20 openings/20 grids/lot of 1000 or 1 opening/sample. The variation in the calibration measurements (2s) is <5% of the mean calibration value.

1.7.1.1.2 Air

All calibrations shall be performed in accordance with Section 1.7.1.1.1, with the exception of magnification. Magnification calibration shall be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 15,000 to 20,000x. A logbook shall be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

1.7.1.1.3 Bulk Samples

All calibrations shall be performed in accordance with Section 1.7.1.1.1.

1.7.1.2 Phase Contrast Microscopy

1.7.1.2.1 At least once daily, the analyst shall use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.

1.7.1.2.2 The phase-shift detection limit of the microscope shall be checked monthly or after modification or relocation using an HSE/NPL phase-contrast test slide for each analyst/microscope combination. This procedure assures that the minimum detectable fiber diameter (<ca. 0.25 µm) for this microscope is achieved.

1.7.1.2.3 Prior to ordering the Walton-Beckett graticule, calibration, in accordance with NIOSH 7400, Issue 2, 15 August 1994, Appendix A, shall be performed to obtain a counting area 100 µm in diameter at the image plane. The diameter, dc (mm), of the circular counting area and the disc diameter shall be specified when ordering the graticule. The field diameter (D) shall be verified (or checked), to a tolerance of 100 µm ± 2 µm, with a stage micrometer upon receipt of the graticule from the manufacturer. When changes (zoom adjustment, disassembly, replacement, etc.) occur in the eyepiece-objective-reticle combination, field diameter shall be re-measured (or recalibrated) to determine field area (mm2). Recalibration of field diameter shall also be required when there is a change in interpupillary distance (i.e., change in analyst). Acceptable range for field area shall be 0.00754 mm2 to 0.00817 mm2. The actual field area shall be documented and used.

1.7.1.3 Polarized Light Microscopy

1.7.1.3.1 Microscope Alignment. To accurately measure the required optical properties, a properly aligned polarized light microscope (PLM) shall be utilized. The PLM shall be aligned before each use.

1.7.1.3.2 Refractive Index Liquids. Series of n_D = 1.49 through 1.72 in intervals less than or equal to 0.005. Refractive index liquids for dispersion staining, high-dispersion series 1.550, 1.605, 1.680. The accurate measurement of the refractive index (RI) of a substance requires the use of calibrated refractive index liquids. These liquids shall be calibrated at first use and semiannually, or next use, whichever is less frequent, to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or RI glass beads.

1.7.2 Quality Control

1.7.2.1 Negative Controls

1.7.2.1.1 Transmission Electron Microscopy

a) Water and Wastewater

i. Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one (1) bottle from each batch, or a minimum of one (1) from each twenty-four (24), shall be tested for background level. When using glass bottles, four (4) bottles from each twenty-four (24) shall be tested. An acceptable bottle blank level is defined as < 0.01 Million Fibers per Liter (MFL) > 10 µm.

ii. A process blank sample consisting of fiber-free water shall be run before the first field sample. The quantity of water shall be > 10 mL for a 25-mm diameter filter and > 50 mL for a 47-mm diameter filter.

b) Air

i. A blank filter shall be prepared with each set of samples. A blank filter shall be left uncovered during preparation of the sample set and a wedge from that blank filter

shall be prepared alongside wedges from the sample filters. At minimum, the blank filter shall be analyzed for each twenty (20) samples analyzed.

ii. Maximum contamination on a single blank filter shall be no more than 53 structures/mm2. Maximum average contamination for all blank filters shall be no more than 18 structures/mm2.

c) Bulk Samples

i. Contamination checks using asbestos-free material, such as the glass fiber blank in SRM 1866, shall be performed at a frequency of one for every twenty samples analyzed. The detection of asbestos at a concentration exceeding 0.1% will require an investigation to detect and remove the source of the asbestos contamination.

ii. The laboratory shall maintain a list of non-asbestos fibers that can be confused with asbestos. The list shall include crystallographic and/or chemical properties that disqualify each fiber being identified as asbestos.

iii. The laboratory shall have a set of reference asbestos materials, from which a set of reference diffraction and x-ray spectra may be developed.

1.7.2.1.2 Phase Contrast Microscopy

At least two (2) field blanks (or 10% of the total samples, whichever is greater) shall be submitted for analysis with each set of samples. Field blanks shall be handled in a manner representative of actual handling of associated samples in the set with a single exception that air shall not be drawn through the blank sample. A blank cassette shall be opened for approximately thirty (30) seconds at the same time other cassettes are opened just prior to analysis. Results from field blank samples shall be used in the calculation to determine final airborne fiber concentration. The identity of blank filters shall be unknown to the counter until all counts have been completed. If a field blank yields greater than seven (7) fibers per one hundred (100) graticule fields, report possible contamination of the samples.

1.7.2.1.3 Polarized Light Microscopy

a) Friable Materials. At least one (1) blank slide shall be prepared daily or with every fifty (50) samples analyzed, whichever is less. This is prepared by mounting a sub-sample of an isotropic verified non-asbestos-containing material (non-ACM) (e.g., fiberglass in SRM 1866) in a drop of immersion oils normally used on a clean slide, rubbing preparation tools (forceps, dissecting needles, etc.) in the mount and placing a clean coverslip on the drop. The entire area under the coverslip shall be scanned to detect any asbestos contamination. A similar check shall be made after every twenty (20) uses of each piece of homogenization equipment. An isotropic verified non-ACM shall be homogenized in the clean equipment, a slide prepared with the material and the slide scanned for asbestos contamination. (This can be substituted for the blank slide mentioned in this Section.)

b) Non-Friable Materials. At least one (1) non-ACM non-friable material shall be prepared and analyzed with every twenty (20) samples analyzed. This non-ACM shall go through the full preparation and analysis regimen for the type of analysis being performed.

1.7.3 Test Variability/Reproducibility

1.7.3.1 Transmission Electron Microscopy

Quality assurance (QA) analyses shall be performed regularly covering all time periods, instruments, tasks, and personnel. The selection of samples shall be random and samples of special interest may be included in the selection of samples for QA analyses. When possible, the

checks on personnel performance shall be executed without their prior knowledge. A disproportionate number of analyses shall not be performed prior to internal or external audits. It is recommended that a laboratory initially be at 100% QC (all samples re-analyzed). The proportion of QC samples can later be lowered gradually, as control indicates, to a minimum of 10%.

1.7.3.1.1 Water and Wastewater

All analyses shall be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings. Quality assurance analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks shall be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from inter-laboratory proficiency testing. Relative standard deviations (RSD) for amphibole asbestos decreased from 50% at 0.8 MFL to 25% at 7 MFL in inter-laboratory proficiency testing, while RSD for chrysotile was higher, 50% at 6 MFL.

a) Replicate. A second, independent analysis shall be performed on the same grids but on different grid openings than used in the original analysis of a sample. Results shall be within 1.5x of Poisson standard deviation. This shall be performed at a frequency of one (1) per one hundred (100) samples.

b) Duplicate. A second aliquot of sample shall be filtered through a second filter, prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0x of Poisson standard deviation. This shall be performed at a frequency of one (1) per one hundred (100) samples.

c) Verified Analyses. A second, independent analysis shall be performed on the same grids and grid openings used in the original analysis of a sample. The two sets of results shall be compared according to Turner and Steel (NISTIR 5351). This shall be performed at a frequency of one (1) per twenty (20) samples. Qualified analysts shall maintain an average of ≥ 80% true positives, ≤ 20% false negatives, and ≤ 10% false positives.

1.7.3.1.2 Air

a) All analyses shall be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings.

b) The laboratory and TEM analysts shall obtain mean analytical results on NIST SRM 1876b so that trimmed mean values fall within 80% of the lower limit and 110% of the upper limit of the 95% confidence limits as published on the certificate. These limits are derived from the allowable false positives and false negatives given in Section 1.7.3.1.1.c, Verified Analysis, below. SRM 1876b shall be analyzed a minimum of once per year by each TEM analyst.

c) The laboratory shall have documentation demonstrating that TEM analysts correctly classify at least 90% of both bundles and single fibrils of asbestos structures greater than or equal to 1 µm in length in known standard materials traceable to NIST, such as NIST bulk asbestos SRM 1866.

d) Inter-laboratory analyses shall be performed to detect laboratory bias. The frequency of inter-laboratory verified analysis shall correspond to a minimum of one (1) per two hundred (200) grid square analyses for clients.

e) If more than one TEM is used for asbestos analysis, intermicroscope analyses shall be performed to detect instrument bias.

i. Replicate. A second, independent analysis shall be performed in accordance with Section 1.7.3.1.1.a.

ii. Duplicate. A second wedge from a sample filter shall be prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0x of Poisson standard deviation. This shall be performed at a frequency of one (1) per one hundred (100) samples.

iii. Verified Analyses. A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section 1.7.3.1.1.c.

1.7.3.1.3 Bulk Samples

Bulk samples with low (< 10%) asbestos content are the most problematic. At least 30% of a laboratory's QC analyses shall be performed on samples containing from 1% to 10% asbestos.

a) Intra-Analyst Precision. At least one (1) out of fifty (50) samples shall be re-analyzed by the same analyst. For single analyst laboratories, at least one (1) out of every ten (10) samples shall be re-analyzed by the same analyst.

b) Inter-Analyst Precision. At least one (1) out of fifteen (15) samples shall be re-analyzed by another analyst. Inter-analyst results will require additional re-analysis, possibly including another analyst, to resolve discrepancies when classification (ACM vs. non-ACM) errors occur, when asbestos identification errors occur, or when inter-analyst precision is found to be unacceptable.

c) Inter-Laboratory Precision. The laboratory shall participate in round robin testing with at least one (1) other laboratory. Samples shall be sent to this other laboratory at least four (4) times per year. These samples shall be samples previously analyzed as QC samples. Results of these analyses shall be assessed in accordance with QC requirements. The QC requirements shall address misclassifications (false positives, false negatives) and misidentification of asbestos types.

1.7.3.2 Phase Contrast Microscopy

a) Inter-Laboratory Precision. Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory quality assurance program that includes participation of at least two (2) other independent laboratories. Each laboratory shall participate in round robin testing at least once every six (6) months with at least all the other laboratories in its inter-laboratory quality assurance group. Each laboratory shall submit slides typical of its own workload for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology. Results of this QA program shall be posted in each laboratory to keep the microscopists informed.

b) Intra- and Inter-Analyst Precision. Each analyst shall select and count a prepared slide from a "reference slide library" on each day on which air counts are performed. Reference slides shall be prepared using well-behaved samples taken from the laboratory workload. Fiber densities shall cover the entire range routinely analyzed by the laboratory. These slides shall be counted by all analysts to establish an original standard deviation and corresponding limits of acceptability. Results from the daily reference sample analysis shall be compared to the statistically derived acceptance limits using a control chart or a database. It is recommended that the labels on the reference slides be periodically changed so that the analysts do not become familiar with the samples. Intra- and inter-analyst precision may be estimated from blind recounts on reference samples. Inter-analyst precision shall be posted in each laboratory to keep the microscopists informed.

1.7.3.3 Polarized Light Microscopy

Refer to Section 1.7.3.1.3

1.7.4 Other Quality Control Measures

1.7.4.1 Transmission Electron Microscopy

a) Water and Wastewater

i. Filter preparations shall be made from all six (6) asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between one (1) and twenty (20) structures (>10µm) per 0.01 mm^2. One of these preparations shall be analyzed independently at a frequency of one (1) per one hundred (100) samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with S. Turner and E.B. Steel, NISTIR 5351, Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0, 1994.

ii. NIST SRM 1876b shall be analyzed annually by each analyst. Results shall be evaluated in accordance with limits published for that SRM.

b) Air

i. Filter preparations shall be made from all six (6) asbestos types in accordance with Section 1.7.4.1 a) i.

ii. NIST SRM 1876b shall be analyzed annually.

c) Bulk Samples

All analysts shall be able to correctly identify the six (6) regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite). Standards for the six (6) asbestos types listed are available from NIST (SRMs 1866 and 1867).

1.7.4.2 Phase Contrast Microscopy

a) Test for Non-Random Fiber Distribution. Blind recounts by the same analyst shall be performed on 10% of the filters counted. A person other than the counter shall re-label slides before the second count. A test for type II error shall be performed to determine whether a pair of counts by the same analyst on the same slide shall be rejected due to non-random fiber distribution. If a pair of counts is rejected by this test, the remaining samples in the set shall be recounted and the new counts shall be tested against first counts. All rejected paired counts shall be discarded.

b) It shall not be necessary to use this statistic on blank recounts.

c) All laboratories shall participate in a national sample testing scheme such as the Proficiency Analytical Testing (PAT) program or the Asbestos Analysts Registry (AAR) program, both sponsored by the American Industrial Hygiene Association (AIHA).

1.7.4.3 Polarized Light Microscopy

a) Friable Materials. Because accuracy cannot be determined by re-analysis of routine field samples, at least one (1) out of one hundred (100) samples shall be a standard or reference sample that has been routinely resubmitted to determine analyst's precision and accuracy. A set of these samples may be accumulated from proficiency testing samples with predetermined weight compositions or from standards generated with weighed quantities of asbestos and other bulk materials. At least half of the reference samples submitted for this QC shall contain between 1 and 10% asbestos.

b) Non-Friable Materials. At least one (1) out of one hundred (100) samples shall be a verified quantitative standard that has routinely been resubmitted to determine analyst precision and accuracy.

1.7.5 Analytical Sensitivity

1.7.5.1 Transmission Electron Microscopy

1.7.5.1.1 Water and Wastewater

An analytical sensitivity of 200,000 fibers per liter (0.2 MFL) is required for each sample analyzed. Analytical sensitivity is defined as the waterborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the fraction of the filter sampled and the dilution factor (if applicable).

1.7.5.1.2 Air

An analytical sensitivity of 0.005 structures/cm^2 is required for each sample analyzed. Analytical sensitivity is defined as the airborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the effective surface area of the filter, the filter area analyzed, and the volume of air sampled.

1.7.5.1.3 Bulk Samples

The range is dependent on the type of bulk material being analyzed. The sensitivity may be as low as 0.0001%.

1.7.5.2 Phase Contrast Microscopy

The normal quantitative working range of the method is 0.04 to 0.5 fiber/ cm^2 for a 1000 L air sample. An ideal counting range on the filter shall be 100 to 1300 fibers/mm^2. The limit of detection (LOD) is estimated to be 5.5 fibers per 100 fields or 7 fibers/mm^2. The LOD in fiber/cc will depend on sample volume and quantity of interfering dust but shall be <0.01 fiber/cm^2 for atmospheres free of interferences.

1.7.5.3 Polarized Light Microscopy

The laboratory shall utilize a method that provides a LOD that is appropriate and relevant for the intended use of the data. LOD shall be determined by the protocol in the method or applicable regulation.

1.7.6 Quality of Standards and Reagents

1.7.6.1 Transmission Electron Microscopy

a) The QC program shall establish and maintain provisions for asbestos standards.

b) Reference standards that are used in an asbestos laboratory shall be obtained from NIST, EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. 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.

c) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

d) All reagents used shall be analytical reagent grade or better.

e) The laboratory shall have mineral fibers or data from mineral fibers that will allow differentiating asbestos from at least the following “look-alikes”: fibrous talc, sepiolite, wollastonite, attapulgite (palygorskite), halloysite, vermiculite scrolls, antigorite, lizardite, pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample.

1.7.6.2 Phase Contrast Microscopy

Standards of known concentration have not been developed for this testing method. Routine workload samples that have been statistically validated and national proficiency testing samples such as Proficiency Analytical Testing (PAT) and Asbestos Analysts Registry (AAR) samples available from the American Industrial Hygiene Association (AIHA) may be utilized as reference samples (refer to Section D.6.2.2 b) to standardize the optical system and analyst. All other testing reagents and devices (HSE/NPL test slide and Walton-Beckett Graticule) shall conform to the specifications of the method (refer to National Institute for Occupational Safety and Health (NIOSH) 7400, Issue 2, 15 August 1994).

1.7.6.3 Polarized Light Microscopy

Refer to Section 1.7.6.1.

1.7.7 Data Acceptance/Rejection Criteria

1.7.7.1 Transmission Electron Microscopy

1.7.7.1.1 Water and Wastewater

a) The concentration of asbestos in a given sample shall be calculated in accordance with EPA/600/R-94/134, Method 100.2, Section 12.1.

b) Measurement Uncertainties. The laboratory shall calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

1.7.7.1.2 Air

a) The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized.

b) Measurement Uncertainties. The laboratory shall calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

1.7.7.1.3 Bulk Samples

a) The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized (e.g. EPA 600/M4-82-020(1982)).

b) Measurement Uncertainties. Proficiency testing for floor tiles analyzed by TEM following careful gravimetric reduction has revealed an inter-laboratory standard deviation of approximately 20% for residues containing 70% or more asbestos. Standard deviations range from 20% to 60% for residues with lower asbestos content.

1.7.7.2 Phase Contrast Microscopy

1.7.7.2.1 Airborne fiber concentration in a given sample shall be calculated in accordance with NIOSH 7400, Issue 2, 15 August 1994, Sections 20 and 21.

1.7.7.2.2 Measurement Uncertainties. The laboratory shall calculate and report the intra-laboratory and inter-laboratory RSD with each set of results (NIOSH 7400, Issue 2, 15 August 1994).

1.7.7.2.3 Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of the filter area covered with particulate shall be reported as “uncountable” or “probably biased”. Other fiber counts outside the 100-1300 fibers/mm^2 range shall be reported as having “greater than optimal variability” and as being “probably biased”.

1.7.7.3 Polarized Light Microscopy

1.7.7.3.1 The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized (e.g., EPA 600/M4-82-020(1982)).

1.7.7.3.2 Method Uncertainties. Precision and accuracy shall be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonable expanded errors shall be generated for different concentrations of asbestos.

1.7.8 Constant and Consistent Test Conditions Sample and Sampling Requirements

1.7.8.1 Samples shall be transported to the laboratory as soon as possible after collection. Date and time of sampling shall be noted on submittal forms. The names of the collectors with their signatures and the site shall be included on the chain-of-custody forms. No preservatives are required during sampling.

1.7.8.2 The laboratory shall establish and adhere to written procedures to minimize the possibility of cross contamination between samples.

1.7.8.3 Refer to the specific method of analysis for additional requirements.

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ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 4: Quality Systems for Chemical 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) Chemistry and Quality Systems Committees. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

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

Standard Revision History

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Sections 1.1 through 1.5.1; 1.5.3 through 1.6.3.2; and 1.7.2 through 1.7.4

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Section 1.5.2

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Section 1.7.1

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Entire Module

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Quality Systems for Chemical Testing

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VOLUME 1, MODULE 4
Quality Systems for Chemical Testing

1.0 Chemical Testing

1.1 Introduction

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

1.2 Scope

The essential QC procedures applicable to chemistry measurements are included in this module. Additional QC requirements that are either 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 are the preferred references. 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

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

When adding a new analyte to a reference method, the inclusion of the analyte in the method shall meet all required calibration requirements and the QC requirements of the method to which the analyte is being added. If no QC exists in the method, the laboratory shall adhere to the requirements outlined in a reference method of the same technology (when available). For example, when adding acetone to EPA Method 624, the calibration and QC requirements shall follow EPA Method 624. A method that meets these requirements shall be identified in such a way so that there is no confusion that the analyte list has been modified.

1.5 Method Validation

1.5.1 Validation of Methods

Prior to acceptance and institution of any method for which data will be reported, all methods shall be validated.

a) The laboratory shall validate reference methods via the procedures specified in Sections 1.5.2 and 1.5.3. For reference methods, the procedures outlined in Section 1.6 can satisfy the requirements of Section 1.5.3.

b) For all methods, except reference methods, the validation must comply with Volume 1, Module 2, Sections 5.4.5.1, 5.4.5.2, and 5.4.5.3. This validation must include the minimum requirements outlined in Sections 1.5.2, 1.5.3, and 1.5.4 of this module.

c) For both reference and non-standard methods, laboratories shall participate in proficiency testing programs. The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data.

1.5.2 Limit of Detection and Limit of Quantitation (however named)

Procedures used for determining limits of detection and quantitation shall be documented. Documentation shall include the quality system matrix type. All supporting data shall be retained.

1.5.2.1 Detection Limit (DL)

If a mandated test method or applicable regulation includes protocols for determining detection limits, they shall be followed. The laboratory shall document the procedure used for determining the DL. If the method or regulation does not contain specific directions for determination of the detection limit, the following requirements shall apply. DL determinations are not required for methods/analytes for which a detection limit is not applicable such as pH, color, odor, temperature, or dissolved oxygen. DL determinations based on low level spikes are not required for analytes for which no spiking solutions are available. If results are not reported below the limit of quantitation (LOQ), an initial DL determination is required, but ongoing verification is not.

1.5.2.1.1 Initial determination of the DL

The laboratory DL procedure, unless following a mandated test method or procedure, at a minimum, shall incorporate language addressing the following requirements:

a) the DL shall reflect current operating conditions;

b) the DL determination shall incorporate the entire analytical process;

c) the DL determination shall include data from low level spikes and routine method blanks prepared and analyzed over multiple days; at least one low level spike and routine method blank must be analyzed on each applicable instrument; a minimum of seven (7) replicates is required for both low level spikes and routine method blanks;

d) results from low level spikes used in the DL determination shall meet qualitative identification criteria in the method, and shall be above zero;

e) the DL procedure shall include criteria for and evaluation of false positive rates in routine method blanks;

f) the DL shall be determined for the analytes of interest in each test method in the quality system matrix of interest in which there are neither target analytes nor interferences at a concentration that would impact the results, or the DL shall be performed in the sample matrix of interest.

NOTE: One option is to follow the United States Environmental Protection Agency Method Detection Limit (MDL) procedure, effective September 27, 2017.

1.5.2.1.2 Ongoing verification of the DL

At a minimum, ongoing verification of the DL shall include assessments of spikes at or below the LOQ and of method blanks. A minimum of one (1) verification spike and one (1) blank shall be analyzed on each instrument during each quarter in which samples are being analyzed and results are being reported below the LOQ. The criteria listed in Section 1.5.2.1.1 shall be met for ongoing verification over the course of a year.

If the method is altered in a way other than routine maintenance, and the change can be expected to elevate the detection limit, then a spike at or below the LOQ concentration and a blank shall be prepared and analyzed. If the spike at the LOQ concentration gives a result meeting qualitative identification criteria above zero, and the blank gives a result below the DL, then the DL is verified. If not, the DL shall be re-determined.

In the event that verification fails, the laboratory shall perform a new DL study within thirty (30) calendar days.

1.5.2.1.3 When a new DL is determined, the laboratory shall verify that the LOQ value is greater than the DL. If it is not, the laboratory shall raise the LOQ value to greater than the DL.

1.5.2.2 Limit of Quantitation (LOQ)

If a mandated test method or applicable regulation includes protocols for determining quantitation limits, they shall be followed. The procedure used for determining the LOQ shall be documented by the laboratory. The laboratory shall select an LOQ for each analyte, consistent with the needs of its clients, and greater than the DL. An LOQ is required for each quality system matrix of interest, technology, method, and analyte, except for any component or property for which spiking solutions are not available or a quantitation limit is not appropriate, such as pH, color, odor, temperature, dissolved oxygen, or turbidity.

a) Each selected LOQ shall be verified through analysis of initial verification samples. An initial verification sample consists of a spiked matrix blank at or below the selected LOQ.

b) All sample processing and analysis steps performed for routine sample analysis shall be included in the LOQ verification testing.

c) The LOQ must be at or above the lowest corresponding calibration standard concentration with the exception of methods using a single point calibration.

d) The laboratory shall establish acceptance criteria for accuracy for the LOQ verification spikes.

1.5.2.2.1 Initial verification of the LOQ

When first establishing an LOQ, or when an LOQ concentration has been selected that is lower than the concentration of the LOQ verification spikes previously performed, an initial verification shall be performed as follows:

a) A minimum of seven (7) low level spikes at or below the LOQ concentration shall be processed through all steps of the method. Both preparation and analysis of these low level spikes shall include at least three (3) batches on three (3) separate days.

NOTE 1: Spiking slightly below the LOQ may help ensure that the results are also suitable for DL determination.

NOTE 2: If low level spikes have been analyzed in order to generate a DL, the results may be used to perform the initial verification of the LOQ.

i. If there are multiple instruments that will be assigned the same LOQ, then these low level spikes shall be distributed across all of the instruments.

ii. A minimum of two (2) low level spikes prepared and analyzed on different days shall be tested on each instrument.

b) Existing data may be used if compliant with the requirements for at least three (3) batches, generated within the last two (2) years and representative of current operations.

c) The LOQ is verified if the following criteria are met:

i. All results are quantitative (above zero and meet the qualitative identification criteria of the method; e.g., recognizable spectra, signal to noise requirements, and presence of qualifier ions).

If a result from an LOQ verification sample is not above zero and/or does not meet the qualitative identification criteria in the method, the problem shall be corrected and the verification repeated, or the LOQ verification shall be repeated at a higher concentration.

ii. Recovery of each analyte is within the laboratory established accuracy acceptance criteria.

iii. The LOQ is greater than the established DL and at or above the spiking concentration.

If the LOQ is less than or equal to the DL, the LOQ shall be raised to greater than the DL.

NOTE: It is **not** necessary to repeat the LOQ verification at a higher concentration when it is necessary to raise the LOQ to greater than the DL.

d) The laboratory shall document the results of the initial LOQ verification as described in Section 1.5.2.4.

1.5.2.2.2 Ongoing verification of the LOQ

The laboratory shall prepare and analyze a minimum of one (1) LOQ verification sample spiked at the same concentration as the initial LOQ verification on each instrument during each quarter in which samples are being analyzed for each quality system matrix, method, and analyte.

a) Results of each LOQ verification sample analysis shall be evaluated at the time of the testing and shall meet the qualitative identification criteria in the method and laboratory Standard Operating Procedure (SOP) and the quantitated result shall be greater than the DL and meet the laboratory established accuracy criteria as established by Section 1.5.2.2 d).

b) If a continuing LOQ verification test does not meet this requirement, the laboratory shall take corrective action and document a technically valid reason for the corrective action. Corrective action shall be one of the following: (i) correcting method or instrument performance and repeating the verification test; (ii) evaluating the laboratory established control limits to ensure they reflect current performance; or (iii) raising the spiking level (and the quantitation limit if the spiking level is above it) and repeating the initial verification study within thirty (30) calendar days of the initial failure. Any samples analyzed in a batch associated with a failing LOQ verification shall be reanalyzed or reported with qualifiers.

1.5.2.3 Verification of DL/LOQ

If no analysis was performed in a given year, the verification of the DL/LOQ is not required, but a new initial DL/LOQ verification shall be performed prior to analysis of client samples.

1.5.2.4 Documentation

At least once per year, the laboratory shall tabulate all results of the ongoing verification sample testing. All data representative of the current operations shall be used, if generated within the last two (2) years. A minimum of seven (7) samples is required.

a) The laboratory shall record the analytical and preparation methods used, dates of preparation and testing, the batch identifiers, the testing instrument, quality system matrix, technology, analyte, concentration in the spiked sample with units, and the test result (if any) for each LOQ and/or DL verification test.

b) For each analyte, the laboratory shall record the percent recovery, the number of results (n), the mean and standard deviation of the percent recovery, and the spiking concentration of the spiked samples with units. These data shall be provided to clients upon request.

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, by criteria given in the reference method or criteria established by the laboratory.

Precision and bias measurements shall evaluate the method across the analytical calibration range of the method. 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.

Examples of a systematic approach to evaluate precision and bias could be the following:

i. Analyze QC samples in triplicate containing the analytes of concern at or near the LOQ, at the upper-range of the calibration (upper 20%), 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 (1) 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. (Note that the three (3) samples at the LOQ concentration can demonstrate sensitivity as well.)

For each analyte, calculate the mean recovery for each day, for each level over each day, and for all nine (9) samples. Calculate the relative standard deviation for each of the separate means obtained. Compare the standard deviations for the different days and the standard deviations for the different concentrations. If the different standard deviations are all statistically insignificant (e.g., F-test), then compare the overall mean and standard deviation with the established criteria from above.

ii. A validation protocol, such as the Tier I, Tier II, and Tier III requirements in US EPA Office of Water's Alternate Test Procedure (ATP) approval process.

1.5.4 Evaluation of Selectivity

The laboratory shall evaluate selectivity by following the checks established within the method, which may include mass spectral tuning, second column confirmation, ICP inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, and electrode response factors.

1.6 Demonstration of Capability (DOC)

1.6.1 General

a) An individual who performs any activity involved with preparation and/or analysis of samples must have constant, close supervision (as defined in the laboratory's training procedure) until a satisfactory initial DOC is completed (see Section 1.6.2).

b) Thereafter, ongoing DOC (Section 1.6.3), as per the QC requirements in Section 1.7.2 (such as laboratory control samples), is required.

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

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

1.6.2 Initial DOC

An individual must successfully perform an initial DOC prior to using any method (see Section 1.6.1.a above), and any time there is a change in instrument type, method, or any time that a method has not been performed by the 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);

d) identification of method(s) performed;

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

f) date(s) of analysis; and

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 the concentration specified, or if unspecified, to a concentration of one (1) to four (4) times the LOQ.

b) At least four (4) aliquots shall be prepared and analyzed according to the method(s) 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 sample (in the same units) for each analyte 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 analytes meet the acceptance criteria, the analysis of actual samples may begin. If any one of the analytes does not meet the acceptance criteria, the performance is unacceptable for that analyte.

e) When one or more of the tested analytes fail at least one (1) 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 analytes of interest beginning with b) above.

ii. Beginning with b) above, repeat the test for all analytes 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 analytes 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 demonstration shall be performed for that analyte.

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 on-going capability by routinely meeting the QC requirements of the method, laboratory SOP, client specifications, and/or this Standard. If the method has not been performed by the analyst in a twelve (12) month period, an initial DOC (Section 1.6.2) shall be performed. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

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

a) acceptable performance of a blind sample (single blind to the analyst) or successful analysis of a blind performance sample on a similar method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 5030/8260);

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 LCSs or reference sample(s) for each method for each analyst each year;

d) a documented process of reviewing QC samples performed by an analyst or groups of analysts relative to the QC requirements of the method, laboratory SOP, client specifications, and/or this Standard. This review can be used 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 a pre-defined acceptance criterion (as defined by the laboratory or method) shall be performed.

1.7 Technical Requirements

1.7.1 Calibration

This module specifies the essential elements that shall define the procedures and documentation for initial calibration with second source verification and continuing calibration verification for methods that use calibration models including, but not limited to, average response factor or linear or quadratic regression, to ensure that the data shall be of known quality for the intended use. Calibration requirements for analytical support equipment are specified in Module 2. 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 by 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 regulation or mandated method are to be followed.

1.7.1.1 Initial Calibration

Samples shall be associated with an acceptable initial calibration. If the initial calibration is not acceptable, 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 calibration shall only be reported with appropriate data qualifiers.

The following items are essential elements of initial calibration:

a) the details of the initial calibration procedures including calculations, integrations, acceptance criteria, and associated statistics shall be included or referenced in the method SOP. When initial calibration procedures are referenced in the test method, then the referenced material shall be retained by the laboratory and be available for review;

b) sufficient raw data records shall be retained to permit reconstruction of the initial calibration (e.g., calibration date, method, instrument, analysis date, each analyte name, and analyst’s initials or signature; concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration);

c) the laboratory shall use the most recent initial calibration analyzed prior to the analytical batch, unless otherwise specified by the method;

d) standards used for calibration shall be traceable to a national standard, when commercially available;

e) the laboratory shall have a written procedure addressing removal and replacement of calibration standards. The procedure shall comply with the following requirements:

i The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.

ii. The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. A laboratory that chooses to remove a calibration standard from the interior of the calibration shall remove that particular standard calibration level for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.

iii. The laboratory shall adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.

iv. The laboratory shall ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for number of initial calibration points as mandated by this Standard, the method, or regulatory requirements.

v. The laboratory may replace a calibration standard provided that:

a. the laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;

b. the laboratory replaces all analytes of the replacement calibration standard if a standard within the interior of the calibration is replaced; and

c. the laboratory limits the replacement of calibration standards to one calibration standard concentration.

vi. The laboratory shall document a technically valid reason for either removal or replacement of any interior calibration point;

f) for regression or average response/calibration factor calibrations, the minimum number of non-zero calibration standards shall be as specified in the table below;

Type of Calibration Curve	Minimum Number of Calibration Standards[b]
Threshold Testing[a]	1
Average Response	4
Linear Fit	5
Quadratic Fit	6

[a] *The initial one-point calibration shall be at the project-specified threshold level.*

[b] *Fewer calibration standards may be used only if equipment firmware or software cannot accommodate the specified number of standards. Documentation detailing that limitation shall be maintained by the laboratory.*

g) the lowest calibration standard shall be at or below the lowest concentration for which quantitative data are to be reported without qualification;

h) the highest calibration standard shall be at or above the highest concentration for which quantitative data are to be reported without qualification;

i) sample results shall be quantitated from the initial calibration and may not be quantitated from any continuing calibration verification unless otherwise required by regulation, method, or program;

j) criteria for the acceptance of an initial calibration shall be established (e.g., correlation coefficient or relative standard deviation);

k) the laboratory shall use and document a measure of relative error in the calibration;

i. for calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error;

ii. for calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either:

a. measurement of the Relative Error (%RE)

Relative error is calculated using the following equation:

$$\%\ Relative\ Error = \frac{x'_i - x_i}{x_i} \times 100$$

x_i = True value for the calibration standard
x'_i = Measured concentration of the calibration standard

This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level.

The Relative Error at both of these levels shall meet the criteria specified in the method. If no criterion for the lowest calibration level is specified in the method, the criterion and the procedure for deriving the criterion shall be specified in the laboratory SOP.

or,

b. measurement of the Relative Standard Error (%RSE)

Relative Standard Error is calculated using the following equation:

$$\%\ RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x'_i - x_i}{x_i}\right]^2 \Big/ (n - p)}$$

x_i = True value of the calibration level i
x'_i = Measured concentration of calibration level i
p = Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3)
n = Number of calibration points

The RSE shall meet the criterion specified in the method. If no criterion is specified in the method, the maximum allowable RSE shall be numerically identical to the requirement for RSD in the method. If there is no specification for RSE or RSD in the method, then the RSE shall be specified in the laboratory SOP.

l) when test procedures are employed that specify calibration with a single calibration standard and a zero point (blank or zero, however specified by the method), the following shall occur:

i. The zero point and single calibration standard within the linear range shall be analyzed at least daily and used to establish the slope of the calibration.

ii. To verify adequate sensitivity a standard shall be analyzed at or below the lowest concentration for which quantitative data are to be reported without qualification. This standard shall be analyzed prior to sample analysis with each calibration and shall meet the quantitation limit criteria established by the method. If no criteria exist the laboratory shall specify criteria in the SOP;

m) for analysis of Aroclors which use a linear through origin model (or average response factor) the minimum requirement is to perform an initial multi-point calibration for a subset of Aroclors (e.g., a mixture of 1016/1260) and to use a one-point initial calibration to determine the calibration factor and pattern recognition for the remaining Aroclors;

n) Initial Calibration Verification (ICV): All initial calibrations shall be verified with a standard obtained from a second manufacturer or a separate lot prepared independently by the same manufacturer;

o) for those methods where reporting non-detected analytes based on successful completion of a sensitivity check is allowed (similar to threshold testing but only for non-detects) the requirements of this Standard shall not prohibit the practice;

p) some methods allow data within the linear range of the instrument, but above the daily calibration, to be reported without qualification. For these methods, the laboratory shall establish the upper reporting limit through analysis of a series of standards. The upper reporting limit is equal to the concentration of the highest standard meeting the method limits for accuracy. The laboratory shall establish linearity annually and check it at least quarterly with a standard at the top of the linear working range, or at the frequency defined by the method. The laboratory shall dilute samples with results above the linear calibration range, or qualify the over-range results as estimated values.

1.7.1.2 Continuing Calibration Verification (CCV)

The validity of the initial calibration shall be verified prior to sample analyses by a continuing calibration verification with each analytical batch. The following items are essential elements of continuing calibration verification.

a) The details of the continuing calibration procedure, calculations and associated statistics shall be included or referenced in the method SOP.

b) Calibration shall be verified for each compound, element, or other discrete chemical species, except for multi-component analytes such as Aroclors, chlordane, total petroleum hydrocarbons, or toxaphene, where a representative chemical, related substance or mixture can be used.

c) The concentration of the calibration verification standard shall be equal to or less than half the highest level in the calibration.

d) Instrument continuing calibration verification shall be performed at the beginning and end of each analytical batch, and at the frequency defined in the method except:

i. if an internal standard is used, calibration verification shall be performed at the beginning of each analytical batch, and at the frequency defined in the method;

ii. a second source initial calibration verification that passes the continuing calibration verification criteria may be used in place of a continuing calibration verification standard;

iii. a laboratory control sample (LCS) may be used in place of a continuing calibration verification (CCV) (but not as a replacement for a failing CCV) for methods where the calibration goes through the same process as the LCS (using the continuing calibration verification acceptance criteria).

e) Sufficient raw data records shall be retained to permit reconstruction of the continuing instrument calibration verification (e.g., method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations). Continuing calibration verification records shall explicitly connect the continuing calibration verification data to the initial calibration.

f) Criteria for the acceptance of a continuing instrument calibration verification shall be established. If the continuing instrument calibration verification results obtained are outside the established acceptance criteria, the following steps shall be taken:

i. if a cause for the calibration verification failure is identified *that impacts only the calibration verification sample* (e.g. a missed autosampler injection), then analysis may proceed if a second calibration verification sample is analyzed immediately and the result is within acceptance criteria. Samples analyzed previously shall be considered valid if bracketed by a passing calibration verification sample (refer to Section 1.7.1.2.d). The cause for the failure of the first calibration verification result shall be documented;

ii. if the cause for the calibration verification failure is not identifiable or has impacted other samples, then corrective action shall be performed and documented. Prior to analyzing samples, the laboratory shall demonstrate acceptable performance after corrective action with calibration verification or a new initial calibration shall be performed. Samples analyzed prior to the calibration verification failure shall be reanalyzed or the results qualified if calibration verification bracketing is required (refer to Section 1.7.1.2.d);

iii. Data associated with an unacceptable calibration verification shall be qualified if reported, and shall not be reported if prohibited by the client, a regulatory program or regulation. Data associated with calibration verifications that fail under the following special conditions shall still be qualified, but may use a different qualifier:

a. when the acceptance criteria for the continuing calibration verification are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b. when the acceptance criteria for the continuing calibration verification are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

1.7.2 Quality Control (QC)

The laboratory shall have QC procedures for monitoring the validity of environmental tests undertaken as specified in this Section.

1.7.2.1 Negative Control – Method Performance: Method Blank

a) The method blank is used to assess the samples in the preparation batch for possible contamination during the preparation and processing steps. 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 is contaminated. Any affected samples associated with a contaminated 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. In those instances for which no separate preparation method is used (for example, volatiles in water), the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of twenty (20) environmental samples, not including method blanks, LCS, matrix spikes and matrix duplicates.

c) The method blank shall consist of a quality system matrix that is similar to the associated samples and is known to be free of the analytes of interest.

d) Method blanks are not applicable for certain analyses, such as pH, Conductivity, Flash Point, and Temperature.

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

1.7.2.2.1 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, indicates that the analytical system is “out of control.” Any affected samples associated with an out of control LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes.

1.7.2.2.2 The LCS shall be analyzed at a minimum of one (1) per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available, such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen, or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water), the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of twenty (20) environmental samples, not including method blanks, LCS, matrix spikes and matrix duplicates.

1.7.2.2.3 The LCS is a quality system matrix, known to be free of analytes of interest, spiked with known 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.

Alternatively, the LCS may consist of a media containing known and verified concentrations of analytes or as Certified Reference Material (CRM). All analyte concentrations shall be within the calibration range of the methods. The following shall be used in choosing components for the spike mixtures.

The components to be spiked shall be as specified by the mandated method or regulation or as requested by the client. In the absence of specified spiking components, the laboratory shall spike per the following:

a) for those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike shall be chosen that represents the chemistries and elution patterns of the components to be reported; and

b) for those methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected shall be representative of all analytes reported.

The following criteria shall be used for determining the minimum number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a two (2) year period:

i. for methods that include one (1) to ten (10) targets, spike all components;

ii. for methods that include eleven (11) to twenty (20) targets, spike at least ten (10) components or 80%, whichever is greater;

iii. for methods with more than twenty (20) targets, spike at least sixteen (16) components.

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 QC samples and are designed as data quality indicators for a specific sample using the designated method. These controls alone are not used to judge laboratory performance.

Examples of matrix-specific QC include: Matrix Spike (MS), Matrix Spike Duplicate (MSD), sample duplicates, and surrogate spikes. 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, and evaluating and reporting results based on performance of the QC samples.

1.7.2.3.1 Matrix spike; matrix spike duplicates

a) Matrix-specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.

b) 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.

c) 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. If there are no specified components, the laboratory shall spike per the following:

i. For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike shall be chosen that represents the chemistries and elution patterns of the components to be reported.

ii. For those methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a two (2) year period.

a. For methods that include one (1) to ten (10) targets, spike all components.

b. For methods that include eleven (11) to twenty (20) targets, spike at least ten (10) components or 80%, whichever is greater.

c. For methods with more than twenty (20) targets, spike at least sixteen (16) components.

1.7.2.3.2 Matrix duplicates

a) Matrix duplicates 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. The matrix duplicate may provide a usable measure of sample homogeneity. It may also provide a measure of precision when target analytes are present.

b) The frequency of the analysis of matrix duplicates are as specified by the method or may be determined as part of the contract review process.

c) Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.

1.7.2.3.3 Surrogate spikes

a) Surrogates, when required, are chosen to reflect the chemistries of the targeted components of the method and are added prior to sample preparation/extraction.

b) Except where the matrix precludes its use or when not commercially available, surrogate compounds shall be added to all samples, standards, and blanks for all appropriate methods.

c) Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method. They are often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of select compounds.

1.7.2.4 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

1.7.2.5 Reagent Quality, Water Quality, and Checks

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

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

c) The laboratory shall verify the concentration of titrants in accordance with written laboratory procedures.

1.7.2.6 Selectivity

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

1.7.3 Data Acceptance/Rejection Criteria

1.7.3.1 Negative Control – Method Performance: Method Blank

While the goal is to have no detectable contaminants, 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 shall be investigated and measures taken to minimize or eliminate the problem and affected samples reprocessed or data shall be appropriately qualified if:

a) the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample;

b) the blank contamination otherwise affects the sample results as per the method requirements or the individual project data quality objectives; and

c) a blank is determined to be contaminated. The cause shall be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank shall be evaluated as to the best corrective action for the samples (e.g., reprocessing or data qualifying codes). In all cases, the corrective action shall be documented.

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.

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.

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. This includes any allowable marginal exceedance as described in b) below.

i. when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; or

ii. when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes.

b) Allowable Marginal Exceedances. If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. An ME is defined as being beyond the LCS control limit (three (3) standard deviations), but within the ME limits. ME limits are between three (3) and four (4) standard deviations around the mean. The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than eleven analytes.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken. Laboratories shall have a written procedure to monitor the application of marginal exceedance allowance to the LCS.

1.7.3.3 Sample Specific Controls

a) Matrix Spike; Matrix Spike Duplicates

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.

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 for that sample reported with appropriate data qualifying codes.

b) Matrix Duplicates

The results from matrix duplicates are primarily designed to assess the homogeneity of the particular sample chosen. If that sample is homogenous it may also describe the precision of analytical results in a given matrix. These may be expressed as RPD or another statistical treatment (e.g., absolute differences).

The laboratory shall document the calculation for RPD or other statistical treatments.

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 duplicates results outside established criteria, corrective action shall be documented or the data for that sample reported with appropriate data qualifying codes.

c) Surrogate Spikes

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. Surrogates outside the acceptance criteria shall be evaluated for the effect indicated for the individual sample results. The appropriate corrective action may be guided by the data quality objectives or other site-specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria shall include appropriate data qualifiers.

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.

iii. 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 sample preservation using readily available techniques, such as pH or chlorine, prior to or during sample preparation or analysis. An exception is allowed for volatile organic analyte analyses; chemical preservation may be checked after analysis.

ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 5: Quality Systems for Microbiological Testing

Approved American National Standard

ANSI

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) Microbiology and Quality Systems Expert Committees. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

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

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VOLUME 1, MODULE 5
Quality Systems for Microbiological Testing

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VOLUME 1, MODULE 5
Quality Systems for Microbiological Testing

1.0 Microbiological Testing

1.1 Introduction

This Standard applies to laboratories undertaking microbiological analysis of environmental samples. Microbiological testing refers to and includes the detection, isolation, enumeration, or identification of microorganisms (and/or their metabolites), or determination of the presence or absence of growth in materials and media. The evaluation of laboratories for this discipline is in conjunction with a quality system as specified in the general requirements module. Adherence to those quality system requirements and all quality control (QC) procedures specified in this module will ensure that microbiological test results are fit for the intended use.

1.2 Scope

The essential QC procedures applicable to microbiological analysis are included in this module. Additional QC or program requirements that are either 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

Source Water: When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.

1.3.2 Exclusions and Exceptions

Reserved

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

a) For methods other than reference methods, validation must comply with Volume 1, Module 2. This validation must include the minimum requirements outlined in Sections 1.5.1, 1.5.2, and 1.5.3 of this module.

b) For both reference and non-standard methods, laboratories shall participate in proficiency testing (PT) programs, where available.

c) The laboratory shall maintain documentation of the validation procedure for as long as the method is in use, and for at least five (5) years past the date of last use.

1.5.1 Accuracy – Use at least one (1) known pure positive reference culture at the anticipated environmental conditions and compare the method results to that of a reference method.

1.5.2 Precision – Perform at least ten (10) replicate analyses with both the proposed and reference method, using a sample containing the target microorganisms of choice. The results shall show that the precision of the proposed method is statistically equivalent or better than that of the reference method.

1.5.3 Selectivity (sensitivity) – Verify all responses in at least ten (10) samples using mixed cultures that include the target organism(s) and at varying concentrations (microbial identification testing or equivalent processes may be used). Calculate the number of false positive and false negative results.

1.6 Demonstration of Capability (DOC)

1.6.1 General

1.6.1.1 An individual who performs any activity involved with preparation and/or analysis of samples must have constant, close supervision (as defined in the laboratory's training procedure) until a satisfactory initial DOC is completed (see Section 1.6.2).

1.6.1.2 Thereafter, ongoing DOC (Section 1.6.3), must be performed and documented at least every twelve (12) months.

1.6.1.3 In cases where an individual has prepared and/or analyzed samples using a method that has been in use by the laboratory for at least one (1) year prior to applying for accreditation and where 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.

1.6.1.4 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) organism(s);

d) identification of method(s) performed;

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

f) date(s) of analysis; and

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 target organism(s) shall be diluted in a volume of sterile, quality system matrix (a sample in which no target organisms or interferences are present at concentrations that will impact the results of a specific method). When required by method, the diluent shall be sterile buffered water and/or sterile peptone water unless specified by the manufacturer. Prepare at least four (4) aliquots at the concentration specified, or if unspecified, to the countable range for plate methods or working range for most probable number (MPN) type methods.

b) At least four (4) aliquots shall be prepared and analyzed concurrently according to the method.

c) Using all of the results, convert these results to logarithmic values, then calculate the mean recovery and standard deviation of the log converted results in the appropriate reporting units for each organism of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence, the laboratory shall assess performance against established and documented criteria.

d) For qualitative tests, acceptable performance in a blind study, either internally or externally generated, may be used to meet this Standard, provided that the study consists of a minimum of a blank, a negative culture, and a positive culture for each target organism.

e) 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 such as relative standard deviation (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.

f) 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 initial DOC for all parameters of interest beginning with b) above.

   ii. Repeat the initial DOC for all parameters that failed to meet criteria.

g) 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 organisms of interest beginning with b) above.

1.6.3 Ongoing DOC

1.6.3.1 The laboratory shall have a documented procedure describing ongoing DOC that includes how the laboratory will identify data associated with ongoing DOCs. The analyst(s) shall demonstrate ongoing capability by routinely meeting the QC requirements of the method, laboratory SOP, client specifications, and/or this Standard. If the method has not been performed by the analyst in a twelve (12) month period, an initial DOC (Section 1.6.2) shall be performed prior to performing analysis. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

1.6.3.2 This ongoing demonstration may include one of the following, or by performing another initial DOC.

a) Analysis of one (1) sample of clean matrix that is fortified with a known quantity of the target organism, with results meeting the laboratory acceptance criteria for accuracy and, where applicable to the testing technique, also meeting the observational details expected for the presumptive, confirmed and completed phases defined in the method.

b) Analysis of one (1) positive sample in duplicate for each target organism and test, with results meeting the laboratory acceptance criterion for precision.

c) Acceptable results for a blind proficiency test sample or sample set, as required by the program, for target organisms in each field of accreditation.

d) Performance of an alternate adequate procedure for the field of accreditation, the procedure and acceptance criteria being documented in the laboratory's quality system.

e) A documented process of reviewing QC samples performed by an analyst, or groups of analysts, relative to the QC requirements of the method, laboratory SOP, client specifications, and/or this Standard. This review can be used to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary.

f) If a) through e) 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 Calibration

1.7.1.1 The laboratory shall have documented procedures for calibration, verification, and QC of support equipment including conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments. These procedures shall refer to applicable reference methods.

1.7.1.2 For instruments that are continuous monitors, such as in-line specific conductance meters:

a) the laboratory shall document acceptable calibration verification at least once a month;

b) an initial calibration shall be performed if a continuing calibration is unacceptable, or when the instrument is being returned to service after having been taken off-line.

1.7.2 Continuing Calibration

Reserved for specific procedures.

1.7.3 Quality Control

1.7.3.1 Quality and Sterility of Standards, Reagents, Materials, and Media

The laboratory shall demonstrate and document that the quality of the reagents and media used is appropriate for the test concerned including, but not limited to, test conditions and incubation times.

a) Sterility Checks – All materials and supplies that are needed to process the sample and are required to be sterile prior to use (whether sterilized in the laboratory or purchased as sterilized) must be checked by the laboratory once per purchased or prepared lot using non-selective growth media as appropriate. Certificates of analysis provided by vendors shall be verified by the laboratory and retained in accordance with V1M2 5.6.4.2.a. These checks shall include, but are not limited to:

i. The laboratory shall perform a sterility check for each lot of prepared, ready-to-use, media and on each batch of media prepared in the laboratory.

a. For chromo/fluorgenic media: add media to sterile deionized water and incubate at the appropriate temperature and time.

b. For all other media, incubate uninocculated at the appropriate temperature and time. Where media are made as concentrates (e.g., double strength), then the medium shall be diluted to working strength with sterile deionized water before testing.

ii. The laboratory shall perform a sterility check on one (1) funnel per lot of pre-sterilized single use funnels using non-selective growth media. The laboratory shall perform a sterility check on one (1) funnel per batch of laboratory-sterilized funnels, using non-selective growth media.

iii. The laboratory shall perform a sterility check on at least one (1) container for each lot of purchased, pre-sterilized sample containers with non-selective growth media. The laboratory shall perform a sterility check on one (1) container/object per sterilization batch sterilized in the laboratory with nonselective growth media.

iv. The laboratory shall perform a sterility check on each batch of dilution water prepared in the laboratory and on each lot of pre-prepared, ready-to-use dilution water with non-selective growth media. The concentration of the non-selective growth media shall be single strength after the addition of dilution water.

v. The laboratory shall perform a sterility check on at least one (1) filter from each new lot of membrane filters with nonselective growth media.

b) Media – Culture media may be prepared from commercial dehydrated powders or may be purchased ready-to-use.

i. All media shall be tested for performance (e.g., for selectivity, sensitivity, sterility, growth promotion, and growth inhibition). These tests shall be performed at a minimum with first use.

ii. The laboratory shall use all media within the expiration date or shelf-life provided by the manufacturer.

iii. The laboratory shall use all laboratory-prepared media within the holding time limits specified in the accredited method.

iv. The laboratory shall have detailed testing criteria information defined in the laboratory's methods, SOPs, or similar documentation.

c) The laboratory shall use reagents, media and commercial dehydrated powders within the shelf-life of the product, and shall maintain documentation as per Volume1, Module2 Quality Systems: General Requirements, Section 5.6.4.2.

d) Reagent Water

i. The laboratory shall monitor the quality of the reagent water used in the laboratory, which will come into contact with test organisms and is used in preparation of media, solutions, and buffers, for bactericidal and inhibitory substances. This water shall be distilled water, deionized water, or reverse-osmosis-produced water.

ii. The laboratory shall monitor the quality of the water for disinfectant residual, specific conductance, total organic carbon, and heterotrophic bacteria plate count monthly (when in use), when maintenance is performed on the water treatment system, or at

startup after a period of disuse longer than one month. Analysis may be performed by another certified laboratory.

iii. The laboratory shall monitor the quality of the water for metals (Cd, Cr, Cu, Ni, Pb, and Zn) and the Bacteriological Water Quality Test (to determine presence of toxic agents or growth promoting substances) annually. An exception to performing the Bacteriological Water Quality Test shall be given to laboratories that can supply documentation to show that their water source meets the criteria, as specified by the method, for High Quality (Type I) or Medium Quality (Type II) reagent water. Analysis may be performed by another certified laboratory.

iv. Results of the above analyses shall meet the specifications of the required method. Records of analyses shall be maintained for five (5) years.

v. Reagent water purchased from an outside source and used for the preparations of media, solutions and buffers shall meet the criteria specified in items ii) and iii) above. The laboratory shall have documented records of this information.

vi. Reagent water that has been opened for longer than the testing intervals specified in items i) through iv), or in the accredited method, shall either be re-tested or discarded.

e) Dilution water, however used, includes buffer water and/or peptone water. The laboratory shall monitor the quality of the dilution water for sterility, pH and volume once per lot or batch whether purchased or lab-prepared.

f) Documentation for media and reagents prepared in the laboratory shall include date of preparation, preparer's initials, type, manufacturer, lot number, final pH, expiration date, and the amount of reagents used. Documentation for media purchased pre-prepared, ready-to-use (including reagent water purchased from outside sources) shall include manufacturer, lot number, type of media received, date of receipt, expiration date of the media, and pH of the media. Records shall be retained by the laboratory in accordance with Volume 1, Module 2, Section 5.4.6.2.

1.7.3.2 Method Blanks

The laboratory shall demonstrate that the filtration equipment and filters, sample containers, media, and reagents have not been contaminated through improper handling or preparation, or environmental exposure.

a) For filtration technique, the laboratory shall conduct method blanks per the analytical method. At a minimum, the filtration series shall include a beginning and ending blank. The filtration series may include single or multiple filtration units, which have been sterilized prior to beginning the series.

b) The filtration series is considered ended when more than thirty (30) minutes elapses between successive filtrations. During a filtration series, filter funnels shall be rinsed with three (3) 20-30 ml portions of sterile rinse water after each sample filtration. In addition, laboratories shall insert a method blank after every ten (10) samples or sanitize filtration units by UV light (254-nm) after sample filtration.

c) For pour plate technique, method blanks of the medium shall be made by pouring, at a minimum, one (1) uninoculated plate for each lot of pre-prepared, ready-to-use media and for each batch of medium prepared in the laboratory.

1.7.3.3 Test Variability/Reproducibility

For methods that specify counts (i.e. cfu/100mL or MPN/100mL), such as membrane filter, plated media or other methods which specify a quantitative result, duplicate counts shall be performed monthly on one (1) positive sample for each month that the test is performed. If the laboratory has two (2) or more analysts, each analyst shall count typical results on the same sample. Counts shall be within ten percent (10%) difference to be acceptable. In a laboratory with only one (1) microbiology analyst, the same sample shall be counted twice by the analyst, with no more than a five percent (5%) difference between the counts.

1.7.3.4 Sample Specific Controls (where applicable)

a) The laboratory shall perform matrix spikes per method requirements.

b) The laboratory shall perform sample matrix duplicates per method requirements.

1.7.3.5 Data Reduction

The calculations, data reduction and statistical interpretations specified by each method shall be identified and followed.

1.7.3.6 Selectivity

a) All growth and recovery media shall be checked to assure that the target organism(s) respond in an acceptable and predictable manner once per lot or batch.

b) To ensure that analysis results are accurate, target organism identity shall be verified as specified in the method (e.g., by use of the completed test or by use of secondary verification tests such as a catalase test, or by the use of a selective medium such as *Brilliant Green Lactose Bile Broth* (BGLB) or *EC* or *EC + MUG* broth).

c) In order to ensure identity and traceability, reference cultures used for positive and negative controls shall be obtained from a recognized national collection, organization, or manufacturer recognized by the accreditation body. Microorganisms may be single-use preparations or cultures maintained for their intended use by documented procedures that demonstrate the continued purity and viability of the organism.

i. Reference cultures may be revived (if freeze-dried) or transferred from slants and sub-cultured once to provide reference stocks. The reference stocks shall be preserved by a technique that maintains the characteristics of the strains. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they shall not be refrozen and re-used.

ii. Working stocks shall not be sequentially cultured more than five (5) times and shall not be sub-cultured to replace reference stocks.

d) Culture Controls (i.e. working cultures)

i. Negative Culture Controls

a. Negative culture controls demonstrate that the medium does not support the growth of non-target organisms or does not exhibit the typical positive reaction of the target organism(s).

b. Each pre-prepared, ready-to-use lot of selective medium (including chromofluorogenic reagent), and each batch of selective medium prepared in the laboratory, shall be analyzed with one (1) or more known negative culture

controls (i.e. non-target organisms), as appropriate to the method. This shall be done prior to first use of the medium.

ii. Positive Culture Controls

a. Positive culture controls demonstrate that the medium can support the growth of the target organism(s), and that the medium produces the specified or expected reaction to the target organism(s).

b. Each pre-prepared, ready-to-use lot of medium (including chromo/fluorogenic reagent) and each batch of medium prepared in the laboratory shall be tested with at least one (1) or more known pure positive culture controls (i.e. target organism) as appropriate to the method and that produce typical results based on the method. This shall be done prior to first use of the medium.

1.7.3.7 Constant and Consistent Test Conditions

a) Laboratory Facilities

Floors and work surfaces shall be non-absorbent and easy to clean and disinfect. Work surfaces shall be adequately sealed. Laboratories shall provide sufficient storage space, and shall be clean and free from dust accumulation.

b) Laboratory Equipment

i. Temperature Measuring Devices

The laboratory shall use temperature measuring devices such as liquid-in-glass thermometers, thermocouples, or platinum-resistance thermometers to assess and document equipment temperatures. The temperature measuring devices shall be appropriate quality to meet specification(s) in the method.

The graduation and range of the temperature measuring devices shall be appropriate for the required accuracy of the measurement. Temperature measuring devices shall be verified to national or international standards for temperature. Verification shall be performed at least annually (see TNI Volume 1, Module 2, Section 5.5.13.1). This verification may be accomplished by a single point provided that it represents the method mandated temperature and use conditions.

ii. Sterilization Equipment

a. Autoclaves

1. The laboratory shall evaluate the performance of each autoclave initially by establishing its functional properties and performance, for example, heat distribution characteristics with respect to typical uses. Autoclaves shall meet specified temperature tolerances. Pressure cookers shall not be used for sterilization of growth media.

2. The laboratory shall demonstrate proper sterilization temperature by use of a continuous temperature recording device or by use of a maximum registering thermometer with every cycle. The laboratory shall, at least once during each month that the autoclave is used, demonstrate the effective sterilization through the use of appropriate biological indicators. The selected biological indicator shall be effective at the sterilization temperature and time needed to sterilize lactose-based media. The

laboratory shall use temperature-sensitive tape with the contents of each autoclave run to indicate that the autoclave contents have been processed.

3. The laboratory shall maintain records of autoclave operations for every cycle. Records shall include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out), and analyst's initials.

4. Autoclave maintenance, internally or by service contract, shall be performed annually, and shall include a pressure check and verification of temperature device. Records of the maintenance shall be maintained in equipment logs. When it has been determined that the autoclave has no leaks, pressure checks can be documented using the formula PV = nRT.

5. The laboratory shall check the autoclave mechanical timing device quarterly against a stopwatch and document the actual time elapsed.

b. Ovens

The laboratory shall check ovens used for sterilization for sterilization effectiveness monthly with appropriate biological indicators. The laboratory shall maintain records for each cycle that include date, cycle time, temperature, contents, and analyst's initials. The laboratory shall use temperature sensitive tape with the contents of each run to indicate that the contents have been processed.

iii. Volumetric Equipment

The laboratory shall verify equipment used for measuring volume as follows:

a. Equipment with movable parts, such as automatic dispensers, dispensers/ diluters, and mechanical hand pipettes, shall be verified for accuracy quarterly.

b. Equipment, such as filter funnels, bottles, non-Class A glassware, and other containers with volumetric markings (including sample analysis vessels), shall be verified once per lot prior to first use.

c. The volume of the disposable volumetric equipment, such as sample bottles and disposable pipettes, shall be checked once per lot.

d. Verification of volume shall be considered acceptable if the accuracy is within 2.5% of expected volume. This verification can be volumetric as compared to Class A or gravimetric.

iv. UV Instruments

The laboratory shall evaluate UV instruments used for sanitization quarterly for effectiveness with an appropriate UV light meter, by plate count, agar spread plates, or other methods providing equivalent results, such as UV-cide strips. Replace bulbs if output is less than 70% of original for light tests or if count reduction is less than 99% for a plate containing 200 to 300 organisms.

v. Incubators, Water Baths

a. The laboratory shall establish the uniformity of temperature distribution and equilibrium conditions in incubators and water baths prior to first use after installation or service. The equilibrium check shall include time required after test

sample addition to re-establish equilibrium conditions under full capacity load appropriate for the intended use.

b. During periods when samples are under test, the laboratory shall have a system in place to monitor and document the temperature of incubators and water baths twice daily, at least four (4) hours apart. “Under test” is defined as the time period that the sample is in the incubation phase of the method. Data loggers, continuous temperature monitoring devices, or other temperature monitoring equipment can be used as long as they can be calibrated in accordance with TNI Volume 1, Module 2, Section 5.5.13.1 for Support Equipment. Records shall be maintained in accordance with Volume 1, Module 2, Section 4.13: Records Maintenance.

NOTE: There is no intent to take the temperature of incubation units during periods when there are no samples under test.

vi. Labware (Glassware and Plasticware)

a. The laboratory shall have a documented procedure for washing labware, if applicable. Detergents designed for laboratory use shall be used.

b. Glassware shall be made of borosilicate or other non-corrosive material, free of chips and cracks, and shall have readable measurement marks.

c. Labware that is washed and reused shall be tested for possible presence of residues that may inhibit or promote growth of microorganisms by performing the Inhibitory Residue Test initially and each time the laboratory changes the detergent formulation or washing procedures.

d. Washed labware shall be tested at least once daily, each day of washing, for possible acid or alkaline residue by testing at least one (1) piece of labware with a suitable pH indicator such as bromothymol blue. Records of tests shall be maintained.

1.7.4 Data Acceptance/Rejection Criteria

Methods criteria and evaluation methods shall be used.

1.7.5 Sample Handling

Receipt of samples must comply with Volume 1, Module 2, Sections 5.8.6 and 5.8.7, as well as:

1.7.5.1 Samples that require thermal preservation shall be considered acceptable if the arrival temperature of a representative sample container meets the method or mandated temperature requirement. Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of this section or the method or the regulatory requirement. In these cases, the samples ***may*** be considered acceptable if the samples are received on ice with evidence that the cooling process has begun.

NOTE: The intent is for the samples to be preserved immediately and analyzed as soon as possible.

1.7.5.2 Microbiological samples from known chlorinated sources (such as wastewater effluent), unknown sources where disinfectant (e.g. chlorine) usage is suspected (such as a new client or a new source), and all potable water supplies (including source water) shall be checked for absence of disinfectant residual in the laboratory unless all of the following conditions are met:

a. The laboratory can show that the received sample containers are from its laboratory or have been appropriately tested and documented;

b. Sufficient sodium thiosulfate was in each container before sample collection to neutralize at minimum 5 mg/L of chlorine for drinking water and 15 mg/L of chlorine for wastewater samples;

c. One (1) container from each batch of laboratory-prepared containers or lot of purchased ready-to-use containers is checked to ensure efficacy of the sodium thiosulfate to 5 mg/L chlorine or 15 mg/L chlorine as appropriate and the check is documented;

d. Disinfectant residual is checked in the field and actual concentration is documented with sample submission.

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THE NELAC INSTITUTE

ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 6: Quality Systems for Radiochemical Testing

Approved American National Standard ANSI

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) Radiochemistry Expert Committee. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

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

Standard Revision History

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Quality Systems for Radiochemical Testing

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VOLUME 1, MODULE 6
Quality Systems for Radiochemical Testing

1.0 Radiochemical Testing

1.1 Introduction

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

1.2 Scope

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

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

1.3 Terms and Definitions

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

1.3.1 Additional Terms and Definitions

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

1.3.2 Exclusions and Exceptions

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

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

1.4 Method Selection

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

1.5 Method Validation

1.5.1 Validation of Methods

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

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

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

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

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

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

g) The laboratory shall analyze for all methods, whenever available, externally-produced QC samples from a nationally- or internationally-recognized source (i.e., a national metrology institute, accredited TNI Proficiency Test (PT) Provider, an accredited ISO 17043: 2010[5] PT Provider, an accredited ISO Guide 34: 2009[6] reference material provider, or from an ANSI N42.22[7] compliant PT manufacturer). The laboratory shall evaluate the results of these analyses to determine its ability to produce acceptable data.

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

1.5.2 Detection Capability

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

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

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[5] ISO Guide 17043:2010, *Conformity assessment - General requirements for proficiency testing;* International Organization for Standardization and International Electrotechnical Commission. Available from: http://www.iso.org/.
[6] ISO Guide 34:2009(E), *General requirements for the competence of reference material producers;* International Organization for Standardization and International Electrotechnical Commission. Available from: http://www.iso.org/.
[7] ANSI N42.22-1995, *American National Standard - Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control,* Institute of Electrical and Electronics Engineers, 1995 (2005). Available at: http://ieeexplore.ieee.org/.

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

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

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

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

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

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

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

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

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

1.5.3 Evaluation of Precision and Bias

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

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;

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

1.5.4 Measurement Uncertainty

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

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

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

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

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

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

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

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

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

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

[8] *Validation and Peer Review of U.S. Environmental Protection Agency Radiochemical Methods of Analysis*; EPA Forum on Environmental Measurements (FEM), FEM Method Validation Team, FEM Document Number 2006-01, September 29, 2006. Available at: https://www.epa.gov/measurements/method-validation-and-peer-review-policies-and-guidelines.
[9] EURACHEM Guide. 2014. *The Fitness for Purpose of Analytical Methods, A Laboratory Guide to Method Validation and Related Topics*. Available at: http://www.eurachem.org/.

ii. A comparison of the experimentally-observed precision evaluation need not be performed for measurements that are required to be reported only with Counting Uncertainty per Section 1.5.4 a) ii).

1.5.5 Evaluation of Selectivity

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

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

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

iii. spectral and instrumental interferences.

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

1.6 Demonstration of Capability (DOC)

1.6.1 General

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

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

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

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

1.6.2 Initial DOC

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

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

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

b) matrix;

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

d) identification of method(s) performed;

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

f) date(s) of analysis;

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

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

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

b) Where gamma-ray spectrometry is used to identify and quantify more than one analyte, the Test Sample shall contain gamma-emitting radionuclides that represent the low (e.g., ^{241}Am), 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 mean of the blank results in the appropriate reporting units and the standard deviations of the population sample (in the same units) for each parameter of interest. When it is not possible to determine means and standard deviation, the laboratory shall assess performance against established and documented criteria.

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

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

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

1.6.3 Ongoing DOC

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

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

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

b) Another initial DOC.

c) At least four (4) consecutive spiked samples (e.g., batch laboratory control samples) each with levels of precision and accuracy consistent with those specified in the method scope; and four (4) consecutive blank samples, each 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 Laboratory Control Samples (LCS) and four (4) consecutive blank samples for each method for each analyst each year. The laboratory shall specify acceptable limits for precision and accuracy prior to analysis.

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

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

1.7 Technical Requirements

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

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

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

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

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

1.7.1.1 Initial Set-Up of Instrumentation

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

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

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

1.7.1.2 Initial Calibration

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

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

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

ii. after a repair 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. Some techniques require multiple-point calibration curves to correlate a number of parameters other than activity. For example:

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

v. mass-crosstalk calibration of gas-flow proportional; and
vi. quench-crosstalk calibration of liquid scintillation detectors.

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

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

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

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

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

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

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

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

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

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

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 (2) forms:

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

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

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

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

1.7.1.4 Instrument Performance Checks

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

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

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

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

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

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

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

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

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

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

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

i. Gamma-ray spectrometry systems

Detection efficiency, energy calibration, and peak resolution:

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

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

ii. Alpha-particle spectrometry systems

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

iii. Gas-proportional and semiconductor alpha/beta detectors

Alpha and beta efficiency: Day of use.

iv. Liquid scintillation detectors

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

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

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

Efficiency: Day of use.

c) Exceptions to minimum frequencies for performance checks:

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

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

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

1.7.1.5 Subtraction Background Measurements

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

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

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

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

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

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

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

a. Gamma-ray spectrometry systems: Monthly.

b. Alpha-particle spectrometry systems: Monthly.

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

d. Liquid scintillation detectors.

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

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

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

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

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

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

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

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

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

1.7.1.6 Short-Term Background Checks

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

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

i. Indicate the frequency and length of checks.

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

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

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

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

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

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

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

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

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

1.7.1.7 Contamination Monitoring

The laboratory shall have written procedures that address cases where radiation detectors have been contaminated, as determined by the subtraction background measurements, short-term background checks, or method blanks (Section 1.7.2.3). 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 QC program that monitors and assesses the performance of the laboratory's analytical systems. At a minimum, the QC program shall incorporate requirements imposed by regulation, methods and this Standard. Where imposed regulations are more stringent than this Standard, the imposed regulations take precedence (see Module 2, Section 5.9.3.c). If it is not apparent which requirement is more stringent, the laboratory shall follow the requirements of the regulation or the mandated method. Where there are no established requirements, the laboratory may reference guidelines consistent with MARLAP[3] or other consensus standard organizations in its quality system.

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

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

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

ii. Where testing is performed that does not involve physical or chemical processing which affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta

counting of air filters, or swipes on gas proportional detectors), an RMB may be initiated in lieu of a Preparation Batch. The samples and associated QC in the RMB shall share similar physical and chemical parameters, and analytical configurations (e.g., analytes, geometry, calibration, and background correction).

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

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

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

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

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

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

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

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

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

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

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

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

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

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

i. The laboratory shall prepare the MB using materials that are free of analytes of interest at levels that will interfere with the evaluation of the results. If an analyte-free matrix is not available, the laboratory shall use a surrogate matrix to simulate the quality system matrix.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

d) The laboratory shall spike the LCS at a level such that the uncertainty of the analytical result is less than one-third (1/3) of the acceptance criteria. For example, if it is required that the LCS result be within +/- 30% of the known value, the laboratory shall spike the LCS at a level such that the uncertainty of the analytical result is less than or equal to 10%. When practical, the LCS should be spiked at a level comparable to the action level if known; or 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 meet the requirements for reference standards provided in Section 1.7.2.6.c. The final prepared LCS need not be traceable to a national standard organization. The LCS shall include all of the radionuclide(s) being determined with the following exceptions:

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

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

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

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

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

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

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

1.7.2.4 Sample-Specific QC Measures

The laboratory shall document procedures for determining the effect of the sample matrix on the analytical results. These procedures relate to the analyses of specific QC samples and are designed as data quality indicators for a specific sample using the designated method. Examples of sample-specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD), Matrix Duplicate (MD), Tracers, and Carriers. The laboratory shall have procedures in place for tracking, managing, and handling sample-specific QC criteria including spiking radionuclides at appropriate activities, calculating recoveries, determining variability (e.g., relative percent difference and/or z-score), and evaluating and reporting results based on the performance of the QC samples.

a) Matrix Spike

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

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

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

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

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

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

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

viii. When available, the standard used to prepare the MS shall meet the requirements for reference standard provided in Section 1.7.2.6.c. The final prepared MS need not be traceable to a national standards organization.

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

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

iii. At a minimum, the laboratory shall analyze one (1) MD per Preparation Batch or RMB. For RMBs, the MD shall consist of a second measurement of one sample. If the batch is counted on more than one (1) detector, the MD shall be performed on a second detector.

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

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

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 QC measures to be used to assess the associated sample result acceptance.

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

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

iv. The chemical yield shall be assessed against acceptance criteria specified in the method, regulation, contract or laboratory SOP. The laboratory shall develop its criteria

for data acceptance based on guidelines established in the MARLAP[3] or other criteria such as control charting developed by the laboratory. This assessment shall meet established project or program MQOs (Section 1.3.1).

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

1.7.2.5 Data Reduction

a) The procedures for data reduction shall be documented.

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

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

1.7.2.6 Reagent Quality, Water Quality, and Checks

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

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

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

i. 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 Guide 34:2009[6] accredited reference material provider, or an ANSI N42.22[7] reference material manufacturer.

ii. Reference standards shall be accompanied with a certificate of calibration that meets the requirements of either ISO Guide 31:2000[11], or ANSI N42.22[7], Section 8, Certificates, and shall include at least the following information: manufacturer, radionuclides calibrated, identification number, calibration method, activities or emission rates with associated uncertainties and the confidence limits, standard quantity, activity reference time (date or time as appropriate to the half-life of the radionuclide), physical and/or chemical description of the source, and radionuclide impurities.

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

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

[11] ISO Guide 31:2000, *Reference materials - Contents of certificates and labels*; International Organization for Standardization, 2000. Available from: http://www.iso.org/.

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

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

vii. 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 test instruments consistently operate within the specifications required of the application for which the equipment is used, according to Section 1.7.1.

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

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

1.7.3 Data Evaluation and Reporting

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

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

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

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

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

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

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

1.7.3.3 Sample-Specific Controls

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

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

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

b) Tracers and Carriers

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

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

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

1.7.3.4 Evaluation of Sample Results

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

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 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 1.5.4.

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

f) Project- or client-specified reporting requirements can take precedence 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 system (if there are no established mandatory criteria).

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

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

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

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

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ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 7: Quality Systems for Toxicity 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. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

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

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Quality Systems for Toxicity Testing

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VOLUME 1, MODULE 7
Quality Systems for Toxicity Testing

1.0 Toxicity Testing

1.1 Introduction

This Standard applies to laboratories measuring the toxicity and/or bioaccumulation of contaminants in effluents (whole effluent toxicity or WET), receiving waters, sediments, elutriates, leachates and soils. In addition to the essential quality control standards described below, some methods may have additional or other requirements based on factors such as the type of organism evaluated and contain detailed quality control requirements for toxicity testing activities. 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

The essential quality control procedures applicable to toxicity 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

When referred to in this module, “sensitivity” relates to the meaning referenced in the accredited method.

1.3.2 Exclusions and Exceptions

Reserved

1.4 Method Selection

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

The characteristics of validated methods (e.g., the uncertainty of the results, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the users’ needs.

1.5 Method Validation

Validation is the confirmation by examination and the objective evidence that the particular requirements for a specific intended use are fulfilled.

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 (QC) requirements in Section 1.7.1.2 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 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 1.6.2.1 shall be completed.

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

In general, this demonstration does not test the performance of the method in real world samples. However, before any results are reported, the initial DOC shall be performed. An initial DOC may be completed by a group of analysts and is for situations in which several individuals perform part of a set of activities that would produce a testing result.

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 significant change in 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 available for each affected employee:

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

b) matrix;

c) species and endpoint(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 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.

Each analyst shall meet the QC requirements as specified in Section 1.7.1.2.

1.6.3 Ongoing DOC

The laboratory shall have a documented procedure describing ongoing DOC. The analyst(s) shall demonstrate on-going capability by meeting the QC 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 on-going DOC are adequate. This on-going demonstration may include performing another initial demonstration of capability as per 1.6.2 or a documented process of analyst review using QC samples can serve as the annual on-going DOC. QC samples shall be reviewed to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary.

1.7 Technical Requirements

1.7.1 Quality Control

The laboratory shall have QC procedures for monitoring the validity of environmental tests undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

a) regular use of certified reference materials and/or internal QC using secondary reference materials;

b) participation in inter-laboratory comparison or proficiency-testing program;

c) replicate tests using the same or different methods;

d) retesting of retained samples; and

e) correlation of results for different characteristics of a sample (for example, total phosphate should be greater than or equal to orthophosphate).

1.7.1.1 Essential Quality Control Procedures

These general QC principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory and are further described in this module. The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have detailed written protocols in place to monitor the following QCs:

i. positive and negative controls to monitor tests such as blanks, spikes, reference toxicants;

ii. tests to define the variability and/or repeatability of the laboratory results such as replicates;

iii. measures to evaluate method capability, such as percent minimum significant difference (PMSD);

iv. selection of appropriate formulae to reduce raw data to final results such as regression and statistical analyses;

v. selection and use of reagents and standards of appropriate quality;

vi. measures to assure the selectivity of the test for its intended purpose; and

vii. measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the method such as temperature, humidity, light or specific equipment conditions.

b) All QC measures shall be assessed and evaluated on an ongoing basis, and QC acceptance criteria shall be used to determine the usability of the data.

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist.

d) The QC protocols specified by the laboratory's method manual shall be followed. The laboratory shall ensure that the essential standards outlined in this document or regulations (whichever are more stringent) are incorporated into their method manuals. When it is not apparent which is more stringent, the QC in the regulations is to be followed.

1.7.1.2 Positive and Negative Controls

a) Positive Control. Reference toxicant tests demonstrate a laboratory's ability to obtain consistent results with the method and evaluate the overall health and sensitivity of test organisms over time.

i. The laboratory shall demonstrate its ability to obtain consistent results with standard reference toxicants (SRT).

ii. Ongoing laboratory performance shall be demonstrated by performing routine SRT testing for each method, species and endpoint in accordance with the minimum frequency requirements specified in Section 1.7.1.2.a.iii.

iii. The frequency of ongoing laboratory reference toxicant testing shall be as follows unless the method specifically requires less frequent SRT tests (e.g., sediment tests).

For methods conducted at a frequency of monthly or greater, SRT tests shall be conducted monthly.

For methods and species commonly used in the laboratory, but which are tested at a frequency of less than monthly, SRT tests shall be conducted concurrently with the environmental test.

If the test organisms are obtained from an outside source, the sensitivity of each batch of organisms received from a supplier shall be determined via a concurrent SRT test unless the supplier can provide control chart data for the last five SRT tests using the same SRT and test conditions. Supplied SRT data may not be older than six (6) months.

iv. These standards do not currently specify a particular reference toxicant and dilution series. However, if the regulation identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements. All reference toxicant tests conducted for a given method and species shall use the same reference toxicant, test concentrations, dilution water and data analysis methods. A dilution factor of 0.5x or greater shall be used for both acute and chronic tests.

v. The reference toxicant tests shall be conducted following the procedures required in the method.

b) Negative Controls – Control, Brine Control, Control Sediment, Control Soil or Dilution Water

i. The standards for the use, type and frequency of testing of negative controls are specified by the methods and by permit or regulation and shall be followed. A negative control is included with each test to evaluate test performance and the health and sensitivity of the specific batch of organisms.

ii. Appropriate additional negative controls shall be included when sample adjustments (for example addition of thiosulfate for dechlorination) or solvent carriers are used in the test.

1.7.1.3 Variability and/or Reproducibility

Intra-laboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described above.

1.7.1.4 Test Sensitivity

a) The PMSD shall be calculated according to the formula specified by the method and reported with the test results.

b) Point estimates: (LCp, ICp, or ECp) – Confidence intervals shall be reported as a measure of the precision around the point estimate value, when the calculation is possible.

1.7.1.5 Selection and Use of Reagents and Standards

a) The grade of all reagents used in toxicity tests is specified in the method except the reference standard. All reference standards shall be prepared from chemicals that are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the Chemistry Module.

c) Only reagent-grade water collected from distillation or de-ionization units is used to prepare reagents.

1.7.1.6 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid cross-contamination.

b) Laboratory space shall be adequate for the types and numbers of tests performed. The building shall provide adequate cooling, heating and illumination for conducting testing and culturing; hot and cold running water shall be available for cleaning equipment.

c) Air used for aeration of test solutions, dilution waters and cultures shall be free of oil and fumes.

d) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s)) and the names(s) of the taxonomic expert(s) shall be kept on file at the laboratory. When organisms are obtained from an outside source the supplier shall provide this same information.

e) Equipment used for routine support measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, ammonia and weight shall be calibrated, and/or standardized per manufacturer's instructions. All measurements and calibrations shall be documented.

f) Test temperature shall be maintained as specified for the method. Temperature control equipment shall be adequate to maintain the required test temperature(s). The average daily temperature of the test solutions shall be maintained within method specified range. The minimum frequency of measurement shall be once per twenty-four (24) hour period. The test temperature for continuous-flow toxicity tests shall be recorded and monitored continuously. Where electronic data loggers are used, temperature shall be monitored at a frequency sufficient to capture temporal variations of the environmental control system.

g) Reagent grade water, prepared by any combination of distillation, reverse osmosis, ion exchange, activated carbon and particle filtration, shall meet the method specified requirements.

h) The quality of the standard dilution water used for testing or culturing shall be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. Water used for culturing and testing shall be analyzed for toxic metals and organics whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified.

i) The quality of the food used for testing or culturing shall be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. The laboratory shall have written procedures for the evaluation of food acceptance.

j) A subset of organisms used in bioaccumulation tests shall be analyzed at the start of the test (baseline) for the target compounds to be measured in the bioaccumulation tests.

k) Test chamber size and test solution volume shall be as specified in the method. All test chambers used in a test shall be identical.

l) Test organisms shall be fed the quantity and type food or nutrients specified in the method. They shall also be fed at the intervals specified in the methods.

m) All organisms in a test shall be from the same source and lot. Where available, certified seeds are used for soil tests.

n) All organisms used in tests, or used as broodstock to produce neonate test organisms (for example cladocerans and larval fish), shall appear healthy, show no signs of stress or disease and exhibit acceptable survival (90% or greater) during the twenty-four (24) hour period immediately preceding use in tests.

o) All materials used for test chambers, culture tanks, tubing, etc. and coming in contact with test samples, solutions, control water, sediment or soil or food shall be non-toxic and cleaned as described in the methods. Materials shall not reduce or add to sample toxicity. Appropriate materials for use in toxicity testing and culturing are described in the methods.

p) Light intensity shall be maintained as specified in the methods. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the methods and shall be documented at least quarterly. For algal and plant tests, the light intensity shall be measured and recorded at the start of each test.

q) The health and culturing conditions of all organisms used for testing shall be documented by the testing laboratory. Such documentation shall include culture conditions (e.g. salinity, hardness, temperature, pH) and observations of any stress, disease or mortality. When organisms are obtained from an outside source, the laboratory shall obtain written documentation of these water quality parameters and biological observations for each lot of organism received. These observations shall adequately address the twenty-four (24) hour time period referenced in item 1.7.1.6 n) above. The laboratory shall also record each of these observations and water quality parameters upon the arrival of the organisms at the testing laboratory.

r) Age and the age range of the test organisms shall be as specified in the method. Supporting information, such as hatch dates and times, times of brood releases and metrics (for example, chironomid head capsule width) shall be documented.

s) The maximum holding time of effluents (elapsed time from sample collection to first use in a test) shall not exceed thirty-six (36) hours; samples may be used for renewal up to seventy-two (72) hours after first use except as prescribed by the method and approved by the regulatory agency having authority for program oversight.

t) All tests shall have at least the minimum number of replicates per treatment as prescribed by the method.

u) The control population of Ceriodaphnia in chronic effluent or receiving water tests shall contain no more than 20% males.

v) The culturing of C. dubia shall be adequate such that blocking by parentage can be established.

w) Dissolved oxygen and pH in aquatic tests shall be within acceptable range at test initiation. Minimal aeration is provided to tests if acceptable dissolved oxygen concentrations cannot be otherwise maintained.

x) Test soils or sediments shall be within the geochemical tolerance range of the test organism.

y) An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each method). The acceptability of the test shall depend on the experience and professional judgment of the technical director and the permitting authority.

1.7.2 Data Acceptance/Rejection Criteria

1.7.2.1 Positive Controls

A laboratory shall record the control performance and statistical endpoints (such as NOEC or ECp) for each method and species on control charts. The laboratory shall also evaluate precision (i.e. coefficient of variation, CV) for these tests against method specific or laboratory-derived criteria to determine validity of the testing result.

For endpoints that are point estimates (ICp, ECp), control charts are constructed by plotting the cumulative mean and the control limits, which consist of the upper and lower 95% confidence limits (+/- 2 standard deviations). For endpoints from hypothesis tests (NOEC, NOAEC) the values are plotted directly, and the control limits consist of one concentration interval above and below the concentration representing the central tendency (i.e. the mode).

For endpoints that are point estimates the cumulative mean CV is calculated. For endpoints from hypothesis tests, the PMSD is calculated. These values are maintained on control charts.

Control chart limits are expected to be exceeded occasionally regardless of how well a laboratory performs. Acceptance limits for point estimates (ICp, ECp) that are based on 95% confidence limits should theoretically be exceeded for one in twenty tests. Depending on the dilution factor and test sensitivity, control charts based on hypothesis test values (NOEC, NOAEC) may be expected to be exceeded on a similar frequency. Test results that fall outside of control chart limits at a frequency of 5% or less, or which fall just outside control chart limits (especially in the case of highly proficient laboratories which may develop relatively narrow acceptance limits over time), are not rejected de facto. Such data are evaluated in comparison with control chart characteristics including the width of the acceptance limits and the degree of departure of the value from acceptance limits.

Laboratories shall develop acceptance/rejection policies, consistent with the methods, for SRT data which considers source of test organisms, the direction of the deviation, test dilution factor, test sensitivity (for hypothesis test values), testing frequency, out-of-control test frequency, relative width of acceptance limits, inter-test CV, and degree of difference between test results and acceptance limits.

In the case of reference toxicant data which fail to meet control chart acceptance criteria, the test data are examined for defects, corrective action taken and the test repeated if necessary, using a different batch of organisms or the data is qualified.

Intra-laboratory precision is determined on an ongoing basis through the use of control charts. The control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC50 for acute tests, or as appropriate hypothesis test values, such as the NOEC or NOAEC, over time within a laboratory.

1.7.2.2 Negative Controls

The test acceptability criteria specified in the method shall be achieved for both the reference toxicant and the effluent or environmental sample toxicity test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity tests.

1.7.2.3 Selection of Appropriate Statistical Analysis Methods

a) Methods of data analysis and reporting as specified by language in the regulation, permit, or the method shall be followed as required.

b) Toxicity data shall be plotted on semi-logarithmic graph paper, relating time, mortality, and effluent concentration to verify computational results.

1.7.3 Sample Handling

All samples shall be chilled to 0-6°C during or immediately after collection except as prescribed by the method and approved by the regulatory agency having authority for program oversight.