U.S. Department of Energy

Quality Systems for Analytical Services Revision 2.8

Approved: _____ January, 2012
Jorge Ferrer Date
DOECAP Manager

Note: DOE requirements are identified in gray boxes. In cases where DOE requirements differ from the NELAC requirements, DOE requirements supersede the NELAC requirements.

Record of Revision for Quality Systems for Analytical Services Document

Revision Number	Effective Date	Reason for Revision	Pages or Sections Affected
0	7/2003	Original issue	NA
1	2/2004		
2.0	8/2005	Revision of NELAC guidance document	Entire document
2.1	11/2005	Resolution of Revision 2.0 issues	
2.1	11/2005	Preface	ii
2.1	11/2005	Definitions	14
2.1	11/2005	Acronyms	26
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Revision Number	Effective Date	Reason for Revision	Pages or Sections Affected
2.1	11/2005	D.4 DOE-15	D-33
2.1	11/2005	D.4 DOE-19	D-36
2.1	11/2005	D.4.9 d) i-iv)	D45-46
2.1	11/2005	D.4.9 i)5)	D-47
2.1	11/2005	D.4.10 c)	D-48
2.1	11/2005	D.4.11 a)1)	D-49
2.1	11/2005	D.4.12 c)3)	D-50
2.1	11/2005	D.4.12 d)5)	D-50
2.1	11/2005	D.4.13 a)2)	D-51
2.2	10/2006	Add DOE box number to grey box (D.1 DOE-10)	D-9
2.2	10/2006	Paragraph b)5) change “less” than to “greater” than	D-11
2.2	10/2006	Text in grey box D.4 DOE-4, 1st and 3rd paragraphs changed	D-25
2.2	10/2006	Text in grey box D.4 DOE-5 changed to reflect all radiochemistry samples and characterized solid materials	D-26
2.2	10/2006	Add new grey box (D.4 DOE-19) under item 4)	D-35
2.2	10/2006	Renumber grey boxes D.4 DOE-19 to D.4 DOE-30	D-35 to D-43
2.2	10/2006	Add MDA formula for use when sample and blank count time durations differ	D-38
2.2	10/2006	Add DOE box number to grey box (D.4 DOE-32)	D-44

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Revision Number	Effective Date	Reason for Revision	Pages or Sections Affected
2.2	10/2006	Added “or Po-210” to item c)4)	D-48
2.2	10/2006	Wording changed in item b)1)i) to reflect the use of 7 peaks over an energy range of “approximately” 0.059 to 2 MeV.	D-50
2.2	10/2006	Item c) reworded to give specific guidance for energy calibration requirements	D-51
2.2	10/2006	General editing for rogue characters and extraneous blank lines	Entire document
2.3	10/2007	Added DOE Order 414.1C to the objectives for the DOE QSAS.	vi
2.3	10/2007	Changed supercede to supersede in document footnotes.	Entire document.
2.3	11/2007	Amended requirements for subcontracting of environmental tests	36
2.3	11/2007	Amended requirements for purchasing services and supplies	36
2.3	10/2007	Added requirements to close logbook pages.	44
2.3	11/2007	Amended the requirements for software change control.	60
2.3	10/2007	Added requirements for the use and availability of fire extinguishers for the LIMS.	60
2.3	10/2007	Added calibration verification requirements for Class 1 check weights.	62
2.3	10/2007	Deleted “by the individual using the device” for the daily calibration check of automatic dispensing devices.	62

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Revision Number	Effective Date	Reason for Revision	Pages or Sections Affected
2.3	10/2007	Deleted the requirement for inventory of routine spare parts.	66
2.3	10/2007	Added additional requirements for refrigerator storage blanks.	75
2.3	10/2007	Added: When DOE requires SW-846 Methods, the additional QC in this appendix is required.	D-1
2.3	10/2007	Added clarification for batch blank corrective actions	D-26
2.3	10/2007	Added the following for gas flow proportional counting planchets: Sample weights shall be documented and stable prior to counting.	D-48
2.3	10/2007	Defined the isotopes to be used for cross talk.	D-48
2.4	10/2008	Deleted DOE Order 450.1.	vii
2.4	10/2008	Deleted Appendix E (from table of contents)	ix
2.4	10/2008	Defined Statistical Control Charts	30
2.4	10/2008	Added gray box for QA plan requirements in 4.2 DOE-2	32
2.4	10/2008	Added further requirements for quality manual in 4.2 DOE-3	33
2.4	10/2008	Defined operator aids in gray box 4.3 DOE-1	34
2.4	10/2008	Added requirements for independent review of records and criteria for quality records	43
2.4	10/2008	Renumbered gray boxes 4.12 DOE-2 to DOE-6	43-45
2.4	10/2008	Added additional requirements to gray box 4.12 DOE-6 for laboratory notebooks	45

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Record of Revision for Quality Systems for Analytical Services Document

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2.4	10/2008	Added requirements for LIMS user identifications, passwords, employee training in computer security awareness, and acceptance criteria for changes to LIMS hardware and communication components - 5.4 DOE-4	60-61
2.4	10/2008	Additional requirements for temperature monitoring and data loggers gray box 5.5 DOE-4	64
2.4	10/2008	Added requirements that the laboratory must maintain a list of major equipment, etc. gray box 5.5 DOE-9	67
2.4	10.2008	Renumber gray boxes 5.5 DOE-9 and DOE-10 to DOE-10 and DOE-11	67
2.4	10/2008	Added expiration date for purchased stock mixtures gray box 5-6 DOE-1	71
2.4	10/2008	Defined refrigerator temperature requirements to 0º to 6º gray box 5.8 DOE-2	73
2.4	10/2008	Renumbered 5.8 DOE-2 to DOE-8 to DOE-3 to DOE-9	74-77
2.4	10/2008	Deleted DOE-EH, added requirements for participation in MAPEP, defined DOECAP policy for MAPEP remedial samples gray box 5-9 DOE-1	78-79
2.4	10/2008	Changed URL for bacteria procurement, DOECAP, and changed URL for AIHA certification. Deleted reference to DOE Order 450.1.	A-2
2.4	10/2008	Added URL for EPA GALP Standard	A-6
2.4	10/2008	Deleted to not correct values that have indirect yields between 100-110%	D-31
2.4	10/2008	Deleted the requirement for same day background	D-35

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Revision Number	Effective Date	Reason for Revision	Pages or Sections Affected
		checks for gamma spec	
2.4	10/2008	Clarification of counting uncertainty when there are zero counts	D-42
2.4	10/2008	Deleted Appendix E	E-1
2.5	11/2009	Added Definition for Batch for NDA (DRAFT)	5
2.5	11/2009	Added Definition for Data Quality Objective (NDA DRAFT)	9
2.5	11/2009	Defined Measurement Control Sample (NDA DRAFT)	14
2.5	11/2009	Defined NDA	25
2.5	11/2009	Defined requirements for willful avoidance of approved corrective action plans	4.10 DOE-1
2.5	11/2009	Added requirement for annual computer security awareness training	5.4 DOE-4
2.5	11/2009	Added requirement for the definition of the frequency for software testing	5.4 DOE-4
2.5	11/2009	Added requirement for the periodic testing of backup LIMS data	5.4 DOE-4
2.5	11/2009	Added that calibration of balances and verification Class 1 weights must be performed using recognized National Metrology Institute, such as NIST, traceable references	5.5 DOE-4
2.5	11/2009	Removed the requirement for the temperature monitoring of refrigerators and freezers when DOE samples are not being stored	5.5 DOE-4
2.5	11/2009	Added requirement that reagents and solvents	5.6 DOE-1

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Record of Revision for Quality Systems for Analytical Services Document

Revision Number	Effective Date	Reason for Revision	Pages or Sections Affected
		shall be checked prior to use	
2.5	11/2009	Added requirements for the receipt and radiological survey of shipping containers for radiological containing samples	5.8 DOE-7
2.5	11/2009	Added requirements for the calibration and operational checks for radiological survey instrumentation	5.8 DOE-7
2.5	11/2009	Added additional criteria for the evaluation of performance evaluation results (MAPEP criteria and will be applied to non-MAPEP results	5.9 DOE-1
2.5	11/2009	Added requirement for a trained, backup RSO	6.2.4
2.5	11/2009	Added requirement for documented site visit safety briefing	6.4.4
2.5	11/2009	Added criteria for evaluations of waste brokering or waste treatment facilities	6.5.3
2.5	11/2009	Deleted reference to standard ASTM E1598 Standard Practice for Conducting Early Seedling Growth Tests which as been withdrawn.	A-1
2.5	11/2009	Added gray box to reference deleted standard ISO 1990. General requirements for the competence of calibration and testing laboratories. ISO/IEC 25	A-3
2.5	11/2009	Added gray box to document the replacement of ISO 1990. Guidelines for auditing quality systems. Part 1 Auditing. ISO 10011-1 with ASQ QE19011S-2004 or 2008.	A-3
2.5	11/2009	Added gray box to document the replacement of ISO 1991. Guidelines for auditing quality systems. Part 2 Qualification criteria for quality system auditors. With ASQ QE19011S-2004 or 2008.	A-3

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2.5	11/2009	Added gray box to document the replacement of ISO 1991. Guidelines for auditing quality systems. Part 3 Management of audit programs. With ASQ QE19011S-2004 or 2008.	A-3
2.5	11/2009	Added gray box to document the replacement of ISO 1992 Quality assurance requirements for measuring equipment – Part 1: Metrological confirmation for measuring equipment. ISO 10012-2003. Requirement has been replaced by AWQ Q10012-2003.	A-3
2.5	11/2009	Added gray box to reference that ISO 1993 Calibration and testing laboratory accreditation systems – General requirements for operation and recognition – ISO/IEC Guide 58 has been withdrawn	A-3
2.5	11/2009	Added gray box to reference that ISO 1993 Quality Management and Quality Systems – Part 4: Guideline for quality improvements has been withdrawn.	A-3
2.5	11/2009	ISO 1993 Statistics – Vocabulary and symbols – Part 1: Probability and general statistical terms. has been replaced by ASQ A3434-1.	A-3
2.5	11/2009	Added gray box to reference that ISO 9002 has been withdrawn and replaced by ISO	A-4
2.5	11/2009	Added gray box to reference that ISO 10012-2 has been withdrawn and replaced by ASQ Q10012.	A-4
2.5	11/2009	Added NDA References	A-7
2.5	11/2009	Draft criteria for non-destructive assay (NDA) measurements	Appendix E
2.6	11/2010	Added references for negative numbers for	A-1, A-4

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		radiochemical testing	
2.6	11/2010	Revised the wording for the quality manual	4.2 DOE-3
2.6	11/2010	Added requirement for the establishment of a hardcopy or electronic log to document all changes to electronic data that affect data quality.	4.2 DOE-3
2.6	11/2010	Added establishment of priority of software change requests.	5.4 DOE-4
2.6	11/10	Add criteria for verification of mechanical volumetric dispensing devices and add requirement that all mechanical dispensing devices shall be checked for accuracy on a quarterly basis	5.5 DOE-5
2.6	11/2010	Moved MAPEP Priority I corrective actions to gray box 5.9 DOE-1	5.9 DOE-1
2.6	11/2010	Corrected incorrect term TNI PEOP/PTPA to PTOB/PTPA	5.9 DOE-1
2.6	11/2010	Defined responsibility for all DOE sites that submit samples to a laboratory and the responsibility of the receiving laboratory.	6.0 DOE-1
2.6	11/2010	Added requirement for the updates of the radiological materials inventory according to the requirements of the RML, or, if the RML does not contain specific requirements, at a minimum of once per seven days from receipt of radiological samples.	6.2.5
2.6	11/2010	Added requirement for backup personnel for Emergency Response personnel.	6.4.5
2.6	11/2010	Defined frequency for inspections of waste brokering and TSDF evaluations to be performed	6.5.3

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Record of Revision for Quality Systems for Analytical Services Document

Revision Number	Effective Date	Reason for Revision	Pages or Sections Affected
		every three years.	
2.6	11/2010	Deleted – For most low background measurements, ~400 net tracer counts are generally adequate to meet the tracer yield requirement,	D.4 DOE-14
2.6	11/2010	Added references for Negative Numbers, Radiochemical Testing	D.4 DOE-26
2.6	11/2010	Added criteria for non-destructive assay (NDA) measurements	Appendix E
2.7	11/2011	Added gray box to the QSAS Preface	
2.7	11/2011	Added DOE Order 414.1D to the objectives section of the QSAS Preface	
2.7	11/2011	Add Duplicate error ratio to definitions and acronyms	3.2
2.7	11/2011	Changed reference to Section 5.9.5 to 5.9 DOE-1	4.1 DOE-2
2.7	11/2011	Added chemical hygiene plan (CHP) to documents that shall be referenced by the quality manual	4.2 DOE-3
2.7	11/2011	Added consumed during analysis to sample disposal record requirements. Added requirements for IH samples consumed during preparation and analysis.	4.12 DOE-1
2.7	11/2011	Revised the requirement to state laboratory management shall develop…….	5.2 DOE-3
2.7	11/2011	Revised SOPS to include Radioactive and/or Hazardous Materials to distinguish between the two types of material	5.4 DOE-1
2.7	11/2011	Changed reference for 5.5 DOE-5 to 5.5.2.1.e	5.5 DOE-5

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2.7	11/2011	Added requirements for ventilation hoods for receiving DOE samples and the requirement for a procedure and records for contamination control.	5.8 DOE-7
2.7	11/2011	Removed “in most cases: for the laboratory requirement for the disposal of derived waste	6.0 DOE-1
2.7	11/2011	Added accountability requirements for samples consumed during analysis	6.5.3
2.7	11/2011	Added an upper energy range for gamma detectors	D.4.13 2)
2.7	11/2011	Added definition for DER – Duplicate Error Ratio	D.4 DOE-15
2.7	11/2011	Add revised requirements for NDA	Appendix E
2.8	1/2012	Added requirements for receiving beryllium, beryllium oxide, and asbestos materials.	5.8 DOE-7
2.8	1/2012	Revised Table of Contents to correct page numbers	xii

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Preface to the U.S. Department of Energy (DOE) Quality Systems for Analytical Services

Purpose: This Quality Systems for Analytical Services (QSAS) establishes a single, integrated quality assurance (QA) program for providers of analytical laboratories supporting the U.S. DOE operations. This document will allow laboratories providing analytical services to DOE to implement a unified standard, thus improving efficiency and quality in a cost-effective manner. Furthermore, this QSAS represents a significant advance toward harmonizing analytical data quality requirements across various Federal agencies and closely follows the approach being taken by the Department of Defense and by the U.S. Environmental Protection Agency (EPA).

Objectives: As the primary requirements document for the DOE QA program for analytical laboratory services, this QSAS conforms to DOE Order 414.1 B, 414.1C and/or 414.1D, "Quality Assurance," This Order applies directly to Government Owned Client Operated (GOCO) and commercial laboratories that perform work for the DOE. To conform to these orders, this QSAS addresses the following objectives:

- Allows line organizations to minimize risk while maximizing reliability and performance of subclient laboratories;
- Provides for the review, evaluation and improvement of performance, based on an approved QA Program;
- Establishes a single, integrated QA program; and
- Establishes criteria for independent assessments, through the DOE Consolidated Audit Program (DOECAP), to measure quality and to promote improvement.

Technical Basis: Providers of laboratory services to DOE shall be committed to generating data of known, documented quality. To present a coherent approach to this objective, the QSAS provides specific technical requirements and clarification for the implementation of DOE requirements. This document is based in total on EPA's National Environmental Laboratory Accreditation Conference (NELAC) Chapter 5, "Quality Systems," as implemented in July 2005, based on ISO 17025, General Requirements for the Competence of Testing and Calibration Laboratories. This QSAS also incorporates EPA's Performance Approach. **The NELAC Chapter 5 requirements do not fully encompass the DOE requirements; therefore, gray texts are added to address DOE-specific additions/clarifications for implementation to DOE analytical laboratory requirements. In cases where DOE requirements differ from the NELAC requirements, DOE requirements supersede the NELAC requirements.**

Applicability: **While NELAC is designed to apply solely to environmental testing laboratories, this QSAS is designed to serve as the standard reference and basis for providers of analytical testing services to the DOE.** For providers of analytical testing services, the QSAS must be incorporated into contract vehicles or agreements.

Implementation and Assessment: The effective implementation of the requirements in this QSAS is the basis for qualification of laboratories providing analytical services to DOE. It is recognized that there may be exceptions to the applicability of portions of this document. In such cases, exceptions must be documented and approved in writing. Requirements that are specific to DOE and that supplement, deviate, or clarify NELAC standards, are highlighted in the QSAS by the use of gray shaded text. The qualification of laboratories is assessed through the DOECAP. Current DOECAP Audit Checklists can be accessed on-line at: https://doecap.oro.doe.gov/.

This document is maintained by the DOECAP Operations Team

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QUALITY SYSTEMS FOR ANALYTICAL SERVICES

INTRODUCTION

Each laboratory shall have a quality system. The laboratory's quality system is the process by which the laboratory conducts its activities so as to provide the client with data of known and documented quality with which to demonstrate regulatory compliance and for other decision-making purposes. This system includes a process by which appropriate analytical methods are selected, their capability is evaluated and their performance is documented. The quality system shall be documented in the laboratory's quality manual.

This QSAS contains detailed quality system requirements for consistent and uniform implementation by both the laboratories conducting testing under these standards and the evaluation of those laboratories by accrediting authorities. Each laboratory seeking accreditation under the National Environmental Laboratory Accreditation Program (NELAP) must ensure that they are implementing their quality system and that all Quality Control (QC) procedures specified in this document are being followed. The Quality Assurance (QA) policies, which establish QC procedure, are applicable to environmental laboratories regardless of size and complexity.

Introduction DOE-1

Laboratories seeking a contract award under this DOE QSAS must ensure implementation of all QA policies and the essential QC Standard Operating Procedures (SOPs) specified in this QSAS.

The growth in use of quality systems has increased the need to ensure that laboratories, which form part of larger organizations or offer other services, can operate to a quality system that is seen as compliant with ISO 9001 or ISO 9002, as well as with this document. Care has been taken, therefore, to incorporate all those requirements of ISO 9001 and ISO 9002 that are relevant to the scope of environmental testing services that are covered by the laboratory's quality system.

Environmental testing laboratories that comply with this Standard will therefore also operate in accordance with ISO 9001 or ISO 9002.

Certification against ISO 9001 and ISO 9002 does not of itself demonstrate the competence of the laboratory to produce technically valid data and results.

NELAC Chapter 5 is organized according to the structure of ISO/IEC 17025, 1999. Where deemed necessary, specific areas within this document may contain more information than specified by ISO/IEC 17025.

Introduction DOE-2

This QSAS is organized to the structure of NELAC Chapter 5.

All items identified in this document shall be available for on-site inspection and data audit.

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1.0 SCOPE

1.1 This document specifies the general requirements for the competence to carry out environmental tests, including sampling. It covers testing performed using standard methods, non-standard methods, and laboratory-developed methods.

1.1 DOE-1

NELAC references to environmental laboratories apply to all DOE laboratories wherein the QSAS has been incorporated into the contract or agreement.

It contains all of the requirements that environmental testing laboratories must meet if they wish to demonstrate that they operate a quality system, are technically competent, and are able to generate technically valid results.

If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. (See the supplemental accreditation requirements in Section 1.8.2 of the NELAC Standard.)

1.1 DOE-2

Note: Section 1.8.2 of the NELAC Standard does not apply to DOE.

1.2 This document is applicable to all organizations performing environmental tests. These include, for example, first-, second- and third-party laboratories, and laboratories where environmental testing forms part of inspection and product certification.

This document is applicable to all laboratories regardless of the number of personnel or the extent of the scope of environmental testing activities. When a laboratory does not undertake one or more of the activities covered by this document, such as sampling and the design/development of new methods, the requirements of those clauses do not apply.

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

1.3 DOE-1

The NELAC Chapter 5 requirements do not fully encompass the DOE requirements; therefore, gray texts are added to address DOE-specific additions/clarifications for implementation to DOE analytical laboratory requirements.

1.4 This document is for use by laboratories in developing their quality, administrative and technical systems that govern their operations. Laboratory clients, regulatory authorities and accreditation authorities may also use it in confirming or recognizing the competence of laboratories.

This document includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority granting the recognition (or approval).

1.5 Compliance with regulatory and safety requirements on the operation of laboratories is not covered by this document. It is the laboratory's responsibility to comply with the relevant health and safety requirements.

1.5 DOE-1

DOE Health and Safety requirements are defined in Section 6.

1.6 If environmental testing laboratories comply with the requirements of this document, they will operate a quality system for their environmental testing activities that also meets the requirements of ISO 9001 when they engage in the design/development of new methods, and/or develop test programs combining standard and non-standard test and calibration methods, and ISO 9002 when they only use standard methods. ISO/IEC 17025 covers several technical competence requirements that are not covered by ISO 9001 and ISO 9002.

1.7 An integral part of a Quality System is the data integrity procedures. The data integrity procedures provide assurance that a highly ethical approach to testing is a key component of all laboratory planning, training and implementation of methods. The following sections in this document address data integrity procedures:

Management Requirements	4.2.6, 4.2.7, and 4.2.8
Training	5.2.7
Control and Documentation	4.14.3

2.0 REFERENCES

See Appendix A.

3.0 TERMS, DEFINITIONS and ACRONYMS

The relevant definitions from ISO/IEC Guide 2, ANSI/ASQC E-4 (1994), and the International vocabulary of basic and general terms in metrology (VIM) are applicable; the most relevant being quoted in Appendix A, "Glossary," of NELAC, Chapter 1, together with further definitions applicable for the purposes of this document. General definitions related to quality are given in ISO 8402, whereas ISO/IEC Guide 2 gives

definitions specifically related to standardization, certification, and laboratory accreditation. Where different definitions are given in ISO 8402, the definitions in ISO/IEC Guide 2 and VIM are preferred.

3.1 Terms and Definitions

Note: The following is a compilation of DOE definitions as well as applicable NELAC and Multi-Agency Radiological Laboratory Analytical Protocol (MARLAP) definitions. The DOE definitions are included in gray boxes. The NELAC definitions are extracted directly from the Appendix A, "Glossary," of NELAC, Chapter 1.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

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. (QAMS)

The characteristics of an analysis or determination that ensures that both the bias and precision of the resulting quantity will remain within specified limits. (ANSI)

Activity, of radionuclides: The expected number of spontaneous nuclear decays (transformations) in unit time from a specified energy state (excluding prompt decays from a lower nuclear level) for a given amount of a radionuclide. Its standard unit (SI) is the Becquerel (Bq), where one Bq equals one decay per second. Activity has often been expressed in curies (Ci), where 3.7×10^{10} Bq equals 1 Ci, exactly. (ANSI)

Activity coefficient (γ): (1) A fractional number that represents the extent that ions deviate from ideal behavior in solution. The activity coefficient multiplied times the molal concentration of ions in solution equals the chemical activity: $a = \gamma * c$, where $\gamma \leq 1$; thus the activity coefficient is a correction factor applied to molal concentrations. At infinite dilution where behavior is ideal, $\gamma = 1.0$, but it decreases as the concentration of ions increases. (2) The ratio of effective (apparent) concentration of an ion in solution to the stoichiometric concentration, $\gamma = a/c$. (MARLAP)

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. (NELAC)

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 NELAC). (NELAC)

Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Assessment Team: the group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

Assessor: one who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)

Audit: a systematic evaluation to determine the conformance to quantitative and qualitative specification of some operational function or activity. (EPA-QAD)

Background: Ambient signal response recorded by measurement instruments that are independent of radioactivity contributed by the radionuclides being measured in the sample. (ANSI)

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 to 20 environmental samples of the same NELAC-defined 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 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 environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Batch (for NDA): (DRAFT) or NDA a measurement batch is a group of measurements that is analyzed with the same instrument and analytical software composed of 1 to 20 measurements with the maximum time between the first and last measurement.

Analytical Batch: a prep batch or part of a prep batch that is analyzed with the same instrumentation, method sequence, and lots of reagents, and with the manipulations common to each sample within the same time period or in continuous sequential time periods. It is preferable that the analytical batch be the prep batch. If equipment restrictions limit the number of samples in any

particular step, the samples in the batch are processed continuously and consecutively until the entire batch is completed.

becquerel (Bq): The standard unit (SI) is the Becquerel (Bq), where one Bq equals one decay per second. Activity has often been expressed in curies (Ci), where 3.7 X 10^{10} Bq equals 1 Ci, exactly. (ANSI)

Bias: the deviation of a single measured value of a random variable from a corresponding expected value or fixed mean deviation from the expected value that remains constant over replicated measurements within the statistical precision of the measurement. (Synonyms: deterministic error, fixed error, systematic error)

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:

Equipment Blank: a sample of analyte-free media that has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

Field Blank: blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

The field blank is used to indicate the presence of contamination due to sample collection and handling.

Instrument Blank: a clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

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. (NELAC)

Reagent Blank: (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Refrigerator Blank: a blank solution that is stored adjacent to samples in a refrigerated storage area. This type of blank is used for determining storage contamination of volatile organics.

Trip Blank: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples. (EPA)

Blank Spike: See Laboratory Control Sample (LCS)

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibration: 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. (VIM, Section 6.11)

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 test 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 graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method: a defined technical procedure for performing a calibration. (NELAC)

Calibration Standard (Source): a substance or reference material used to calibrate an instrument. (QAMS)

Carrier: (1) A stable isotopic form of a tracer element or nonisotopic material added to effectively increase the quantity of a tracer element during radiochemical procedures, ensuring conventional behavior of the element in solution. (2) A substance in appreciable amount that, when associated with a tracer of a specified substance, will carry the tracer with it through a chemical or physical process, or prevent the tracer from undergoing nonspecific processes due to its low concentration (IUPAC, 1995). A stable isotope of a radionuclide (usually the analyte) added to increase the total amount of that element so that a measurable mass of the element is present. (MARLAP)

Certified Reference Material (CRM): a reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body. (ISO Guide 30-2.2)

Chain of Custody (COC) 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; collector; time of collection; preservation; and requested analyses. (NELAC)

Check source: a radioactive source, not necessarily traceable to a national standards body such as NIST in the USA that is used to confirm the continuing satisfactory operation of an instrument. (ASTM)

Clean Air Act (CAA): the enabling legislation in 42 U.S.C. 7401 *et seq*., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, and to monitor and enforce them.

Combined Standard Uncertainty (Total Propagated Uncertainty): An estimate or approximation of the error associated with a measured value by propagation of individual uncertainties. The Combined Standard Uncertainty (CSU) shall include both systematic and random error. The CSU is, by ISO and MARLAP definition, 1-sigma.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 *et seq*., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 *et seq*., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

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. (NELAC)

Conformance: an affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Control Chart: A graphical representation of data taken from a repetitive measurement or process. Control charts may be developed for various characteristics, (e.g., mean, standard deviation, range, etc.) of the data.

"A control chart has two basic uses: (1) as a tool to judge if a process was in control, and (2) as an aid in achieving and maintaining statistical control. For applications related to radiation detection instrumentation or radiochemical processes, the mean (center line) value of a historical characteristic (e.g., mean detector response), subsequent data values and control limits placed symmetrically above and below the center line are displayed on a control chart." (MARLAP)

Control limit: Predetermined values, usually plotted on a control chart, which define the acceptable range of the monitored variable. There can be both upper and lower limits; however, when changes in only one direction are of concern, only one limit is necessary. When a measured value exceeds the control limits, the measurement process must be stopped, the problem investigated, and corrective action(s) taken. (MARLAP)

Correction factor: A numerical factor by which the result of an uncorrected result of a measurement is multiplied to compensate for a systematic effect. (MARLAP)

Corrective Action: the action taken to eliminate the cause(s of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Count rate: The rate at which detector pulses are being registered in a selected voltage interval. The unit is reciprocal seconds (i.e., s^{-1}). Generally the count rate is uncorrected for detector efficiency. The count rate divided by the detector efficiency for a specific particle and energy will yield the source activity.

Count time: The time interval for the counting of a sample or source by a radiation detector. Depending upon the context used, this can be either the

"clock" time (the entire period required to count the sample), or "live" time (the period during which the detector is actually counting). Live time is always less than or equal to clock time. (MARLAP)

Counting efficiency: The ratio of the count rate to the disintegration rate, usually expressed as a percentage (*E=(R/A) X 100).* The counting efficiency may be a function of many variables, such as radiation energy, source composition, and source or detector geometry.

Counting uncertainty: Component of measurement uncertainty caused by the random nature of radioactive decay and radiation counting. (MARLAP)

Critical level: See Decision Level.

Critical value (S_c): See Decision Level.

Cross-contamination: Cross-contamination occurs when material in the sample is inadvertently transferred to an uncontaminated sample, which can result from using contaminated equipment and chemicals. Cross contamination may also occur from spills, as well as airborne dust of contaminated materials.

Crosstalk: The fraction of all recorded pulses from alpha particles that are recorded in the beta channel due to degradation in their pulse height or the fraction or all recorded pulses from beta particles that are recorded in the alpha channel due to pulse pileup or other phenomenon. Crosstalk is often used to indicate that the method of discrimination is pulse-height discrimination. (ANSI)

Curies (Ci): The traditional unit used to express the activity (amount) of radioactive material. The SI unit for activity is the Becquerel (Bq).

1 curie (Ci)	=	2.22×10^{12} disintegrations/minute
1 millicurie (mCi)	=	2.22×10^{9} disintegrations/minute
1 microcurie (µCi)	=	2.22×10^{6} disintegrations/minute
1 picocurie (pCi)	=	2.22 disintegrations/minute
1 becquerel (Bq)	=	1 disintegrations/second

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Quality Objective (DQO) (DRAFT for NDA): DQOs are qualitative and quantitative statements specified to ensure that data of known and appropriate

quality are obtained. The DQO process is a series of planning steps, typically conducted during the assessment and investigation, designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate. The DQO process involves a logical, step-by-step procedure for determining which of the complex issues affecting a site are the most relevant to planning a site investigation before any data are collected.

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Decision Level (DL): In the context of analyte detection, the minimum measured value (e.g., of the instrument signal or the analyte concentration) required to give confidence that a positive (nonzero) amount of analyte is present in the material analyzed. The DL is sometimes called the critical level (Lc) or critical value (MARLAP). It is the quantity of analyte at or above which an *a posteriori* decision is made that a positive quantity of the analyte is present. For this document, the probability of a Type I error (probability of erroneously reporting a detectable nuclide in an appropriate blank or sample) is assumed to be set at 0.05; however, other confidence levels may be dictated by the MQOs.

Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)

Design: method and software development.

Detection Limit: the lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Dilution Factor (DF): The factor by which the dilution level of the sample differs from that of a predefined method blank. The method blank is prepared within the prescribed parameters of the method and has a dilution factor of one. The dilution factor does not include percent moisture.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicates: identical splits of individual samples that are analyzed by the laboratory to test for method reproducibility in a given matrix.

Duplicate Error Ration (DER): Refer to Section D-32 for calculation criteria.

Energy Calibration: The correlation of the multi-channel analyzer (MCA) channel number to decay photon energy, obtained from the location of peaks from known radioactive standards.

Error: An estimation of the analytical measurement uncertainty, expressed as an error term with a specific analytical result, or as a typical value for a specific analytical technique.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): the enabling legislation under 7 U.S.C. 135 *et seq*., as amended, that empowers the EPA to register insecticides, fungicides and rodenticides. (NELAC)

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C. 1251 *et seq*., Public Law 92-59986 Stat. 816, that empowers EPA to set discharge limitations, write discharge permits, monitor and bring enforcement action for non-compliance. (NELAC)

Field Measurement: The determination of physical, biological or radiological properties or chemical constituents that are measured onsite, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory. (NELAC)

Half-life ($T_{1/2}$): The time required for one-half of the atoms of a particular radionuclide to disintegrate or undergo nuclear transformation.

Holding Times: The duration between date/time of sample collection and dates/times of sample preparation (extraction/distillation) and/or analysis.

Inspection: an activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Instrument Detection Limit (IDL): The concentration of an analyte that produces an output signal twice the root mean square of the background noise, or the parameter determined by multiplying by three the standard deviation obtained for the analysis of a standard solution at a concentration of three to five times the desired IDL on three nonconsecutive days with seven consecutive measurements per day. IDL is only required for metals analysis.

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. (NELAC)

International Standard of Units (SI): the coherent system of units adopted and recommended by the General Conference of Weights and Measures. (CCGPM)(VIM, Section 1.12)

Isotope: One of two or more forms of a single element. The atoms of each isotope have the same number of protons, but different numbers of neutrons in their nuclei; thus, isotopes have the same atomic number but differ in atomic mass. Isotopes may be stable (not spontaneously decaying) or unstable (spontaneously decaying, emitting ionizing radiation).

Key Peak: A spectral peak used for identification or quantification of an isotope:

Laboratory: a body that calibrates and/or tests. (ISO 17025)

Laboratory Control Sample (LCS) (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. 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. (NELAC)

The spiking standard must be from a different source or lot than the calibration standards.

Laboratory Duplicate: aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Laboratory Information Management System (LIMS): The automated information system used at a laboratory to collect and track data regarding sample analysis, laboratory QC, operability, final result calculation, report generation, etc.

Legal COC Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the client. These protocols include the use of a COC 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.** (NELAC)

Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte-and matrix-specific and may be laboratory dependent.

The LOD is equivalent to the MDL.

Limit of Quantification (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.

The LOQ is equivalent to the Practical Quantitation Limit (PQL).

For analyte calibration curves of more than two points, the lowest point above LOD determines the LOQ. The LOQ shall be no less than three times the LOD.

Matrix: the substrate of a test sample, including:

Field of Accreditation Matrix: These matrix definitions shall be used when accrediting a laboratory.

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

Non-potable Water: any aqueous sample excluded from the definition of Drinking Water matrix. Includes surface water, groundwater, effluents, water treatment chemicals, and Toxicity Characteristic Leaching Procedure (TCLP) or other extracts.

Solid and Chemical Materials: includes soils, sediments, sludges, products and by-products of an industrial process that results in a matrix not previously defined.

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

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 sorbent tube, impinger solution, filter, or other device. (NELAC)

Quality System Matrix: These matrix definitions are an expansion of the field of accreditation matrices and shall be used for purposes of batch and QC requirements (See Appendix D). These matrix distinctions shall be used:

Aqueous: any aqueous sample excluded from the definition of Drinking water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

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

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

Non-aqueous Liquid: any organic liquid with less than 15 percent settleable solids.

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

Solids: Includes soils, sediments, sludges, and other matrices with greater than 15 percent settleable solids.

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

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 sorbent tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate 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. (QAMS)

Matrix spikes are not required for radiochemical analyses if an isotopic tracer or chemical carrier is used in the analysis to determine chemical recovery (yield) for the chemical separation and sample mounting SOPs. Matrix spikes are not required for Gross Alpha, Gross Beta, or Gamma Analysis.

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

Measurement Control Sample (MCS) (DRAFT for NDA): A sample matrix, free of analytes of interest, spiked with known values of analytes or materials containing known and verified amounts of analytes.

Measurement Quality Objectives (MQOs): the desired sensitivity, range, precision, and bias of a measurement.

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

Method Detection Limit (MDL): see LOD

MDLs are determined and reported by the laboratory.

Minimum Detectable Amount (MDA): The smallest amount (activity or mass) of an analyte in a sample that will be detected with a probability β of non-detection (Type II error) while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). For the purposes of this document, the alpha (α) and beta β) probabilities are both set at 0.05 unless otherwise specified. MDA is not comparable to MDL since both Type I and Type II errors are considered.

Minimum Detectable Concentration (MDC): The MDA expressed in units of concentration. (ANSI)

Mixed Waste: Waste containing both radioactive and hazardous components as defined by the Atomic Energy Act of 1954 and the Resource Conservation Recovery Act (RCRA) respectively.

National Environmental Laboratory Accreditation Conference (NELAC): a voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP): the overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

National Institute of Standards and Technology (NIST): an agency of the U.S. Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable Performance Evaluation (PE) to those laboratories testing drinking water and wastewater. (NIST)

National Voluntary Laboratory Accreditation Program (NVLAP): a program administered by NIST that is used by providers of PE to gain accreditation for all compounds/matrices for which NVLAP accreditation is available, and for which the provider intends to provide NELAP PE samples. (NELAC)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Nuclide: A species of atom characterized by its mass number, atomic number, and nuclear energy state, providing that the mean half-life in that state is long enough to be observable. (IUPAC, 1995)

Operator Aid: A technical posting, other than formal procedures, rules, instructions, etc., that assists workers in accomplishing specific tasks and are not required to be posted or displayed by any organization or procedure. All operator aids must be controlled by the facility.

Performance Audit: the routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Evaluation (PE) 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. (NELAC)

PE Study Provider: any person, private party, or government entity that meets stringent criteria to produce and distribute PE samples, evaluate study results against published performance criteria, and report the results to the laboratories.

PE Test Sample: a sample, the composition of which is unknown to the analyst that is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Positive Control: measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

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. (NELAC)

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Probability: "A real number in the scale 0 to 1 attached to a random event" (ISO, 1993b). The total number of occurrences of a selection/event "a", divided

by the total number of occurrences (a) plus the number of failures of those occurrences "b" (i.e., total possible outcomes).

This leads to the basic formula: P = a /(a+b).

Procedure: specified way to carry out an activity or a process. Procedures can be documented or not. (ISO 9000:2000 and Note 1)

For the purpose of this QSAS, all procedures shall be documented.

Proficiency Testing: See Performance Evaluation (PE).

Progeny: the product resulting from the radioactive disintegration or nuclear transformation of its parent radionuclide. (MARLAP)

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

Quality Assurance (QA): an integrated system of activities involving planning, QC, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP): a formal document describing the detailed QC procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample: a sample used to assess the performance of all or a portion of the measurement system. QC samples may be CRMs, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

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. (NELAC)

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. (ANSI/ASQC E-4 1994)

Quench: any factor that reduces the efficiency of the energy transfer or causes the absorption of photons (light) during liquid scintillation counting.

Radioactive: exhibiting radioactivity or containing radionuclides. (MARLAP)

Radioactive decay: process by which a spontaneous change in nuclear state takes place. This process is accompanied by the emission of energy and subatomic particles.

Radioactive waste: solid, liquid, or gaseous materials containing radionuclides regulated under the Atomic Energy Act of 1954 as amended, and of negligible economic value considering recovery costs.

Radioactivity: spontaneous emission of radiation, either directly from unstable atomic nuclei or as a consequence of a nuclear reaction.

Radiochemical Analysis: the analysis of a sample matrix for its radionuclide content, both qualitatively and quantitatively. (MARLAP)

Radionuclide: a nuclide that is radioactive (capable of undergoing radioactive decay). (MARLAP)

Radiation Yield: the amount of radiation of the type being measured that is produced per each disintegration that occurs. For gamma spectrometry, this is commonly called "gamma abundance."

Random Error: a result of a measurement minus the mean that would result from an infinite number of measurements of the same measure and carried out under repeatability conditions (ISO 1993a) (MARLAP)

Raw data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm, or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes that have been transcribed verbatim, data and verified accurate by signature), the exact copy of exact transcript may be submitted. (EPA-QAD)

Recovery: the ratio of the amount of analyte measured in a spiked or laboratory control sample, to the amount of analyte added, usually expressed as a percentage. For a matrix spike, the measured amount of analyte is first

decreased by the measured amount of analyte in the sample that was present before spiking. Compare with yield. (MARLAP)

Reference Material: a material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30, Section 2.1)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM, Section 6.08)

Reference Toxicant: the toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results (see Appendix D, Section 2.d.1.a.1). (NELAC)

Region of Interest (ROI): in radiochemical analysis, the Multi-channel Analyzer region defining the isotope or analysis parameter of interest displayed in terms of energy or channels.

Relative bias: the quotient of the bias divided by the expected value.

Relative Standard Uncertainty: the ratio of the standard uncertainty of a measured result to the result itself. The relative standard uncertainty of x may be denoted by $u_r(x)$. (MARLAP)

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.

DOE considers the term replicate to mean three or more sub-samples.

Reproducibility (of results of measurement): the closeness of the agreement between the results of measurements of the same measure and carried out under changed conditions of measurement. A valid statement of reproducibility requires specification of the conditions changed. The changed conditions may include principle of measurement, method of measurement, observer (or analyst), measuring instrument, reference standard, location, conditions of use, and time. Reproducibility may be expressed quantitatively in terms of the dispersion characteristics of the results. Results are usually understood to be corrected results. (Adapted from ISO, 1993a)

Required Detection Limit (RDL): A contractually specified detection limit that, under typical analytical circumstances, shall be achieved.

Requirement: denotes a mandatory specification; often designated by the term "shall." (NELAC)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 U.S.C. 321 *et seq*., (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA): the enabling legislation, 42 U.S.C. 300f et. Seq., (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample: A single container or series of containers identified by a unique number comprised of material drawn from a single location or composite of locations during a fixed period representative of that location(s) and time period(s) for the purpose of analytical testing or physical evaluation.

Sample tracking: procedures employed to record the possession of the samples from the time of sampling until analysis, reporting, and archiving. These procedures include the use of a COC form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples. (NELAC)

Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

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

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification, so long as the requirement is fulfilled. (ANSI)

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Sigma (σ): the symbol σ and the term "sigma" are properly used to denote a true standard deviation.

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other QC purposes. (NELAC)

Standard: the document describing the elements of laboratory accreditation, which has been developed and established within the consensus principles of NELAC, and that meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Deviation: the square root of a variance of a random variable. The variance is a measure of the variation of the observations within a measurement set. The standard deviation is often estimated using a set of measurements of the random variable. The standard deviation has the same units as the measured quantity and therefore, is particularly convenient when describing the variability of the measured quantity. (ANSI)

Standard Operating Procedure (SOP): a written document that details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and that is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standard Reference Material (SRM): a CRM produced by the NIST or other equivalent organization and characterized for absolute content, independent of analytical method. (QAMS)

Standard Uncertainty: the standard deviation of a mean of a set of measurements. The Standard Uncertainty reduces to the standard deviation of the measurement when there is only one determination. (ANSI)

Statistical Minimum Significant Difference (SMSD): the minimum difference between the control and a test concentration that is statistically significant, a measure of test sensitivity or power. The power of a test depends in part on the number of replicates per concentration, the significance level selected, e.g., 0.05, and the type of statistical analysis. If the variability remains constant, the sensitivity of the test increases as the number of replicates is increased. (NELAC)

Sub-sample: (1) A portion of a sample removed for testing. (2) To remove a portion of a sample for testing. The sub-sample shall be representative of the sample being sub-sampled.

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, QA/QC duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for QC purposes. (QAMS)

Suspension: temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed six months, to allow the laboratory time to correct deficiencies or areas of non-compliance with the NELAC standards. (NELAC)

Systematic error: An error component that produces a fixed bias in the underlying expected value of a determination, from measurement to measurement. (ANSI)

Technical requirements: detailed instructions identifying the specific analysis or parameter desired and the requested regulatory method of analysis. Any deviation(s) from the regulatory methods regarding preparation and analysis protocol(s) and or reporting will be defined and approved.

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

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.4)

Test Method: an adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

Testing Laboratory: a laboratory that performs tests. (ISO/IEC Guide 2-12.4)

Test Sensitivity/Power: the minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis (see Appendix D, Section 2.4.a)(NELAC)

Total Propagated Uncertainty: See Combined Standard Uncertainty.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 et seq., (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the U.S. for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM, Section 6.12) Demonstrated lineage of measurement process quality to the national physical standards. A progeny that has been removed from the parent(s) in the decay chain in which it was produced.

Tracer: a radioactive isotope that chemically mimics and does not interfere with the target analyte through radiochemical separations. Isotopic tracers are typically radioactive materials (e.g., Pu-242, Sr-85). They are added to samples to determine the overall chemical yield for the analytical preparation steps. When tracers are used, each sample (including any batch associated QC samples) shall be spiked separately with the same materials and individual sample yields will be determined. The tracer shall be added to the sample at the very beginning of the sample preparation. For solid samples, the tracer shall be added after grinding, sieving, etc. but prior to any muffling or dissolution of the sample.

Unsupported Nuclide Material: a progeny that has been removed from the parent(s) in the decay chain in which it was produced. A reference material usually prepared by a single laboratory for its own use as a calibration standard, as a control standard, or for the qualification of a measurement method.

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (U.S.-EPA)

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

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

> Note: 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.
>
> The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: a well-defined group of analysts that, together, perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Working Range: The difference between the LOQ and the upper limit of measurement system calibration.

Working Reference Material: A reference material usually prepared by a single laboratory for its own use as a calibration standard, a control standard, or for the qualification of a measurement method. (ASTM C1128)

3.2 Acronyms

%R	percent recovery
^{o}C	degrees Celsius
AEA	Atomic Energy Act
AHERA	Asbestos Hazard Emergency Response Act
AIHA	American Industrial Hygiene Association
ANSI	American National Standards Institute
ARAR	applicable or relevant and appropriate requirement (CERCLA/Superfund)
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
BOA	Basic Ordering Agreement
Bq	becquerel
CAA	Clean Air Act
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (“Superfund”)
CFR	*Code of Federal Regulations*
CHP	Chemical Hygiene Plan
Ci	Curie

CLP	Contract Laboratory Program
COC	chain of custody
cpm	counts per minute
cps	counts per second
CRM	certified reference material
CSU	combined standard uncertainty
CV	coefficient of variation
CWA	Clean Water Act
DER	Duplicate Error Ratio
DF	Dilution Factor
DO	dissolved oxygen
DOC	demonstration of capability
DOE	U.S. Department of Energy
DOECAP	DOE Consolidated Audit Program
DOT	U.S. Department of Transportation
dpm	disintegrations per minute
DQA	data quality assessment
DQO	data quality objective
EC25	electrical conductivity
ECp	effective concentration point estimate
EDD	electronic data deliverable
EM	Environmental Management
EPA	Environmental Protection Agency

FAR	*Federal Acquisition Regulations*, CFR Title 48
FIFRA	Federal Insecticide, Fungicide and Rodenticide Act
FWHM	full width half maximum
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
GM	Geiger-Mueller detector
GOCO	government owned contractor operated
GOGO	government owned government operated
GPC	Gas-flow Proportional Counter
h	hour
HPGe	high-purity germanium
HPLC	high-pressure liquid chromatography; high performance liquid chromatography
IAEA	International Atomic Energy Agency
ICp	inhibition concentration point estimate
ICP-MS	inductively coupled plasma-mass spectrometry
IDL	Instrument Detection Limit
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
keV	kilo electron volts
KPA	kinetic phosphorescence analysis
LAN	local area network
LC 50	lethal concentration 50

LCL	lower control limit
LCS	laboratory control sample
LIMS	Laboratory Information Management System
LLD	lower level of detection
LOD	Limit of Detection (MDL)
LLQ	Lower Level of Quantitation
LOQ	Limit of Quantitation (PQL)
LSC	liquid scintillation counter
MAPEP	Mixed Analyte Performance Evaluation Program
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocol
MCE	mixed-cellulose ester
MCL	maximum contaminant limit
MDA	minimum detectable activity
MDC	minimum detectable concentration
MDL	method detection limit
ME	marginal exceedance
MeV	mega electron volts
MFL	million fibers per liter
MQC	minimum quantifiable concentration
MQO	measurement quality objective
MS	matrix spike
MSA	Method of Standard Additions
MSD	matrix spike duplicate

NDA	nondestructive assay
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIOSH	National Institute of Safety and Health
NIST	National Institute of Standards and Technology
NOAEC	no observed adverse effects concentration
NOEC	no observed effects concentration
NVLAP	National Voluntary Laboratory Accreditation Program
PARCC	precision, accuracy, representativeness, completeness, and comparability
PBMS	Performance Based Measurement Systems
PCB	polychlorinated biphenyl
PCM	phase contrast microscopy
pCi	picocurie
PE	Performance Evaluation
PEOB	Performance Evaluation Oversight Body
PEPA	Performance Evaluation Provider Accreditor
PLM	polarized light microscopy
PT	Proficiency Testing (See PE)
QA	Quality Assurance
QAD	Quality Assurance Division
QAMS	Quality Assurance Management Systems
QAPP	Quality Assurance Project Plan
QB	quarterly blind

QC	Quality Control
QSAS	Quality Systems for Analytical Services
RCRA	Resource Conservation and Recovery Act
RDG	Reporting Delivery Group
RDL	required detection limit
RESL	Radiological and Environmental Science Laboratory
RI/FS	remedial investigation/feasibility study
ROI	region of interest
RPD	relative percent difference
RPP	Radiation Protection Plan
RSD	relative standard deviation
RSO	radiation safety officer
S_c	critical value
SAED	selected area electron diffraction
SAP	sampling and analysis plan
SDWA	Safe Drinking Water Act
SI	international system of units
SMO	Sample Management Office
SMSD	statistical minimum significant difference
SOP	standard operating procedure
SOW	statement of work
SQC	statistical quality control
SRM	standard reference material

SRT	standard reference toxicants
$T_{1/2}$	half-life
TAC	test acceptance criteria
TAT	turnaround time
TCLP	toxicity characteristic leaching procedure
TDS	total dissolved solids
TEDE	total effective dose equivalent
TEM	transmission electron microscopy
TIC	tentatively identified compounds
TIMS	thermal ionization mass spectrometry
TLD	thermoluminescent dosimeter
TPU	total propagated uncertainty
TQM	Total Quality Management
TSCA	Toxic Substances Control Act
TSDF	treatment, storage, and disposal facility
TSS	total suspended solids
$\mu_c(y)$	combined standard uncertainty
µohms	resistivity unit of measure
WET	whole effluent toxicity
WMP	Waste Management Plan
WP	water pollution

4.0 MANAGEMENT REQUIREMENTS

Note: DOE requirements are identified in gray boxes. In cases where DOE requirements differ from the NELAC requirements, DOE requirements supersede the NELAC requirements.

4.1 Organization

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 environmental testing activities in such a way as to meet the requirements of this document and to satisfy the needs of the client, the regulatory authorities or organizations providing recognition.

4.1.3 The laboratory 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 environmental testing, the responsibilities of key personnel in the organization that have an involvement or influence on the environmental testing activities of the laboratory shall be defined in order to identify potential conflicts of interest.

a) Where a laboratory is part of a larger organization, the organizational arrangements shall 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 document.

b) The laboratory must be able to demonstrate that it is impartial and that it and its personnel are free from any undue commercial, financial and other pressures that might influence their technical judgment. Environmental testing laboratories shall not engage in any activities that may endanger the trust in its independence of judgment and integrity in relation to its environmental testing activities

4.1.5 The laboratory shall:

a) have managerial and technical personnel with the authority and resources needed to carry out their duties and to identify the occurrence of departures from the quality system or from the procedures for performing environmental tests, and to initiate actions to prevent or minimize such departures (see also Section 5.2);

b) have processes 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 clients' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results;

Note: The policy and procedures to ensure the protection of clients' confidential information and proprietary rights may not apply to in-house laboratories.

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 environmental tests;

Note: Documentation shall include a clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured.

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

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

i) The technical director(s) (however named) shall meet the requirements specified in the Accreditation Process and shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented.

j) 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 quality system is implemented and followed at all times; the quality manager (and/or his/her designee(s) shall:

    1) have direct access to the highest level of management at which decisions are made on laboratory policy or resources; and, where staffing is limited, the quality manager may also be the technical director or deputy technical director;

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

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

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

5) have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAC;

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

7) arrange for or conduct internal audits as per the requirements of Section 4.13 annually; and,

8) notify laboratory management of deficiencies in the quality system and monitor corrective action.

4.1 DOE-1

The QA Officer or designee shall periodically review control charts at a specified frequency for out-of-control conditions and initiate appropriate corrective actions. Data analysis software may also be used for the statistical evaluation of data for trends and biases.

Statistical control charts are defined as a graphical representation of data taken from a repetitive measurement or process. Control charts may be developed for various characteristics, (e.g., mean, standard deviation, range, etc.) of the data.

The QA officer shall be empowered to stop unsatisfactory work, or prevent the reporting of results generated from an out-of-control measurement system.

k) appoint deputies for key managerial personnel, including the technical director(s) and/or quality manager;

l) for purposes of qualifying for and maintaining accreditation, participate in a PE program.

4.1 DOE-2

DOE PE requirements are outlined in gray box 5.9 DOE-1.

4.2 Quality System

4.2.1 The laboratory shall establish, implement and maintain a quality system based on the required elements contained in this Chapter and appropriate to the type, range and volume of environmental testing activities it undertakes. The laboratory shall document its policies, systems, programs, procedures and instructions to the extent necessary to ensure the quality of the environmental test 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 quality system policies and objectives shall be defined and documented in a quality manual (however named). The overall objectives shall be documented in a quality policy statement. The quality policy statement shall be issued under the authority of the chief executive. It shall include at least the following:

a) the laboratory management's commitment to good professional practice and to the quality of its environmental testing in servicing its clients; the laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.
b) the management's statement of the laboratory's standard of service;
c) a requirement that all personnel concerned with environmental testing activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work; and
d) the laboratory management's commitment to compliance with this document.

4.2.3 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 quality system.

The quality manual, and related quality documentation, shall state the laboratory's policies and operational procedures established in order to meet the requirements of this document.

Where a laboratory's quality manual contains the necessary requirements, a separate SOP or policy is not required.

The quality manual shall list on the title page: a document title; the laboratory's full name and address; the name, address (if different), and telephone number of individual(s) responsible for the laboratory; the name of the quality manager (however named); the identification of all major organizational units which are to be covered by this quality manual and the effective date of the version;

The quality manual and related quality documentation shall also contain:

a) a quality policy statement, including objectives and commitments, by top management (see Section 4.2.2);

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

c) the relationship between management, technical operations, support services and the quality system;

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

4.2 DOE-1

Note: The laboratory shall establish a reasonable minimum frequency for reviewing, updating, and distributing current revisions of controlled documents and procedures (e.g., SOPs).

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

f) identification of the laboratory's approved signatories (Note: at a minimum, the title page of the quality manual must have the signed and dated concurrence, (with appropriate titles) of all responsible parties including the quality manager(s), technical director(s), and the agent who is in charge of all laboratory activities, such as the laboratory director or laboratory manager);

g) the laboratory's procedures for achieving traceability of measurements;

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

4.2 DOE-2

The QA Plan includes a listing of certifications and accreditations and a list of all test methods under which the laboratory performs its accredited testing. If this list is not a part of the QA plan, the QA plan should include a reference to its location.

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

j) reference to the calibration and/or verification test procedures used;

k) procedures for handling submitted samples;

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

m) reference to procedures for calibration, verification and maintenance of equipment;

n) reference to verification practices, which may include interlaboratory comparisons, PE programs, use of reference materials and internal QC schemes;

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

p) the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

q) procedures for dealing with complaints;

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

s) procedures for audits and data review;

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

u) reference to procedures for reporting analytical results; and,

v) a Table of Contents, and applicable lists of references, glossaries, and appendices.

4.2 DOE-3

The quality manual shall also include or reference SOPs for:

a) Procurement of standards;

b) Data management including LIMS, software validation, verification, and control;
c) Manual entry of raw data from analytical measurements that are not directly interfaced to the LIMS;
d) Verification and documentation of the accuracy of data that are manually entered into LIMS;
e) Making changes to electronic data (including establishing the requirements for a hardcopy or electronic log to document all changes to electronic data that affect data quality);
f) How electronic data are processed and maintained by LIMS;
g) How electronic data are reported by LIMS;
h) Retention of electronic data, documentation and records pertaining to LIMS;
i) Ensuring that data review is inclusive of all quality-related steps in the analytical process, including sample preparation, dilution calculations, chromatography evaluation, and spectral interpretations. SOP shall require that documentation of data review will be maintained and available for review;
j) Health and safety;
k) Chemical Hygiene Plan, and
l) Materials Management

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

4.2.5 The quality manual shall be maintained current under the responsibility of the quality manager.

4.2.6 The laboratory shall establish and maintain data integrity procedures. These procedures shall be defined in detail within the quality manual. There are four required elements within a data integrity system. These are:

a) data Integrity training,
b) signed data integrity documentation for all laboratory employees,
c) in-depth, periodic monitoring of data integrity, and
d) data integrity procedure documentation.

The data integrity procedures shall be signed and dated by senior management. These procedures and the associated implementation records shall be properly maintained and made available for assessor review. The data integrity procedures shall be annually reviewed and updated by management.

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

4.2.8 In instances of ethical concern, the mechanism shall include a process whereby laboratory management are to be informed of the need for any further detailed investigation.

4.3 Document Control

4.3.1 General

The laboratory shall establish and maintain procedures to control all documents that form part of its quality system (internally generated or from external sources). Documents include policy statements, procedures, specifications, calibration tables, charts, textbooks, 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.

4.3 DOE-1

"Operator aids" shall be included in the document control process.

Operator aid is defined as a technical posting, other than formal procedures, rules, instructions, etc., that assists workers in accomplishing specific tasks and are not required to be posted or displayed by any organization or procedure. All operator aids must be controlled by the facility.

The control of data related to environmental testing is covered in Section 5.4.7. The control of records is covered in Section 4.12.

4.3.2 Document Approval and Issue

4.3.2.1 All documents issued to personnel in the laboratory as part of the quality 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 quality system shall be established and 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 Quality system documents generated by the laboratory shall be uniquely identified. Such identification shall include the date of issue and/or revision identification, page numbering, 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 documentation 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, initialed and dated. A revised document shall be formally re-issued as soon as practicable.

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

4.4 Review of Requests, Tenders and Contracts

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 environmental testing shall ensure that:

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

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

The purpose of this review of capability is to 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 environmental tests in question. The review may encompass results of earlier participation in interlaboratory comparisons or PE and/or the running of trial environmental test programs using samples or

items of known value in order to determine uncertainties of measurement, detection limits, confidence limits, or other essential QC requirements. The current accreditation status of the laboratory must also be reviewed. The laboratory must inform the client of the results of this review if it indicates any potential conflict, deficiency, lack of appropriate accreditation status, or inability on the laboratory's part to complete the client's work.

c) the appropriate environmental test method is selected and capable of meeting the clients' requirements (see Section 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 to the client.

A contract may be any written or oral agreement to provide a client with environmental testing services.

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

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 inquiry stage or on granting of the contract for ongoing routine work performed under a general agreement with the client, provided that the client's requirements remain unchanged. For new, complex or advanced environmental testing 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 client 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. Suspension of accreditation, revocation of accreditation, or voluntary withdrawal of accreditation must be reported to the client.

4.5 Subcontracting of Environmental Tests

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 laboratory accredited under

NELAP 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 and non-NELAP accredited work shall be clearly identified.

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

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

4.5.4 The laboratory shall maintain a register of all subclients that it uses for environmental tests and a record of the evidence of compliance with Section 4.5.1.

4.5 DOE-1

The laboratory shall not use any sub-tier laboratories or subclients (including those possessing the same or similar corporate name) for performance of work under this specification without written approval from the Procurement Representative. The laboratory using the sub-tier laboratory or subclient shall document and is responsible for ensuring that such subclient meets all of the requirements of this specification, including being available for client inspections and audits.

Some clients may not allow any subcontracting to third party (sub-tier) laboratories. If this is the case, then this will be specifically noted in site-specific contracts via Contracts, Task Orders, Laboratory Delivery Orders, etc.

4.6 Purchasing Services and Supplies

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 environmental tests. Procedures shall exist for the purchase, reception and storage of reagents and laboratory consumable materials relevant for the environmental tests.

4.6 DOE-1

The laboratory policy for the selection and purchasing of services and/or supplies shall include requirements for training of laboratory personnel involved in the procurement process.

Contractual requirements for contracted items and services must include one or more of the following:

- Source evaluation and selection (pre-performance/pre-award survey);
- Source verification;
- Audit; and/or;
- Examination of items or services before use.

4.6.2 The laboratory shall ensure that purchased supplies and reagents and consumable materials that affect the quality of environmental tests are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the environmental tests 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.

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

4.6 DOE-2

When there are indications that subcontractors knowingly supplied items or services of substandard quality, this information shall be forwarded to appropriate management for action and notification to the affected clients.

4.7 Service to the Client

The laboratory shall afford clients or their representatives' cooperation to clarify the client's request(s) and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients.

4.8 Complaints

The laboratory shall have a policy and procedure for the resolution of complaints received from clients or other parties. Records shall be maintained of all complaints and of the investigations and corrective actions taken by the laboratory (see also Section 4.10).

4.8 DOE-1

The laboratory shall immediately notify all affected clients of potential data quality issues. Corrective actions taken to resolve the issue shall be submitted to the client in a timely and responsive manner.

4.9 Control of Nonconforming Environmental Testing Work

4.9.1 The laboratory shall have a policy and procedures that shall be implemented when any aspect of its environmental testing work, or the results of this work, do not conform to its own procedures or the agreed requirements of the client. 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, as necessary) are defined and taken when nonconforming work is identified;

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

c) corrective actions are taken immediately, together with any decision about the acceptability of the nonconforming work;

d) where the data quality is or may be impacted, the client is notified; and

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

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 Section 4.10 shall be promptly followed.

4.10 Corrective Action

4.10.1 General

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

4.10.2 Cause Analysis

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

4.10.3 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.

4.10.4 Monitoring of Corrective Actions

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

4.10 DOE-1

Prior to implementation of corrective actions where client data is affected, the laboratory shall notify the client of the proposed corrective action.

Written SOPs shall be in place for the notification of affected organizations regarding nonconforming items.

The laboratory shall have a system that tracks corrective actions to completion.

The laboratory shall have a system for tracking and trending lessons learned to prevent the reoccurrence of nonconformances.

Approved corrective actions that are developed to address findings identified during the annual DOECAP audit, must be implemented. Changes to approved corrective action plan must be approved by the DOECAP Operations Team. Willful avoidance of corrective action implementation of approved corrective actions may result in the elevation of the open finding to a Priority I finding. As a result, work may be discontinued until the corrective action has been implemented.

4.10.5 Additional Audits

Where the identification of nonconformances or departures casts doubts on the laboratory's compliance with its own policies and procedures or on its compliance with this document, the laboratory shall ensure that the appropriate areas of activity are audited in accordance with Section 4.13 as soon as possible.

4.10.6 Technical Corrective Action

a) In addition to providing acceptance criteria and specific protocols for corrective actions in the Method SOPs (see Section 5.4.1.1), the laboratory shall implement general procedures to be followed to determine when departures from documented policies, procedures and QC have occurred. These procedures shall include, but are not limited to the following:

   1) identify the individual(s) responsible for assessing each QC data type;

   2) identify the individual(s) responsible for initiating and/or recommending corrective actions;

   3) define how the analyst shall treat a data set if the associated QC measurements are unacceptable;

   4) specify how out-of-control situations and subsequent corrective actions are to be documented; and,

   5) specify procedures for management (including the quality manager) to review corrective action reports.

b) To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out-of-control, and the data is to be reported, all samples associated with the failed QC measure shall be reported with the appropriate laboratory defined data qualifier(s) and noted in the case narrative.

4.11 Preventive Action

Preventive action is a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

4.11.1 Needed improvements and potential sources of nonconformances, either technical or concerning the quality system, shall be identified. If preventive action is required, action plans shall be developed, implemented and monitored to reduce the likelihood of the occurrence of such nonconformances and to take advantage of the opportunities for improvement.

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

4.11 DOE-1

Documentation of preventive actions shall be maintained for review.

4.12 Control of Records

The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. The system shall produce unequivocal, accurate records that document all laboratory activities. The laboratory shall retain all original observations, calculations and derived data, calibration records and a copy of test reports for a minimum of five years.

There are two levels of sample handling: 1) sample tracking and 2) legal COC protocols, which are used for evidentiary or legal purposes. All essential requirements for sample tracking (e.g., COC form) are outlined in Sections 4.12.1.5, 4.12.2.4 and 4.12.2.5. If a client specifies that a sample will be used for evidentiary purposes, then a laboratory shall have a written SOP for how that laboratory will carry out legal COC for example, ASTM D 4840-95 and "Manual for the Certification of Laboratories Analyzing Drinking Water," March 1997, Appendix A.

4.12 DOE-1

A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record shall be maintained by the laboratory.

Legal/Evidentiary Custody

The use of legal COC protocols may be required by some State or federal programs. The following protocols shall be incorporated if legal COC is implemented by the organization.

Basic Requirements

The legal COC records shall establish an intact, continuous record of the physical possession, storage and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. The COC records shall account for all time periods associated with the samples. For ease of discussion, the above-mentioned items shall be referred to as samples:

a) A sample is in someone's custody if:
   1) It is in one's actual physical possession;
   2) It is in one's view, after being in one's physical possession;
   3) It is in one's physical possession and then locked or sealed so that no one can tamper with it; and/or
   4) It is kept in a secured area, restricted to authorized personnel only.

b) The COC records shall identify all individuals who physically handled individual samples

c) In order to simplify record keeping, the number of people who physically handle the sample should be minimized.

d) It is recommended that a designated sample custodian be appointed to be responsible for receiving, storing and distributing samples.
e) The COC records are not limited to a single form or document; however, organizations should attempt to limit the number of documents that would be required to establish COC.
f) Legal COC shall begin at the point established by the federal or State oversight program. This may begin at the point that cleaned sample containers are provided by the laboratory or the time sample collection occurs.
g) The COC forms shall remain with the samples during transport or shipment.
h) If shipping containers and/or individual sample containers are submitted with sample custody seals and any seals are not intact, the custodian shall note this on the COC.
i) Mailed packages should be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent COC documentation.
j) Once received by the laboratory, laboratory personnel are responsible for the care and custody of the sample and must be prepared to testify that the sample was in their possession and within view or secured in the laboratory at all times, from the moment it was received from the custodian until the time that the analyses are completed or the time of sample disposal.

Required Information in Custody Records

Tracking records shall include, by direct entry or linkage to other records:

a) Time of day and calendar date of each transfer or handling SOP;
b) Signatures of all personnel who physically handle the samples;
c) All information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis and reporting; and
d) Common carrier documents.

Controlled Access to Samples

Access to all legal samples and subsamples shall be controlled and documented.

a) A clean, dry, isolated room, building, and/or refrigerated space that can be securely locked from the outside must be designated as a custody room.
b) Where possible, distribution of samples to the analyst performing the analysis must be made by the custodian(s).
c) The laboratory area must be maintained as a secured area, restricted to authorized personnel only.

d) Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample must be retained in the custody room until permission to destroy the sample is received by the custodian or other authority.

Transfer of Samples to Another Party

Transfer of samples, subsamples, digestates or extracts to another party are subject to all of the requirements for legal COC.

Sample Disposal

a) Disposal of the physical sample shall occur only with the concurrence of the sample management office who submitted the sample.
b) All conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample shall be recorded and retained.
c) Records shall indicate the date and disposal, the nature of disposal (such as sample depleted, consumed during analysis, sample disposed in hazardous waste facility, or sample returned to client), and the name of the individual who performed the task.
d) For industrial hygiene analyses where sample are consumed during analysis, the laboratory must identify the tests and maintain preparation and analysis records that meet the requirements in 4.12.2.1.

4.12.1 General

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

4.12.1.2 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.

4.12.1.3 All records shall be held secure and in confidence.

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

4.12 DOE-2

The laboratory shall have procedures for independent review of records, logbooks, etc to ensure that quality records are legible, accurate, and complete.

4.12.1.5 The record keeping system must allow historical reconstruction of all laboratory activities that produced the analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples and/or extracts.

a) The records shall include the identity of personnel involved in sampling, sample receipt, preparation or testing.

b) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification shall be documented.

c) The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files.

d) All changes to records shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records such as “sampled by,” “prepared by,” or “reviewed by.”

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

f) Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria also shall apply to electronically maintained records.

4.12 DOE-3

If not self-explanatory, corrections to quality records shall also include a justification for the change.

g) Refer to Section 5.4.7.2 for Computer and Electronic Data.

4.12.2 Technical Records

4.12.2.1 The laboratory shall retain records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records

(including training and proficiency), and a copy of each test report issued, for a defined period. The records for each environmental test shall contain sufficient information to facilitate identification of factors affecting the uncertainty and to enable the environmental test 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 environmental test, and checking of results.

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

4.12.2.3 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 initialed 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.

When corrections are due to reasons other than transcription errors, the reason for the correction shall be documented.

4.12 DOE-4

Documentation for changes made to data (either hardcopy or electronic) shall include the identification of the person who authorized the change.

4.12.2.4 Records Management and Storage

a) All records (including those pertaining to test equipment), certificates and reports shall be safely stored, held secure and in confidence to the client. NELAP-related records shall be available to the accrediting authority.

b) All records, including those specified in Section 4.12.2.5 shall be retained for a minimum of five years from generation of the last entry in the records. All information necessary for the historical reconstruction of data must be maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

4.12 DOE-5

Written approval must be received from all affected clients, prior to disposal of any records associated with DOE analytical data.

c) Records that are stored or generated by computers or personal computers shall have hard copy or write-protected backup copies.

d) The laboratory shall establish a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting.

4.12 DOE-6

Laboratories must establish a review frequency for all laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, verification, validation, and record archival. Documentation of the reviews shall be maintained and available for review.

Permanent, bound laboratory notebooks (logbooks) are required and loose leaf binders shall not be used.

Laboratory notebooks (logbooks) shall have sequentially numbered pages, shall have entries that are signed by the person responsible for performing the activity at the time the activity is performed, and shall have entries in chronological order.
All logbook pages must be closed when the activities documented are completed or carried over to another logbook page. The person responsible for performing the closure shall be the one who performed the last activity documented. Closure shall occur at the end of the last activity performed or as soon as practicable thereafter.
Each laboratory notebook (logbook) shall have a unique serial number clearly displayed.

e) Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

4.12 DOE-7

Dual storage at a separate location is considered an acceptable option for this requirement.

The records control system SOP shall address the requirements for access to and control of the files, including accountability of records removed from the storage location.

f) 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, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

4.12.2.5 Laboratory Sample Tracking

4.12.2.5.1 Sample Handling

A record(s) of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These records shall include, but are not limited to, all records pertaining to:

a) sample preservation, including appropriateness of sample container and compliance with holding time requirement;

b) sample identification, receipt, acceptance or rejection and log-in;

c) sample storage and tracking, including shipping receipts, sample transmittal forms, (COC form); and

d) documented procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

4.12.2.5.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following shall be retained:

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

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

c) copies of final reports;

d) archived SOPs;

e) correspondence relating to laboratory activities for a specific project;

f) all corrective action reports, audits and audit responses;

g) PE results and raw data; and,

h) results of data review, verification, and cross-checking procedures.

4.12.2.5.3 Analytical Records

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, shall include:

a) laboratory sample identification code;

b) date of analysis and time of analysis is required if the holding time is 72 hours or less or when time critical steps are included in the analysis, e.g., extractions, and incubations;

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

d) analysis type;

e) all manual calculations, e.g., manual integrations; and,

f) analyst's or operator's initials/signature;

g) sample preparation activities, including cleanup, separation protocols, incubation periods or subculture, identification codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

h) sample analysis;

i) standard and reagent origin, receipt, preparation, and use;

j) calibration criteria, frequency and acceptance criteria;

k) data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

l) QC protocols and assessment;

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

n) method performance criteria including expected QC requirements.

4.12.2.5.4 Administrative Records

The following administrative records shall be maintained:

a) personnel qualifications, experience and training records;

b) records of demonstration of capability for each analyst; and

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

4.13 Internal Audits

4.13.1 The laboratory shall periodically, in accordance with a predetermined schedule and procedure, and at least annually, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the quality system and this document. The internal audit program shall address all elements of the quality system, including the environmental testing 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. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out.

4.13 DOE-1

DOE does not recognize the exemption "wherever resources permit" allowed under NELAC. Under the terms of this QSAS, management shall ensure that sufficient resources are available so that all internal audits shall be conducted by personnel independent of the activity to be audited.

Personnel conducting independent assessments shall have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the results of such assessments to laboratory management.

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

The laboratory shall notify clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or test certificate or amendment to a report or certificate.

The laboratory must specify, in the laboratory's quality manual, the time frame for notifying a client of events that cast doubt on the validity results.

4.13 DOE-2

The laboratory shall immediately notify all affected clients of potential data quality issues. Corrective actions taken to resolve the issue, including a summary of the investigation, shall be submitted to the client in a timely and responsive manner.

4.13.3 The area of activity audited, the audit findings, and corrective actions that arise from findings shall be recorded. The laboratory management shall ensure that these actions are discharged within the agreed time frame as indicated in the quality manual and/or SOPs.

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

4.14 Management Reviews

4.14 DOE-1

The Management Review shall not be performed in lieu of an internal audit. It is an independent, executive review.

4.14.1 In accordance with a predetermined schedule and procedure, the laboratory's executive management shall periodically, and at least annually, conduct a review of the laboratory's quality system and environmental testing activities to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. The review shall take account of:

a) the suitability and implementation of policies and procedures;

b) reports from managerial and supervisory personnel;

c) the outcome of recent internal audits;

d) corrective and preventive actions;

e) assessments by external bodies;

f) the results of interlaboratory comparisons or proficiency tests;

4.14 DOE-2

DOE requires results of both interlaboratory comparisons and proficiency tests.

a.) g) changes in the volume and type of the work;

b.) h) client feedback;

c.) i) complaints;

d.) j) other relevant factors, such as QC activities, resources and staff training.

4.14 DOE-3

Management review shall also include laboratory health and safety; hazardous; and radioactive materials management functions.

4.14.2 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.

The laboratory shall have a procedure for review by management and maintain records of review findings and actions.

4.14.3 The laboratory, as part of their overall internal auditing program, shall ensure that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery of potential issues shall be handled in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and the issues clarified. All investigations that result in finding of inappropriate activity shall be documented and shall include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. All documentation of these investigations and any actions taken shall be maintained for at least five years.

5.0 TECHNICAL REQUIREMENTS

5.1 General

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

a) human factors (see Section 5.2);

b) accommodation and environmental conditions (see Section 5.3);

c) environmental test methods and method validation (see Section 5.4);

d) equipment (see Section 5.5);

e) measurement traceability (see Section 5.6);

f) sampling (see Section 5.7);

g) the handling of samples (see Section 5.8).

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

5.2 Personnel

5.2.1 The laboratory management shall ensure the competence of all staff who operate specific equipment, perform environmental tests, evaluate results, and sign test reports. 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.

The laboratory shall have sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned functions.

All personnel shall be responsible for complying with all QA/QC requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, test methods, QA/QC procedures and records management.

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 program shall be relevant to the present and anticipated tasks of the laboratory.

5.2 DOE-1

The laboratory management shall ensure that all personnel, including part-time, temporary, contracted, and administrative personnel, are trained in the basic laboratory QA and health and safety programs.

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 quality system.

5.2.4 The laboratory shall maintain current job descriptions for all personnel who manage, perform, or verify work affecting the quality of the environmental tests.

5.2.5 The management shall authorize specific personnel to perform particular types of sampling, environmental testing, to issue test reports, 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.

Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory (see Section 5.2.6.c), including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in Section 5.4.2.2 for chemical testing.

5.2.6 The laboratory management shall be responsible for:

a) defining the minimal level of qualification, experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills such as using a balance, colony counting, and use of aseptic or quantitative techniques shall be considered;

b) ensuring that all technical laboratory staff have demonstrated capability in the activities for which they are responsible. Such demonstration shall be documented. (See Appendix C);

Note: In laboratories with specialized "work cells" (a well defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

c) ensuring that the training of each member of the technical staff is kept up-to-date (ongoing) by the following:

1) Evidence must be on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation, which relates to his/her job responsibilities.

2) Training courses or workshops on specific equipment, analytical techniques or laboratory procedures shall all be documented.

3) Analyst training shall be considered current if an employee training file contains a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or SOP as defined by the laboratory document control system, (see Section 4.2.3.d) and documentation of continued proficiency by at least one of the following once per year:

   i. acceptable performance of a blind sample (single blind to the analyst);

      Note: successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for EPA Methods 524.2, 624 or 5030/8260) would only require documentation for one of the test methods. The laboratory must determine the acceptable limits of the blind performance sample prior to analysis.

   ii. an initial measurement system evaluation or another demonstration of capability;

   iii. at least four consecutive laboratory control samples with acceptable levels of precision and accuracy;

      Note: The laboratory must determine the acceptable limits for precision and accuracy prior to analysis; and/or

   iv. if items i-iii cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

d) documenting all analytical and operational activities of the laboratory;

5.2 DOE-2

The laboratory shall have a system in place to record incidents involving spillage of reagents and client samples.

e) supervising all personnel employed by the laboratory.

f) ensuring that all sample acceptance criteria (see Section 5.8) are verified and that samples are logged into the sample tracking system and properly labeled and stored; and

g) documenting the quality of all data reported by the laboratory.

5.2 DOE-3

The laboratory management shall develop, maintain and implement a Chemical Hygiene Plan, Waste Management Plan, and if applicable, a Radiation Protection Plan.

5.2.7 Data integrity training shall be provided as a formal part of new employee orientation and must also be provided on an annual basis for all current employees. Topics covered shall be documented in writing and provided to all trainees. Key topics covered during training must include 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. Training shall include discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation. Employees are required to understand that any infractions of the laboratory data integrity procedures will 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 demonstrating that all staff have participated and understand their obligations related to data integrity. Senior managers acknowledge their support of these procedures by 1) upholding the spirit and intent of the organization's data integrity procedures, and 2) effectively implementing the specific requirements of the procedures.

Specific examples of breaches of ethical behavior should be discussed, including improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards. 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 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

5.3.1 Laboratory facilities for environmental testing, including but not limited to energy sources, lighting and environmental conditions, shall be such as to facilitate correct performance of the environmental tests.

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 environmental tests are

undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of environmental tests 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. Environmental tests shall be stopped when the environmental conditions jeopardize the results of the environmental tests.

In instances where monitoring or control of any of the above-mentioned items are specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.

5.3.3 There shall be effective separation between neighboring areas in which there are incompatible activities, including culture handling or incubation areas and volatile organic chemicals handling areas. Measures shall be taken to prevent cross-contamination.

5.3.4 Access to and use of areas affecting the quality of the environmental tests 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.

5.3 DOE-1

The laboratory shall have a safety inspection program in place that includes routine walk-downs of laboratory areas for safety-related concerns.

5.3.6 Work spaces must be available to ensure an unencumbered work area. Work areas include:

a) access and entryways to the laboratory;

b) sample receipt area(s);

c) sample storage area(s);

d) chemical and waste storage area(s); and,

e) data handling and storage area(s).

5.3 DOE-2

If contamination is discovered, the laboratory shall have a corrective action plan in place to identify and eliminate the source; determine which samples may have been impacted, and implement measures to prevent recurrence.

5.3 DOE-3

See Section 6 for additional DOE Materials Management and Health and Safety Requirements.

5.4 Environmental Test Methods and Method Validation

5.4.1 General

The laboratory shall use appropriate methods and procedures for all environmental tests within its scope. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of environmental test data.

The laboratory shall have instructions on the use and operation of all relevant equipment, and on the handling and preparation of samples where the absence of such instructions could jeopardize the results of environmental tests. All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be kept current and shall be made readily available to laboratory personnel (see Section 4.3). Deviation from environmental test methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the client.

5.4.1.1 Standard Operating Procedures (SOPs)

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 test 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 test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the methods manual (see Section 5.4.1.2).

c) Current copies of all SOPs shall be accessible to all personnel.

d) The SOPs shall be organized.

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

5.4 DOE-1

SOPs shall be in place for (but not limited to) the following areas:

- Sample Management
- Reagent/Standard Preparation
- General Laboratory Techniques
- Test Methods
- Glassware Cleaning
- Equipment Calibration and Maintenance
- Quality Control
- Corrective Action
- Data Reduction and Validation
- Reporting
- Records Management
- Radioactive and/or Hazardous Material Management.

f) The documents specified in Sections 5.4.1.1.a and 5.4.1.1.b that contain sufficient information to perform the tests do not need to be supplemented or rewritten as internal procedures, if they are written in a way that they can be used as written. Any changes, including the use of a selected option must be documented and included in the laboratory's methods manual.

5.4.1.2 Laboratory Method Manual(s)

a) The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.

5.4 DOE-2

The laboratory shall have documented procedures for all requested DOE analytes.

The laboratory may use either hardcopy or electronic format for the methods documents.

b) This manual may consist of copies of published or referenced test methods or SOPs that have been written by qualified laboratory personnel. In cases where modifications to the published method have been made by the laboratory, or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described. Each test method shall include or reference where applicable:

1) identification of the test method;
2) applicable matrix or matrices;
3) detection limit;
4) scope and application, including components to be analyzed;
5) summary of the test method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies;
10) reagents and standards;
11) sample collection, preservation, shipment and storage;
12) QC;
13) calibration and standardization;
14) procedure;
15) data analysis and calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for QC measures;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and,
23) any tables, diagrams, flowcharts and validation data.

5.4.2 Selection of Methods

The laboratory shall use methods for environmental testing, including methods for sampling, which both meet the needs of the client and are appropriate for the environmental tests it undertakes.

5.4.2.1 Sources of Methods

a) 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.

b) When the use of specific methods for a sample analysis are mandated or requested, only those methods shall be used.

c) When the client does not specify the method to be used or where methods are employed that are not required, the methods shall be fully documented and validated (see Sections 5.4.2.2, 5.4.5, and Appendix C), and be available to the client and other recipients of the relevant reports. 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 client shall be informed as to the method chosen.

5.4 DOE-3

Laboratory selected or modified methods shall be approved by the DOE client prior to use.

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

5.4.2.2 Demonstration of Capability

The laboratory shall confirm that it can properly operate all methods before introducing the environmental tests. If the method changes, the confirmation of the method shall be repeated.

a) Prior to acceptance and institution of any method, satisfactory demonstration of method capability is required. (See Appendix C and Section 5.2.6.b) In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean quality system matrix sample (a quality system matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., drinking water, solids, biological tissue and air. In addition, for analytes that do not lend themselves to spiking, the demonstration of capability may be performed using QC samples.

b) Thereafter, continuing demonstration of method performance, as per the QC requirements in Appendix D (such as laboratory control samples) is required.

c) In cases where a laboratory analyzes samples using a method that has been in use by the laboratory before July 1999, and there have been no significant changes in instrument type, personnel or method, the continuing demonstration of method performance and the analyst's documentation of continued proficiency shall be acceptable. The laboratory shall have records on file to demonstrate that a demonstration of capability is not required.

d) In all cases, the appropriate forms such as the Certification Statement (Appendix C) must be completed and retained by the laboratory to be made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the Certification Statement must be retained by the laboratory. (See Appendix C for Certification Statement)

e) A demonstration of capability must be completed each time there is a change in instrument type, personnel, or method.

f) In laboratories with a specialized "work cell(s)" (group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit must meet the above criteria and this demonstration of capability must be fully documented.

g) When a work cell(s) is employed, and the members of the cell change, the new employee(s) must work with experienced analyst(s) in that area of the work cell where they are employed. This new work cell must demonstrate acceptable performance through acceptable continuing performance checks (appropriate sections of Appendix D, such as laboratory control samples). Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. In addition, if the entire work cell is changed/replaced, the work cell must perform the demonstration of capability (Appendix C).

h) When a work cell(s) is employed, the performance of the group must be linked to the training record of the individual members of the work cell (see Section 5.2.6).

5.4.3 Laboratory-Developed Methods

The introduction of environmental test 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

When it is necessary to use methods not covered by standard 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.

5.4.5 Validation of Methods

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 published 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. The minimum requirements shall be the initial test method evaluation requirements given in Appendix C.3.

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 clients' needs.

5.4.6 Estimation of Uncertainty of Measurement

5.4.6.1 Environmental 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.

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 Section 5.10).

5.4.6.2 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.

5.4.7 Control of Data

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

a) The laboratory shall establish SOPs to ensure that the reported data are free from transcription and calculation errors.

b) The laboratory shall establish SOPs to ensure that all QC measures are reviewed, and evaluated before data are reported.

c) The laboratory shall establish SOPs addressing manual calculations including manual integrations.

5.4.7.2 When computers, automated equipment, or microprocessors are used for the acquisition, processing, recording, reporting, storage or retrieval of environmental test 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, including, but not 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 environmental test data;

d) appropriate procedures are established and implemented for the maintenance of security of data, including the prevention of unauthorized access to and the unauthorized amendment of, computer records;

Note: Commercial off-the-shelf software (e.g., word processing, database and statistical programs) in general use within its designed application range is considered to be sufficiently validated. However, laboratory software configuration or modifications must be validated as in Section 5.4.7.2a.

5.4 DOE-4

Individual user names and passwords are required for all LIMS users. LIMS passwords shall be changed on a regular basis, at a minimum of once per year.

Laboratory employees shall have initial training in computer security awareness upon employment and shall have ongoing refresher training on annual basis. Documentation of the training shall be maintained and available for review.

Periodic inspections of the electronic operations shall be performed by the QA Organization to ensure the integrity of electronic data. The QA Organization shall maintain records of inspections and submit reports to laboratory management, noting any problems identified with electronic data processing stating the corrective actions taken.

Spreadsheets used for calculations shall be verified before initial use and following any changes to equations or formulas, including software revision upgrades, and documentation shall be readily available for review. Formula cells must be write-protected to minimize inadvertent changes to the formulas. Printouts from any spreadsheets shall include all information used to calculate the data.

The Laboratory shall have SOPs for:

- Software development methodologies that are based on the size and nature of the software being developed;
- Testing and QA methods to ensure that all software accurately performs its intended functions, including:

- o Acceptance criteria;
- o Tests to be used,
- o Personnel responsible for conducting the tests,
- o Documentation of test results,
- o Frequency of continuing verification of the software, and,
- o Test review and approval.

- Software change control methods that include instructions for requesting, authorizing, requirements to be met by the software change, testing, QA, approving, implementing changes, and establishing priority of change requests.
- Software version control methods that document the software version currently used. Data sets are documented with the date and time of generation and/or the software version used to generate the data set; and
- Maintaining a historical file of software, software operating procedures, software changes, and software version numbers.
- Defining the acceptance criteria, testing, documentation, and approval required for changes to LIMS hardware and communication equipment.

Documents available in the laboratory to demonstrate the validity of laboratory-generated software include:

- Software description and functional requirements;
- Listing of algorithms and formulas;
- Testing and QA documentation; and,
- Installation, operation and maintenance records.
-

LIMS Safeguards

- Fire extinguishers designed to avoid damage to computer equipment must be available and mounted in visible, accessible areas.

Electronic Data Security

- Individual user names and passwords have been implemented;
- Operating system privileges and file access safeguards are implemented to restrict the use of the LIMS data to users with unauthorized access;
- All LIMS Users are trained in computer awareness security on an annual basis;
- System events, such as log-on failures or break-in attempts are monitored;
- The electronic data management system is protected from the introduction of computer viruses;
- System backups occur on a regular and published schedule and can be performed by more than one person within an organization;

- Testing of the system backups must be performed and documented to demonstrate that the backup tapes contain all required data; and,
- Physical access to the servers is limited by security measures such as locating the system within a secured facility or room, and/or utilizing cipher locks or key cards.

The laboratory shall notify the DOE client prior to major changes in software or hardware configuration of the electronic data management system.

5.5 Equipment

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

5.5.2 Equipment and its software used for testing and sampling shall be capable of achieving the accuracy required and shall comply with specifications relevant to the environmental tests concerned. 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.

Calibration requirements are divided into two parts: 1) requirements for analytical support equipment, and 2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

5.5.2.1 Support Equipment

These standards apply 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 volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume.

5.5 DOE-1

The support equipment also includes ultrasonic disruptors and microwave digestion units. Documentation of sonication equipment tuning and microwave calibration shall be maintained.

a) All support equipment shall be maintained in proper working order. The records of all repair and maintenance activities including service calls shall be kept.

5.5 DOE-2

The laboratory shall have a system that records and documents catastrophic failure of refrigerators and freezers and addresses identification of affected samples and client notification.

b) All support equipment shall be calibrated or verified at least annually, using NIST-traceable references when available, over the entire range of use. The results of such calibration or verification shall be within the specifications required of the application for which this equipment is used or:

   1) the equipment shall be removed from service until repaired; or

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

5.5 DOE-3

Other international traceable standards may be used when NIST-traceable standards are not available.

When a traceable standard is not available, written approval for use of a non-traceable standard must be obtained from the DOE client.

Support equipment shall be uniquely identified by an identification protocol.

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

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST-traceable references where commercially available. 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.

5.5 DOE-4

Balances shall be calibrated before initial use and annually thereafter and labeled to that effect by an independent (third party) professional technician, not associated with the laboratory daily operation. The calibration or verification must be performed using recognized National Metrology Institute, such as NIST, traceable references when available, bracketing the range of use. Laboratory personnel shall check balances daily or before use and the balance check shall bracket the range of use. Daily balance checks shall be documented. The results of such calibration or verification shall be within the specifications required of the application for which this equipment is used or the equipment shall be removed from service until repaired. The laboratory shall maintain records of established correction factors to correct all measurements.

Class 1 (formerly referred to as Class S) certified check weights shall be verified every five years using recognized National Metrology Institute, such as NIST, traceable references when available, bracketing the range of use. Alternatively, Class 1 check weights may be reverified using controlled check weight standards that are used exclusively for this purpose. Initial and verification checks shall be documented.

Daily monitoring of temperatures (refrigerators, freezers, and ovens) shall be documented. For temperature monitoring, “daily” refers to calendar days, not working days. Temperature monitoring data loggers are acceptable provided they have the capability of providing notification of an out of control event to responsible individual(s) during routine and non-routine work periods. Corrective actions shall be performed in the event of an out of control condition or catastrophic failure of a refrigerator or freezer. The requirement for daily monitoring of temperature for refrigerators and freezers will not apply in the event that samples are not being stored from a DOE site.

The conductivity and/or resistivity of the water from the purification system shall be monitored daily or before use and the results recorded in a logbook. The laboratory shall have an SOP for reagent and deionized water production that includes (at a minimum): preventative maintenance of water purification equipment, control criteria, and corrective action processes for out-of-spec water.

e) Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be checked for accuracy on at least a quarterly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory.

5.5 DOE-5

The accuracy of all mechanical volumetric dispensing devices used for quantitative measurements shall be verified (checked for accuracy daily or prior to use).

Volumetric dispensing devices (except Class A glassware and glass microliter syringes) shall be checked for accuracy on a quarterly basis. Refer to 5.5.2.1 e

f) For chemical tests, the temperature, cycle time, and pressure of each run of autoclaves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

g) For biological tests that employ autoclave sterilization, see Appendix D, Section 3.8.

5.5.2.2 Instrument Calibration

This document specifies the essential elements that shall define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data must be of known quality and be appropriate for a given regulation or decision. This document 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 test 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 test method are to be followed.

5.5.2.2.1 Initial Instrument Calibration

The following items are essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics must be included or referenced in the test method SOP. When initial instrument calibration procedures are referenced in the test method, then the referenced material must be retained by the laboratory and be available for review.

b) Sufficient raw data records must be retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, 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) Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method, or program.

d) All initial instrument calibrations must be verified with a standard obtained from a second manufacturer or lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Traceability shall be to a national standard, when commercially available.

e) Criteria for the acceptance of an initial instrument calibration must be established, e.g., correlation coefficient or relative percent difference. The criteria used must be appropriate to the calibration technique employed.

f) The lowest calibration standard shall be the lowest concentration for which quantitative data are to be reported (see Appendix C). Any data reported below the lower limit of quantitation should be considered to have an increased quantitative uncertainty and shall be reported using defined qualifiers or flags or explained in the case narrative.

g) The highest calibration standard shall be the highest concentration for which quantitative data are to be reported (see Appendix C). Any data reported above this highest standard should be considered to have an increased quantitative uncertainty and shall be reported using defined qualifiers or flags or explained in the case narrative.

h) Measured concentrations outside the working range shall be reported as having less certainty and shall be reported using defined qualifiers or flags or explained in the case narrative. The lowest calibration standard must be above the limit of detection. Noted exception: The following shall occur for instrument technology (such as ICP or ICP/MS) with validated techniques from manufacturers or methods employing standardization with a zero point and a single point calibration standard:

5.5 DOE-6

Samples with concentrations that exceed the calibration range must be diluted to fall within the range, if there is enough sample and prep/analysis will fall within holding time criteria.

Samples shall not be initially diluted unless all the project required quantitation limits can be met; otherwise, samples shall be reanalyzed at a lower dilution. All exceptions shall be discussed with the contract technical point of contact.

1) Prior to the analysis of samples, the zero point and single point calibration must be analyzed and the linear range of the instrument must be established by analyzing a series of standards, one of which must be at the lowest quantitation level. Sample results within the established linear range will not require data qualifier flags.

2) Zero point and single point calibration standard must be analyzed with each analytical batch.

3) A standard corresponding to the limit of quantitation must be analyzed with each analytical batch and must meet established acceptance criteria.

5.5 DOE-7

The limit of quantitation is equivalent to the practical quantitation limit.

4) The linearity is verified at a frequency established by the method and/or the manufacturer.

5.5 DOE-8

The requirements defined in Sections 5.5.2.2.1.f - h (above) do not apply to measurement techniques not using a calibration curve (e.g., radiological measurements).

i) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed. If reanalysis of the samples is not possible, data associated with an unacceptable initial instrument calibration shall be reported with appropriate data qualifiers.

j) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, (one of which must be at the limit of quantitation) not including blanks or a zero standard, with the noted exception of instrument technology for which it has been established by methodologies and procedures that a zero and a single point standard are appropriate for calibrations (see Section 5.5.2.2.1.h above). The laboratory must have an SOP for determining the number of points for establishing the initial instrument calibration.

5.5.3 Equipment shall be operated by authorized personnel. Current 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.

All equipment shall be properly maintained, inspected and cleaned. Maintenance procedures shall be documented.

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

5.5.5 The laboratory shall maintain records of each major item of equipment and its software significant to the environmental tests 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;

5.5 DOE-9

The laboratory must maintain a current list of available major equipment that includes equipment type, date of purchase, repair history, and calibration status.

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

d) the current location;

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;

5.5 DOE-10

These shall include instrument configuration and settings.

g) the maintenance plan, where appropriate, and maintenance carried out to date; documentation on all routine and non-routine maintenance activities and reference material verifications;

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

5.5 DOE-11

Instrument shall be set up according to the manufacturer's instructions. Any deviations shall be documented in the instrument maintenance log.

i) date received and date placed in service (if available);

j) if available, condition when received (e.g., new, used, reconditioned);

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.

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 labeled 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 environmental tests and shall institute the "Control of nonconforming work" procedure.

5.5.8 Whenever practicable, all equipment under the control of the laboratory and requiring calibration shall be labeled, 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 an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analysis by continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

a) The details of the continuing instrument calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.

b) Calibration shall be verified for each compound, element, or other discrete chemical species, except for multi-component analytes such as Aroclors, Total Petroleum Hydrocarbons, or Toxaphene, where a representative chemically related substance or mixture can be used.

c) Instrument calibration verification must be performed:

1) at the beginning and end of each analytical batch (except, if an internal standard is used, only one verification needs to be performed at the beginning of the analytical batch);

2) whenever it is expected that the analytical system may be out-of-calibration or might not meet the verification acceptance criteria;

3) if the time period for calibration or the most previous calibration verification has expired; or

4) for analytical systems that contain a calibration verification requirement.

d) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test 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 must explicitly connect the continuing verification data to the initial instrument calibration.

e) Criteria for the acceptance of a continuing instrument calibration verification must be established, e.g., relative percent difference.

If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial instrument calibration must be performed. If the laboratory has not verified calibration, sample analyses may not occur until the analytical system is calibrated or calibration verified. If samples are analyzed using a system on which the calibration has not yet been verified the results shall be flagged. Data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

1) 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 reanalyzed after a new calibration curve has been established, evaluated and accepted; or

2) 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 reanalyzed after a new calibration curve has been established, evaluated and accepted.

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 equipment, including both hardware and software, shall be safeguarded from adjustments that would invalidate the test results.

5.6 Measurement Traceability

5.6.1 General

All equipment used for environmental tests, including equipment for subsidiary measurements (e.g., for environmental conditions) having a significant effect on the accuracy or validity of the result of the environmental test or sampling, shall be calibrated before being put into service and on a continuing basis. The laboratory shall have an established program and procedure for the calibration of its equipment. This includes balances, thermometers, and control standards. Such a program shall 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 environmental tests.

5.6.2 Testing Laboratories

5.6.2.1 For testing laboratories, the laboratory shall ensure that the equipment used can provide the uncertainty of measurement needed.

The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that measurements made by the laboratory are traceable to national standards of measurement.

5.6.2.2 Where traceability of measurements to SI units is not possible or not relevant, the same requirements for traceability to, e.g., CRMs, agreed methods and/or consensus standards are required. The laboratory shall provide satisfactory evidence of correlation of results, e.g., by participation in a suitable program of interlaboratory comparisons, PE, or independent analysis.

5.6.3 Reference Standards and Reference Materials

5.6.3.1 Reference Standards

The laboratory shall have a program and procedure for the calibration of its reference standards. Reference standards shall be calibrated by a body that can provide traceability as described in Section 5.6.2.1. Such reference standards of measurement held by the laboratory (such as Class S or equivalent weights or traceable thermometers) 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. Where commercially available, this traceability shall be to a national standard of measurement.

5.6.3.2 Reference Materials

Reference materials shall, where commercially available, be traceable to SI units of measurement, or to CRMs. 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.

5.6.3.3 Intermediate Checks

Checks needed to maintain confidence in the 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.

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

Documented procedures shall exist for the purchase, reception 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 supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material shall not be used unless its reliability is verified by the laboratory.

5.6 DOE-1

If the standards are not re-verified, as stated above, the laboratory shall remove the standard or clearly designate it as acceptable for qualitative purposes only.

Reagents and solvents shall be checked for purity prior to use and the supporting documentation of the checks shall be filed in a manner that is easily retrievable.

Standards and reference materials shall be stored separately from samples and protected in a controlled cabinet or refrigerator.

Purchased stock mixtures and reagents shall be labeled to indicate the date on which the mixtures/reagents were opened and the expiration date.

b) Original containers (such as provided by the manufacturer or vendor) shall be labeled with an expiration date.

c) Records shall be maintained on standard and reference material 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, and reference materials must bear a unique identifier and expiration date and be linked to the documentation requirements in Section 5.6.4.c above.

e) Procedures shall be in place to ensure that prepared reagents meet the requirements of the test method. The source of reagents shall comply with Sections 5.9.2.a and 5.9.2.a.6 and Appendix D, Section 1.4 b.

f) All containers of prepared reagents must bear a preparation date. An expiration date shall be defined on the container or documented elsewhere as indicated in the laboratory's quality manual or SOP.

5.6 DOE-2

The expiration date of the secondary standard shall not exceed the expiration date of the primary standard.

5.7 Sampling

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 environmental testing. 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 environmental test results.

Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples.

5.7.2 Where the client 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 environmental 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 environmental testing 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.

5.8 Handling of Samples

While the laboratory may not have control of field sampling activities, the following are essential to ensure the validity of the laboratory's data.

5.8.1 The laboratory shall have procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of samples, including all provisions necessary to protect the integrity of the sample, and to protect the interests of the laboratory and the client.

5.8 DOE-1

Personnel dealing with sample receipt, radioactive waste management and materials shipping shall be trained in waste management, shipping (49 CFR 172) and handling, and radioactive material control.

5.8.2 The laboratory shall have a system for identifying samples. The identification shall be retained throughout the life of the sample in the laboratory. The system shall be designed and operated so as to ensure that samples cannot be

confused physically or when referred to in records or other documents. The system shall, if appropriate, accommodate a subdivision of groups of samples and the transfer of samples within and from the laboratory.

a) The laboratory shall have a documented system for uniquely identifying the 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, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification code to each sample container received in the laboratory. The use of container shape, size or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.

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

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

d) The laboratory identification code shall be entered into the laboratory records (see Section 5.8.3.1.d) 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 preassigns numbers to sample containers, the laboratory identification code may be the same as the field identification code.

5.8.3 Upon receipt of the samples, the condition, including any abnormalities or departures from normal or specified conditions as described in the environmental test method, shall be recorded. When there is doubt as to the suitability of a sample for environmental test, or when a sample does not conform to the description provided, or the environmental test required is not specified in sufficient detail, the laboratory shall consult the client for further instructions before proceeding and shall record the discussion.

5.8.3.1 Sample Receipt Protocols

a) All items specified in Section 5.8.3.2 below shall be checked.

1) All samples that require thermal preservation shall be considered acceptable if the arrival temperature 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. Samples that are hand delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples shall

be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

5.8 DOE-2

All samples that require thermal preservation shall be considered acceptable if the arrival temperature is between 0°C and 6°C, or the method specified range.

Refrigerators that are used to store samples that require thermal preservation shall be monitored and controlled between 0°C and 6°C.

2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or chlorine, prior to or during sample preparation or analysis.

5.8 DOE-3

Chemical preservation is matrix specific. The laboratory shall refer to the COC for the matrix definition. In the case where the matrix is not identified on the COC, the laboratory shall contact the client prior to proceeding.

The laboratory shall have the ability to meet method specific guidance/ requirements for verification of preservation.

3) Microbiological samples from chlorinated water systems do not require an additional chlorine residual check in the laboratory if the following conditions are met:

   i. sufficient sodium thiosulfate is added to each container to neutralize at minimum five mg/l of chlorine for drinking water and 15 mg/l of chlorine for wastewater samples;

   ii. one 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 five mg/l chlorine or 15 mg/l chlorine as appropriate and the check is documented;

   iii. chlorine residual is checked in the field and actual concentration is documented with sample submission.

b) The results of all checks shall be recorded.

c) If the sample does not meet the sample receipt acceptance criteria listed in this document, the laboratory shall either:

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

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

   i. The condition of these samples shall, at a minimum, be noted on the COC or transmittal form and laboratory receipt documents.

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

5.8 DOE-4

The laboratory shall establish whether the sample has received all necessary preparation or whether the client requires preparation to be undertaken or arranged by the laboratory.

d) The laboratory shall utilize a permanent chronological record, such as a log book or electronic database, to document receipt of all sample containers.

   1) This sample receipt log shall record the following:

      i. client/project name,

      ii. date and time of laboratory receipt,

      iii. unique laboratory identification code (see Section 5.8.2), and,

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

   2) During the log-in process, the following information must 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. Note: the placement of the laboratory identification number on the sample container is not considered a permanent record.

      i. The field identification code that identifies each container must be linked to the laboratory identification code in the sample receipt log.

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

      iii. The requested analyses (including applicable approved test method numbers) must be linked to the laboratory identification code.

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

e) All documentation, such as memos or transmittal forms that is transmitted to the laboratory by the sample transmitter shall be retained.

f) A complete COC record form (Section 4.12.2.5 and Appendix E), if utilized, shall be maintained.

5.8 DOE-5

When the laboratory receives samples, an internal COC procedure shall be initiated.

Internal custody shall be maintained until final disposition or return of sample to the client.

5.8.3.2 Sample Acceptance Policy

The laboratory must have a written sample acceptance policy that clearly outlines the circumstances under which samples shall be accepted or rejected. Data from any samples that do not meet the following criteria must be flagged in an unambiguous manner clearly defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collection personnel and shall include, but is not limited to, the following areas of concern:

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;

5.8 DOE-6

The laboratory shall have a procedure in place to address notification of laboratory personnel of short holding times.
The maximum radiological sample holding time allowable is 180 days. Additionally, the maximum sample holding time shall not exceed five half-

lives of an unsupported nuclide of interest when five half-lives is shorter than 180 days. The laboratory shall be able to accommodate sample-specific requirements for specific isotopes or very short-lived isotopes.

e) adequate sample volume, i.e., sufficient sample volume must be available to perform the necessary tests; and

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

5.8 DOE-7

The laboratory shall have SOPs in place to address the use of fume hoods or suitable containment for opening shipping containers, and radiation screening of samples, laboratory notification and labeling requirements for radioactive samples.

Shipping containers from DOE sites must be opened under a ventilation hood to ensure employees are protected. The laboratory must have a procedure and records to verify contamination control on a semiannual basis such as smoke test or flow meter measurements.

Materials submitted for industrial hygiene or asbestos analysis must be opened in an established manner to prevent worker exposure. Therefore, receiving practices must be developed and implemented for the receipt of beryllium, beryllium oxide, and asbestos materials.

All shipping containers from known radiological areas must be surveyed for radiological contamination on all external surfaces. The laboratory must develop and implement administrative policies for the receipt of radiological shipping containers and samples. Radiological surveys of sample shipping containers shall be performed as soon as possible from the time of receipt by the laboratory.

Instrumentation and equipment used for monitoring shall be:

Periodically maintained and calibrated on an established frequency;
Appropriate for the type(s), levels, and energies of the radiation encountered;
Appropriate for existing environmental conditions; and
Routinely tested for operability. (10CFR835.401(b))

Prior to performing radiological surveys, the radiological survey instrumentation shall be checked for performance using a radiological source, a battery check shall be performed and the nominal radiological background

will be measured. All performance checks shall be documented and available for review.

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

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

1) Samples that require thermal preservation shall be stored under refrigeration that is +/-2 of the specified preservation temperature unless 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.

2) Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in such a manner as to prevent cross contamination.

5.8 DOE-8

A refrigerator storage blank shall be present for the storage of all volatile organic samples. Specific SOPs for assessing the adequacy of these storage blank data and taking action for nonconforming conditions shall be established. The storage blanks shall be stored in the same manner as the client samples. The refrigerator storage blank shall be analyzed every 14 days when samples are being stored in the laboratory. The data from the analysis of the refrigerator storage blanks shall be available for review.

b) Sample fractions, extracts, leachates and other sample preparation products shall be stored according to Section 5.8.4.a above or according to specifications in the test method.

5.8 DOE-9

The laboratory shall maintain an indexed sample storage system that facilitates sample retrieval.

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

5.9 Assuring the Quality of Environmental Test and Calibration Results

5.9.1 General

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 CRMs and/or internal QC using secondary reference materials;

b) participation in interlaboratory comparison and/or PE program (see NELAC, Chapter 2)

5.9 DOE-1

The laboratory shall demonstrate successful participation for a minimum of one year in a nationally recognized PE program prior to contract award under this QSAS. The laboratory shall demonstrate continued proficiency throughout the term of the contract award.

The single-blind studies must be related to regulatory or environmental programs, matrix types, or analytes for each of the analytical disciplines (i.e., inorganic, organic, radiochemistry) that each laboratory is awarded contract under this QSAS. A laboratory is only required to analyze samples containing analytes, and samples of matrices, applicable to data they report under DOE contracts.

Required PE programs include, as applicable:

MAPEP, conducted by DOE RESL at INL, or another accredited PE program for laboratories performing either radiochemical, organic, or inorganic analyses for DOE. A laboratory must possess a radioactive materials license from the Nuclear Regulatory Commission or an Agreement State, or a DOE exemption to receive MAPEP samples.

MAPEP is required for all laboratories that possess a radiological materials license and that perform Inorganic, Semivolatile Organic, or radiochemical analyses for DOE. Laboratories that provide volatile organic and wet chemistry analyses to DOE will be required to maintain proficiency in nationally recognized PE program for all matrices.

RadCheM™ PE Program, conducted by Environmental Resource Associates (or equivalent programs offered by other commercial suppliers

if such suppliers become NVLAP-accredited in the future), for radioactivity measurements in drinking water.

NELAC Fields of Testing for CWA-Water [formerly known as WP]. Under the terms of this QSAS, a laboratory may participate in two single-blind, single-concentration PE studies provided by an approved supplier. The PE suppliers must be approved by the PTOB/PTPA administered by the NELAP.

NELAC Fields of Testing for SDWA-Water [formerly known as WS]. Under the terms of this QSAS, a laboratory must participate in two single-blind, single-concentration PE studies provided by an approved supplier. The PE suppliers must be approved by the PEOP/PEPA administered by the NELAP.

AIHA Proficiency accreditation for Asbestos and Beryllium (if applicable).

Recommended Programs:

DMR QA program for NPDES analysis.

NELAC Fields of Testing for RCRA Solid. Under the terms of this QSAS, a laboratory may participate in two single-blind, single-concentration PE studies provided by an approved supplier. The PE suppliers must be approved by the PEOP/PEPA administered by the NELAP.

In addition, the client reserves the right to submit blind PE samples. Each laboratory shall continue to participate in all applicable rounds of external PE programs. The results of all PE programs will be utilized in the reports produced for DOE laboratory users. Therefore, DOE will provide the laboratories operating to this QSAS instruction for ensuring the results of commercially PE studies are made available to DOE and the sites that have contracts with the laboratories

Reporting an unacceptable value, as calculated by the PE program, will result in a probationary period until the next reporting period for that analyte. Any applicable analyte for which individual laboratory results are entered as NR or “not reported” will not be considered an acceptable result. Any individual analyte failures must be corrected within the next PE program performance cycle period. If the laboratory fails two consecutive evaluations, the laboratory may not receive samples for analysis by the failed method until an acceptable PE score has been achieved. The laboratory can demonstrate proficiency in non-MAPEP PT studies by acceptable performance in an unscheduled evaluation by the same PE program at the expense of the laboratory.

For two or more consecutive failed (Not Acceptable) MAPEP results, the laboratory may not receive samples for analysis by the failed method until an acceptable remedial MAPEP PT sample score has been achieved. The decision to withhold sample shipments will be at the discretion of the individual DOE contract holder.

For all PE studies other than acceptable results, the following will be considered when evaluating the reported results:

Consistent bias, either positive or negative, at the "Warning" level (greater than +/- 20% bias) for a targeted analyte in a given sample matrix for the two most recent test sessions (e.g., Sr-90 in air filter 13 "+W" (+26%), Sr-90 in air filter test 14 "+W" (+28%));

Quality issues (flags other than "Acceptable") that were not identified by the above for a targeted analyte in a given sample matrix over the last three test sessions, (e.g., Am-241 in soil test 12 "-N" (-47%), Am-241 in soil test 13 "+W" (+24%) in soil test 13 "+W" (+24%). Am-241 in soil test 14 "-N" (-38%)); and

Any other performance indicator and/or historical trending that demonstrate an obvious quality concern (e.g., consistent "False Positive" results for Pu-238 in all tested matrices over the last three test sessions.

The laboratory shall document the cause(s) for failed PE results and develop corrective action(s) to address the cause(s) within 21 calendar days from receipt of the results. These actions should then be available for DOECAP review upon request. In the event of multiple failures that result in the issuance of a DOECAP Priority I finding, the laboratory should identify the root cause of the failure using a sample from a previous MAPEP study or the laboratory can request that DOECAP contact RESL to provide a sample from previous MAPEP studies. The previous study samples are to be used to aide in the determination of the root cause of the unacceptable result(s). The samples from a previous round of testing will not be scored by MAPEP.

Once a laboratory has demonstrated that they can achieve acceptable results, based on the previously determined limits of the test session, DOECAP will contact RESL to provide one new remedial PE sample to the laboratory for analysis. The laboratory will provide the results of the remedial study to RESL and the results will be evaluated using the same evaluation criteria that are used for the normal MAPEP studies. If the results are acceptable, the Priority I finding can be evaluated for closure by DOECAP. If the results are not acceptable, the laboratory will be encouraged to continue resolution of any technical problems and will not be provided a second remedial PT sample. The requests for remedial PT samples will be made solely at the request of DOECAP and not from the participating laboratories.

Following the resolution of failed PT samples that result in a Priority I finding, the laboratories are required to achieve acceptable results in the next MAPEP testing round. If the results of the next round of testing are not acceptable, the laboratory will be evaluated for further corrective actions or suspension of further work. The decision for any suspension will be determined by the DOE contract holders.

c) replicate tests using the same or different methods;

d) retesting of retained samples;

e) correlation of results for different characteristics of a sample (e.g., total phosphate should be greater than or equal to orthophosphate).

5.9.2 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., chemical, whole effluent toxicity, microbiological, radiological, air) and are further described in Appendix D. 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:

1) positive and negative controls to monitor tests such as blanks, spikes, reference toxicants;

5.9 DOE-2

Positive and negative controls shall be used to monitor accuracy, bias, and cross-contamination potential, e.g. blanks, matrix spikes, tracer recoveries, laboratory control standards and standard reference materials.

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

3) measures to ensure the accuracy of the test method including calibration and/or continuing calibrations, use of CRMs, PE samples, or other measures;

4) measures to evaluate test method capability, such as limit of detection and limit of quantitation or range of applicability such as linearity;

5.9 DOE-3

The laboratory shall maintain a list of typical detection limits, achieved for water, soil, and other matrices commonly analyzed, and procedures for determining limits of detection and frequency of verification.

Method detection limits shall be updated or verified on an annual basis.

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

6) selection and use of reagents and standards of appropriate quality;

7) measures to ensure the selectivity of the test for its intended purpose; and

8) measures to ensure constant and consistent test conditions (both instrumental and environmental) where required by the test method, such as temperature, humidity, light, or specific instrument 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. (See Appendix D.)

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See Section 5.8.3.2, Sample Acceptance Policy)

d) The QC protocols specified by the laboratory's method manual (see Section 5.4.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D 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.

e) The essential QC measures for testing are found in Appendix D.

5.10 Reporting the Results

5.10.1 General

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

The results shall be reported, in a test report, and shall include all the information requested by the client and necessary for the interpretation of the environmental test or calibration results and all information required by the method used. This information is normally that required by Sections 5.10.2 and 5.10.3.

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

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

Laboratories that are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes (in-house or captive laboratories) shall have all applicable information specified in Sections 5.10.2.a through 5.10.2.m (below) readily available for review by the accrediting authority. 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 must ensure that the appropriate report items are in the report to the regulatory authority if such information is required.

5.10.2 Test Reports

Each test report shall include at least the following information, unless the laboratory has valid reasons for not doing so, as indicated by the following:

a) a title (e.g., “Test Report,” “Certificate of Results,” or “Laboratory Results”);

b) the name and address of the laboratory, the location where the environmental tests were carried out (if different from the address of the laboratory), and phone number with name of contact person for questions;

c) unique identification of the test report (such as the serial number), and on each page, an identification to ensure that the page is recognized as a part of the test report and a clear identification of the end of the test report;

    1) This requirement may be presented in several ways:

i. The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or

ii. Each page is identified with the unique report identification. The pages are identified as a number of the total report pages (example: 3 of 10, or 1 of 20).

2) Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report, and that the report contains a specified number of pages.

d) the name and address of the client and project name if applicable;

e) identification of the method used;

f) a description of, the condition of, and unambiguous identification of the sample(s), including the client identification code;

g) the date of receipt of the sample(s) where this is critical to the validity and application of the results, date and time of sample collection, the date(s) of performance of the environmental test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours;

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 environmental test results with, where appropriate, the units of measurement, and any failures identified; identification of whether data are calculated on a dry weight or wet weight basis; identification of the reporting units, such as µg/l or mg/kg; and for Whole Effluent Toxicity, identification of the statistical package used to provide data;

j) the name(s), function(s) and signature(s) or equivalent electronic identification of person(s) authorizing the test report, and date of issue;

k) a statement to the effect that the results relate only to the samples;

l) at the laboratory's discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;

m) laboratories accredited to be in compliance with these standards shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

5.10.3 Supplemental Information for Test Reports

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 (such as failed QC), additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions and any non-standard conditions that may have affected the quality of results, including the use and definitions of data qualifiers;

b) where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;

c) where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed when a client's instruction so requires;

d) where appropriate and needed, opinions and interpretations (see Section 5.10.4);

e) additional information that may be required by specific methods, clients or groups of clients;

f) qualification of numerical results with values outside the working range.

5.10.3.2 In addition to the requirements listed in Sections 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 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.

5.10 DOE-1

Opinions and interpretations shall not be included in the report without notification of the client. Opinions and interpretations shall be only in the case narrative.

5.10.5 Environmental Testing Obtained from Subclients

When the test report contains results of tests performed by subclients, these results shall be clearly identified by subclient name or applicable accreditation number. The subclient shall report the results in writing or electronically. The laboratory shall make a copy of the subclient's report available to the client when requested by the client.

5.10.6 Electronic Transmission of Results

In the case of transmission of environmental test results by telephone, telex, facsimile or other electronic or electromagnetic means, the requirements of this document shall be met and all reasonable steps shall be taken to preserve confidentiality (see also Section 5.4.7).

5.10.7 Format of Reports

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

5.10.8 Amendments to Test Reports

a) Material amendments to a test report after issue shall be made only in the form of a further document, or data transfer, which includes the statement:

b) "Supplement to Test Report, serial number ... [or as otherwise identified]", or an equivalent form of wording.

c) Such amendments shall meet all the requirements of this document.

d) When it is necessary to issue a complete new test report, the new test report shall be uniquely identified and shall contain a reference to the original report that it replaces.

6.0 DOE-1

6.0 Hazardous and Radioactive Materials Management and Health and Safety Practices

DOE is concerned with ensuring that analytical laboratories handling DOE samples and analysis- derived waste conduct these operations in a manner that is protective of human health and the environment. DOE frequently sends samples with hazardous and/or radioactive constituents that require special handling to avoid worker, public, and environmental vulnerabilities and risks. The emphasis of DOE on general safety in the workplace is paramount. DOE chooses to use only those analytical laboratories that can demonstrate management controls and good health and safety practices.

All DOE sites submitting environmental and waste samples to environmental laboratories shall disclose known or suspected hazards associated with the samples. Based on a good faith effort based on available process knowledge or other information, hazards (radiological, toxicity or biological) shall be provided to the receiving laboratories prior to shipment of the samples unless prior arrangements have been made regarding sample receipt. Laboratories shall determine their ability to receive the samples. Laboratories shall have the appropriate capabilities, procedures, or licenses to receive samples from a DOE site. After receipt of any sample, the laboratories shall assume the responsibility and liability for the safe and compliant management of all samples, including regulatory storage and disposal of all samples and associated derived wastes. Some DOE sites permit the return of sample residuals and prior arrangements must be established prior to the receipt of samples. Derived wastes must be disposed by the laboratory.

6.1 Sample Receiving (see Section 5.8)

6.2 Radioactive Materials Management and Control

6.2.1 The laboratory shall comply with all applicable federal and state regulations governing radioactive materials control and radiological protection.

6.2.2 The radioactive materials license shall authorize possession of isotopes, quantity, physical form and use of radioactive material sufficient for the laboratory's scope of work in support of DOE sites.

6.2.3 The laboratory shall have facilities and current procedures in place to handle the isotopes, quantity and physical form of radioactive material specified on the radioactive material license. The laboratory shall ensure adherence to all radioactive materials license and procedural requirements.

6.2.4 The Radiation Safety Officer (RSO) listed in the Radioactive Materials License shall be available to monitor the radioactive materials and control programs and provide rapid response to any radiological emergencies. The laboratory shall

have an alternate or backup RSO that shall have the necessary training and experience to perform the duties of the RSO in the event that the RSO is not available.

6.2.5 The laboratory shall have in place a radioactive materials inventory program capable of tracking standards, tracers and all radiological samples. The radioactive material inventory shall be updated according to the schedule established by laboratory Radioactive Materials License. If no schedule is established by the license, then the laboratory shall update the inventory within seven days of receipt of radioactive materials.

6.2.6 Radioactive and mixed wastes shall be segregated from non-radioactive waste.

6.3 TSCA materials

6.3.1 The laboratory shall comply with all federal regulations governing TSCA materials control and protection.

6.3.2 The laboratory shall segregate all radioactive TSCA materials from all other analytical samples and residues (as defined in Section 3.0, “Terms, Definitions and Acronyms”).

6.3.3 The laboratory shall have a procedure for return of radioactive TSCA materials for which there is no commercial treatment or disposal options to the DOE client.

6.4 Laboratory Health and Safety

6.4.1 The laboratory shall comply with all state and federal regulations governing laboratory health and safety.

6.4.2 A laboratory safety inspection program is in place that includes routine walk-downs of laboratory areas for safety related concerns. (See also Section 5.3)

6.4.3 Chemical hazards labeling on chemical containers is in accordance with the laboratory’s approved Chemical Hygiene Plan.

6.4.4 On an annual frequency, all visitors, maintenance personnel and auditors shall have a documented safety orientation prior to entering the laboratory. All visitors shall be briefed on the safety practices and policies.

6.4.5 The laboratory shall have a Hazardous Waste Operator and Emergency Response (HAZWOPER) trained person on staff. Backup personnel with appropriate training for the Emergency Response (HAZWOPER) trained personnel shall be required.

6.4.6 The laboratory shall have reentry procedures defined in the Emergency Action

Plan.

6.5 Waste Management and Disposal

6.5.1 The laboratory shall comply with all federal and state regulations governing waste management and disposal.

6.5.2 The laboratory shall have a waste management plan in place which is capable of:

a) Identifying all waste streams generated by the laboratory including universal wastes such as batteries, thermostats, etc.;
b) Identifying the process for managing and disposition of the various waste streams; and
c) Tracking the disposition of waste samples by Sample Delivery Group (SDG).

6.5.3 The waste management plan shall include (but not limited to) the following:

a) Administrative programs to demonstrate compliance for effluent discharges as required by regulatory agencies and applicable DOE Orders;
b) Training procedures, schedules and management of training records in the areas of waste management, shipping, waste handling and radioactive materials control;
c) Radioactive volumetric and surface release policies;
d) Permits and licenses to handle hazardous and radioactive waste;
e) Policy or direction on how to conduct waste brokering and Transport, Storage and Disposal Facility (TSDF) evaluation to ensure proper disposition of waste;
f) Tracking of individual sample containers from receipt to final disposition
g) Accountability for samples consumed during analysis will be maintained
h) Waste minimization and pollution prevention programs including substitution (when permitted), segregation, recycling, etc.

Waste brokering and TSDF evaluations shall be based upon the results of a site visit to the waste facility or a desktop review that includes information from audits of the facilities conducted by state or federal agencies. The evaluation shall include liability coverage, financial stability, any notices of violations from the last three years, relevant permits and licenses to accept the waste, and other relevant information. Reviews of waste brokering and TSDF evaluations shall be performed every three years, unless there are changes in the facilities operations that require the reviews to be conducted on a more frequent basis (NOVs, change of ownership, notices of fines and penalties, etc,). (Refer to EPA public domain ECHO and Envirofacts websites for information on TSDFs.)

6.5.4 The laboratory shall remove or deface all sample container labels prior to container disposal such that they are rendered illegible.

6.5.5 Analytical process waste is segregated and removed to a designated storage area to minimize the potential for cross contamination.

6.5.6 Laboratory analysis derived waste characterization is repeated at a frequency adequate to account for all known variations in the waste streams.

6.5.7 The laboratory shall have provisions for the disposition of excess samples.

6.5.8 For excess samples that are bulked and drain disposed, the laboratory is aware of the requirements for the receiving POTW or wastewater treatment system and has a program that meets and demonstrates compliance with these requirements.

QUALITY SYSTEMS
FOR ANALYTICAL SERVICES

APPENDIX A

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Appendix A - REFERENCES

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QUALITY SYSTEMS
FOR ANALYTICAL SERVICES

APPENDIX B

(RESERVED)

QUALITY SYSTEMS
FOR ANALYTICAL SERVICES

APPENDIX C

DEMONSTRATION OF CAPABILITY

Appendix C - DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR DEMONSTRATION OF CAPABILITY

A Demonstration of Capability (DOC) must be made prior to using any test method, and at any time there is a change in instrument type, personnel or test method (see Section 5.4.2.2).

Note: In laboratories with specialized "work cells" (a well defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available quality system matrix (a sample in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., drinking water, solids, biological tissue and air. However, before any results are reported using this method, actual sample spike results may be used to meet this standard, i.e., at least four consecutive matrix spikes within the last twelve months. In addition, for analytes that do not lend themselves to spiking, e.g., Total Suspended Solids (TSS), the demonstration of capability may be performed using QC samples.

All demonstrations shall be documented through the use of the form in this Appendix. All data applicable to the demonstration need not be attached to the form, but must be retained and available.

When an analyte not currently found on the laboratory's list of accredited analytes is added to an existing accredited test method, an initial evaluation must be performed for that analyte.

The following steps shall be performed if required by mandatory test method or regulation. It is the responsibility of the laboratory to document that other approaches to DOC are adequate. This shall be documented in the laboratory's Quality Manual, e.g., for Whole Effluent Toxicity Testing, see Section 2.1.a.1 of this Appendix.

a) A QC sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

C.1 DOE-1

The test samples shall be a representative matrix as those expected in field samples.

b) The analyte(s) shall be diluted in a volume of clean quality system matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration of 1-4 times the limit of quantitation.

c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory must assess performance against established and documented criteria.

e) Compare the information from Section d above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters does not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to Section 1 or 2 below.

   1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section c above.

   2) Beginning with Section c above, repeat the test for all parameters that failed to meet criteria. 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 Section c above.

C.2 CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee (see Sections 5.2.5 and 4.12.2.5.4.b).

Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix: (examples: laboratory pure water, soil, air, solid, biological tissue)
Method number, SOP#, Rev#, and Analyte, or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Technical Director's Name and Title	Signature	Date
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance evaluation.
Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

Note: DOE requirements are identified in gray boxes. In cases where DOE requirements differ from the NELAC requirements, DOE requirements supersede the NELAC requirements.

C.3 INITIAL TEST METHOD EVALUATION

For all test methods other than toxicity and microbiology the requirements of Sections 3.1 and 3.2 of this Appendix apply. For Toxicity testing, and Microbiology testing, the initial test method evaluation requirements are contained at Appendix D, Sections 2 and 3, respectively. For the evaluation of precision and bias (Section 3.3 of this Appendix), the requirements of Section 3.3.a of this Appendix apply to standard methods. The requirements of Section 3.3.b of this
Appendix apply to the methods referenced therein.

C.3.1 Limit of Detection (LOD)

C.3 DOE-1

LOD is equivalent to Method Detection Limit (MDL).

a) The laboratory shall determine the LOD for the method for each target analyte of concern in the quality system matrices. All sample-processing steps of the analytical method shall be included in the determination of the LOD.

b) The validity of the LOD shall be confirmed by qualitative identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3 times the LOD for single analyte tests and 1-4 times the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data.

c) An LOD study is not required for any component for which spiking solutions or QC samples are not available such as temperature, or, when test results are not to be reported to the LOD (versus the limit of quantitation or working range of instrument calibration), according to Appendix D, Sections 1.2, 4.5, 5.4, and 6.6 . Where an LOD study is not performed, the laboratory may not report a value below the Limit of Quantitation.

C.3.2 Limit of Quantitation (LOQ)

C.3 DOE-2

LOQ is equivalent to Practical Quantitation Limit (PQL)

The laboratory shall determine the LOQ for each analyte of concern according to a defined, documented procedure.

a) The LOQ study is not required for any component or property for which spiking solutions or QC samples are not commercially available or otherwise inappropriate (e.g., pH).

b) The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objectives for accuracy. This single analysis is not required if the bias and precision of the measurement system is evaluated at the LOQ.

C.3.3 Evaluation of Precision and Bias

a) Standard methods -- The laboratory shall evaluate the Precision and Bias of a Standard Method for each analyte of concern for each quality system matrix according to the single-concentration four-replicate recovery study procedures in Section 1 of this Appendix (above) (or alternate procedure documented in the quality manual when the analyte cannot be spiked into the sample matrix and QC samples are not commercially available).

b) Non-standard methods -- For Laboratory-developed test methods or non-standard test methods as defined in Sections 5.4.3 and 5.4.4 that were not in use by the laboratory before July 2003, the laboratory must have a documented procedure to evaluate precision and bias. The laboratory must 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 must evaluate the method across the analytical calibration range of the method. The laboratory must also evaluate precision and bias in the relevant quality system matrices and must 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:

Analyze QC samples in triplicate containing the analytes of concern at or near the limit of quantitation, at the upper-range of the calibration (upper 20%) and at a mid-range concentration. Process these samples on different days as three sets of samples through the entire measurement system for each analyte of interest. Each day, one QC sample at each concentration is analyzed. A separate method blank shall be subjected to the analytical method along with the QC samples on each of the three days. (Note that the three samples at the LOQ concentration can

demonstrate sensitivity as well.) For each analyte, calculate the mean recovery for each day, for each level over days, and for all nine 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.

A validation protocol such as the Tier I, Tier II, and Tier III requirements in U.S. EPA Office of Water's Alternate Test Procedure (ATP) approval process.

C.3.4 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.

C.3 DOE-3

The checks shall also include column separation efficiency for radiochemical separations.

QUALITY SYSTEMS
FOR ANALYTICAL SERVICES

APPENDIX D

ESSENTIAL QUALITY CONTROL REQUIREMENTS

Appendix D - ESSENTIAL QUALITY CONTROL (QC) REQUIREMENTS

The QC protocols specified by the laboratory's method manual (see Section 5.4.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in this Appendix are incorporated into their method manuals and/or the Laboratory Quality Manual.

All QC measures shall be assessed and evaluated on an ongoing basis and QC acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of this QSAS, e.g., Section 5.9.2, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing, Whole Effluent Toxicity (WET) testing, microbiology testing, radiochemical testing and air testing.

D.1 CHEMICAL TESTING

D.1 DOE-1

When DOE requires SW-846 Methods, the additional QA in this appendix is required.

D.1.1 Positive and Negative Controls

D.1.1.1 Negative Control - Method Performance

a) Purpose: The method blank is used to assess 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) Frequency: The method blank shall be analyzed at a minimum of 1 per preparation batch. 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 20 environmental samples.

c) Composition: 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) Evaluation Criteria and Corrective Action: While the goal is to have no detectable contaminants, each method blank must 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:

1) The concentration of a targeted analyte in the blank is at or above the reporting limit as established by the test method or by regulation, AND is greater than 1/10 of the amount measured in any sample.

2) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

3) When a blank is determined to be contaminated, the cause must 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 must be documented.

D.1 DOE-2

The client shall be contacted to discuss implementation of corrective action and the documentation of the corrective action shall be included in the case narrative.

D.1.1.2 Positive Control - Method Performance

D.1.1.2.1 Laboratory Control Sample (LCS)

a) Purpose: 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 reanalysis or the results reported with appropriate data qualifying codes.

b) Frequency: The LCS shall be analyzed at a minimum of 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 20 environmental samples.

c) Composition: The LCS is a quality system matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. Note: the matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. Alternatively, the LCS may consist of a media containing known and verified concentrations of analytes or as 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 test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components the laboratory shall spike per the following:

For those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene, or PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should 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 ensure that all targeted components are included in the spike mixture over a two-year period.

1) For methods that include 1-10 targets, spike all components;

2) For methods that include 11-20 targets, spike at least 10 components or 80%, whichever is greater;

3) For methods with more than 20 targets, spike at least 16 components.

D.1 DOE-3

For soil samples for metals or radiochemical analysis where a digestion is required to get the analyte into solution, the use of a spike (which is already in solution) of an analyte free matrix will not meet the purpose of an LCS. Since the analyte is already in solution, the performance of the digestion is not evaluated. The only acceptable LCS for metals and radionuclides in soils that include digestions is a CRM. While a CRM with all requested analytes may not be available, a CRM with a representative amount of analytes of concern should be used. When the suggested solid CRM is not available for the LCS, a matrix spike and LCS pair may be used. Composition of the soil LCS shall be documented in the case narrative.

d) Evaluation Criteria and Corrective Action: 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 test 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.

A 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 a LCS determined to be out-of-control shall be considered suspect and the samples reprocessed and reanalyzed or the data reported with appropriate data qualifying codes.

e) 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 (3 standard deviations), but within the ME limits. ME limits are between 3 and 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 11 analytes.

The number of allowable marginal exceedances is as follows:

1) >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit;

2) 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit;

3) 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit;

4) 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit;

5) 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit;

6) <11 analytes in LCS, no analytes allowed in ME of the LCS control limit.

Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken. Laboratories must have a written procedure to monitor the application of marginal exceedance allowance to the LCS to ensure random behavior.

D.1 DOE-4

The ME allowance applies only to organic analysis.

D.1.1.3 Sample-Specific Controls

The laboratory must 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 test 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, evaluating and reporting results based on performance of the QC samples.

D.1.1.3.1 Matrix Spike; Matrix Spike Duplicates

a) Purpose: 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) Frequency: The frequency of the analysis of matrix-specific samples shall be determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the test method.

c) Composition: The components to be spiked shall be as specified by the mandated test 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:

For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test 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 ensure that all targeted components are included in the spike mixture over a 2-year period.

1) For methods that include 1-10 targets, spike all components;

2) For methods that include 11-20 targets, spike at least 10 components or 80%, whichever is greater;

3) For methods with more than 20 targets, spike at least 16 components.

D.1 DOE-5

Analytical method being performed under a specific regulatory method, the requirements for the calculation of the RPD will be performed according to that method specific requirement, e.g., CLP. In the absence of specific regulatory method, the requirements of SW-846 shall be followed for the calculation of the RPD.

d) Evaluation Criteria and Corrective Action: 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 test method or established client criteria. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix spike results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

D.1.1.3.2 Matrix Duplicates

a) Purpose: 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 provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.

b) Frequency: The frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the mandated test method.

c) Composition: Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.

d) Evaluation Criteria and Corrective Action: The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e.g., absolute differences). The laboratory shall document the calculation for relative percent difference or other statistical treatments.

Results are compared to the acceptance criteria as published in the mandated test method or established client criteria. 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 reported with appropriate data qualifying codes.

D.1.1.3.3 Surrogate Spikes

a) Purpose: Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for every sample matrix.

b) Frequency: Except where the matrix precludes its use or when not commercially available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate test methods.

c) Composition: Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method or MQO. 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.

d) Evaluation Criteria and Corrective Action: The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory should determine internal criteria and document the method used to establish the limits. Surrogates outside the acceptance criteria must 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 should include appropriate data qualifiers.

D.1.2 Limit of Detection and Limit of Quantitation

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

D.1.2.1 Limit of Detection (LOD)

D.1 DOE-6

LOD is equivalent to MDL

The laboratory shall utilize a test method that provides an LOD that is appropriate and relevant for the intended use of the data. An LOD is not required for a test method when test results are not reported outside of the calibration range. LODs shall be determined by the protocol in the mandated test method or applicable regulation. If the protocol for determining LODs is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

D.1 DOE-7

The protocol for determining LODs shall be documented.

a) The LOD shall be initially determined for the compounds of interest in each test method in a quality system matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the LOD must be determined in the quality system matrix of interest (see definition of matrix).

b) LODs must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

c) The laboratory must have established procedures to relate LOD with LOQ.

D.1 DOE-8

All sample processing steps of the analytical method shall be included in the determination of the LOD.

d) The LOD must be verified annually for each quality system matrix, method and analyte according to the procedure specified in Appendix C, Section 3.

D.1.2.2 Limit of Quantitation (LOQ)

D.1 DOE-9

LOQ is equivalent to PQL.

a) Any established LOQ must be above the LOD.

b) The LOQ must be verified annually for each quality system matrix, method and analyte according to the procedure specified in Appendix C, Section 3. Alternatively, the annual LOQ verification is not required if the LOD is reevaluated or verified according to Section 1.2.d of this Appendix (above).

D.1.3 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.1.4 Quality of Standards and Reagents

a) The source of standards shall comply with Section 5.6.2.2.

b) Reagent Quality, Water Quality and Checks:

1) Reagents - 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 test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

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

3) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.

D.1.5 Selectivity

a) 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.

D.1 DOE- 10

The checks may also include partition coefficient studies.

The laboratory shall document acceptance criteria.

b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

c) The laboratory shall document acceptance criteria for mass spectral tuning.

D.1.6 Constant and Consistent Test Conditions

a) The laboratory shall ensure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

D.1 DOE-11

D.1.7 Total Uranium by Laser Induced Kinetic Phosphorescence

a) Sample Preparation, Glassware, Water

1) Water samples shall be at least evaporated to dryness and wet-ashed as described in ASTM Specification D 5174-91, Trace Uranium by Pulsed-Laser Phosphorimetry, prior to KPA measurement.

2) For each sample, both the sample and sample-plus-spike shall be measured to demonstrate that there are no quenching interferences.

3) For all low-level uranium analysis, prior to initial use, all new glassware with the exception of cuvettes used in KPA measurement, shall be soaked in hot 8M nitric acid for at least two hours and then in room temperature 8M nitric acid overnight.

4) Reagent-grade water shall be used to prepare, but is not limited to: standards, preparation of all reagents, and for final rinsing of glassware for items used in the determination of low-level uranium.

5) The sample preparation must yield samples such that lifetimes shall fall in the range of 150 µs to 350 µs.

b) Method Detection Limit (MDL) Determination

1) The method detection limit (MDL) is determined for each matrix analyzed and represents the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The

MDL is estimated by analyzing a minimum of seven replicate samples containing uranium at a concentration less than or equal to three times the calculated MDL. The MDL study samples shall be subjected to all preparation and analysis steps and shall reflect the volume used for routine sample analysis.

MDL Calculation: The following equation shall be used to calculate the MDL:

$$MDL = [(T_{stud} * S_b) * DIL] / V$$

where:

T_{stud} = students' t value appropriate for a 99% confidence level and alpha standard deviation estimate with n-1 degrees of freedom (3.143 for seven standards)
S_b = standard deviation(s) of the replicate detection limit standard measurements
DIL = dilution factor (ratio of total sample taken for
V = sample volume or weight (liters or grams)
Units are in µg/l or µg/g

The method of implementation of MDL studies shall be described in detail in an SOP.

The MDL shall be determined at least quarterly and any time significant system components (laser, optics, power supply, etc.) are service, or when instrument performance indicates that the detection threshold may have changed.

If the concentration of the MDL test samples is less than one-third the calculated MDL, the MDL study shall be iteratively repeated until the MDL determined is less than or equal to three times the spiking concentration of the test samples used to determine the MDL.

2) The sample volume used for routine sample analysis shall be equivalent to that used for the MDL study. If a given sample size differs from that used in the determination of the MDL, this shall be addressed in the Case Narrative. It is not expected that sample volume should vary.

3) The analyte MDL, as calculated above, must be less than or equal to the RDL at the lowest dilution factor.

i. Sample and Analyte-Specific Conditions - that require reanalysis for a particular sample beginning with KPA measurement. If succeeding KPA measurement does not provide an adequate result, another aliquot must be started with the sample preparation.

4) The lifetime of the phosphorescence is less than 150 µs or greater than 350 µs.

5) The linear regression coefficient of the decay plot is greater than 0.96 for samples where the measured concentration is greater than the RDL. Every effort should be made to keep this greater than 0.98. It is expected that, for most samples, this will be greater than 0.99.

6) If the standard addition recovery is less than 90%, reanalysis is required:
Standard Addition Recovery =
(Conc. Spike-Conc. Sample)/ (Std.Conc. × Std.Vol) × 100
(Sample Vol.+Std.Vol.)

7) Analyte concentration is not in the range of the calibration curve used.

8) The Reference Ratio is less than 0.9 or greater than 1.1.

9) The Continuing Calibration Check Standard is not within 10% of the known value.

c) Instrument Calibration – The requirements set down in 5.5.2.2.1 f), h) and i) apply to KPA calibrations.

1) The KPA shall be calibrated daily when in use. The order for performing calibration shall be: a background, followed by calibration curve, followed by calibration check standard.

2) The background shall be sufficiently low to permit attaining RDLs.

3) At least three standards shall be used for each calibration range. The calibration range shall include the range of the samples to be measured. R^2 (linear regression coefficient) for the calibration curve shall be greater than or equal to 0.99.

4) The LCS shall be measured in the same calibration range as the samples in the batch. Observe the instrument manufacturer's recommendations for calibrating high and low ranges. If the

measurements are performed in more than one calibration range, then a separate LCS shall be prepared for each range.

d) Calibration Check Standard

1) A detection limit verification standard shall be analyzed following calibration and prior to analysis of samples. The concentration of the low-point standard shall be at or below the required detection limit. The relative bias for the low-point verification check shall be in the range -0.3 to +0.3.

2) A mid-point calibration verification standard shall be performed upon completion of calibration and subsequently after every 10 samples are analyzed using a separately prepared standard at a different concentration from the calibration standards. The relative bias of the calibration verification check shall be in the range -0.10 to +0.10.

D.2 TOXICITY TESTING

These standards apply to laboratories measuring the toxicity and/or bioaccumulation of contaminants in effluents (WET), receiving waters, sediments, elutriates, leachates and soils. In addition to the essential QC standards described below, some methods may have additional or other requirements based on factors such as the type of quality system matrix evaluated.

D.2.1 Positive and Negative Controls

a) Positive Control - Reference toxicant tests demonstrate a laboratory's ability to obtain consistent results with the test method and evaluate the overall health and sensitivity of test organisms over time.

1) The laboratory must demonstrate its ability to obtain consistent results with Standard Reference Toxicants (SRT) and complete an initial Demonstration of Capability (DOC) in order to attain accreditation in toxicity testing methods.

i. An initial DOC shall consist of five or more acceptable SRT tests for each test method, species and endpoint with different batches of organisms. Appropriate negative controls (water, sediment, or soil) shall be tested at the frequency and duration specified in the test method. Initial DOCs shall be prepared in accordance with the requirements of Appendix C.

ii. Initial DOC is established by maintenance of SRT test results on control charts. A laboratory shall record the control performance and statistical endpoints (such as NOEC or ECp) for each method species and endpoint on control charts. Initial DOC is established where 95% of the test results required in Section 2.1.a.1.i of this Appendix fall within the control limits established in accordance with Section 2.1.a.1.iii) of this Appendix and meet Test Acceptability Criteria (TAC). The laboratory shall evaluate precision (i.e., coefficient of variation, [CV]) or sensitivity (i.e., statistical minimum significant difference, [SMSD] measures [see Section 2.1.a.1.iv of this Appendix) for these tests against method-specific or (lacking the former) laboratory-derived criteria to determine validity of the initial DOC.

iii. 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). In case of highly variable point estimates that exceed method-specific criteria, the control chart limits are adjusted accordingly. 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).

iv. For endpoints that are point estimates the cumulative mean, CV is calculated. For endpoints from hypothesis tests, the SMSD is calculated. These values are maintained on a control chart.

2) Ongoing laboratory performance shall be demonstrated by routine SRT testing for each test method and species and endpoint in accordance with the minimum frequency requirements specified in D.2.1.a.3.

i. Intralaboratory precision is determined on an ongoing basis through the use of control charts as established in D.2.1 a) 1) ii. 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.

ii. After initial laboratory DOC is determined, the control limits and CV for an individual test method, endpoints and species shall be adjusted as additional test results are obtained. After 20 data points are collected for a test method and species, the control chart is maintained using only the last 20 data points, i.e. each successive mean value and control limit is calculated using only the last 20 values.

iii. 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 that 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.

iv. Laboratories shall develop acceptance/rejection policies that are consistent with the test methods for SRT data and which consider 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.

v. In the case of reference toxicant data that fails 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.

3) 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):

i. For test methods conducted at a frequency of monthly or greater, SRT tests shall be conducted at an ongoing frequency of monthly.

ii. For test 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.

iii. 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 months.

iv. The DOC for an analyst shall be consistent with Section 5.2.6.c.3, but the frequency need not exceed the method specified requirements and those found in Section 2.1.a.3 of this Appendix.

4) These standards do not currently specify a particular reference toxicant and dilution series; however, if the state or permitting authority 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 test method and species must use the same reference toxicant, test concentrations, dilution water and data analysis methods. A dilution factor of 0.5 times or greater shall be used for both acute and chronic tests.

5) The reference toxicant tests shall be conducted following the same procedures as the environmental toxicity tests for which the precision is being evaluated~~,~~ unless otherwise specified in the test method e.g., 10-day sediment tests employ 96-h water-only reference toxicant tests. The test duration, laboratory dilution water, feeding, organism age, range and density, test volumes, renewal frequency, water quality measurements, and the number of test concentrations, replicates and organisms per replicate shall be the same as specified for the environmental toxicity test.

b) Negative Control - Control, Brine Control, Control Sediment, Control Soil or Dilution Water -

1) The standards for the use, type and frequency of testing of negative controls are specified by the test 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.

2) 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.

3) Test Acceptability Criteria (TAC) - The test acceptability criteria specified in the test method must 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.

D.2.2 Variability and/or Reproducibility

Intralaboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in Section 2.1.a of this Appendix (above).

D.2.3 Accuracy

This principle is not applicable to Toxicity Testing.

D.2.4 Test Sensitivity

a) The SMSD shall be calculated according to the formula specified by the test 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.

c) The SMSD shall be calculated and reported for only hypothesis test values, such as the NOEC or NOAEC.

D.2.5 Selection of Appropriate Statistical Analysis Methods

a) If required, methods of data analysis and endpoints are specified by language in the regulation, permit or the test method.

b) Dose Response Curves – The data shall be plotted in the form of a curve relating the dose of the chemical or concentration of sample to cumulative percentage of test organisms demonstrating a response such as death. Evaluation criteria shall be established for interpretation of concentration or dose response curves.

D.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in toxicity tests is specified in the test method except for 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 standards outlined in Section 5.5.2.1.d.

c) Only reagent-grade water collected from distillation or deionization units is used to prepare reagents.

D.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit or regulation.

D.2.8 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 must be adequate for the types and numbers of tests performed. The building must provide adequate cooling, heating and illumination for conducting testing and culturing; hot and cold running water must be available for cleaning equipment.

c) Air used for aeration of test solutions, dilution waters and cultures must 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) must be kept on file at the laboratory. When organisms are obtained from an outside source, the supplier must provide this same information.

e) Instruments 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. As these are support measurements, only the calibration and verification requirements specified in Section 5.5.2.1 apply. All measurements and calibrations shall be documented.

f) Test temperature shall be maintained as specified for the test method. Temperature control equipment must be adequate to maintain the required test temperature(s). The average daily temperature of the test solutions must be maintained within the method-specified range. The minimum frequency of measurement shall be once per 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 must 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. It is recognized that the analyte lists of some methods manuals may not include all potential toxicants, are based on estimates of chemical toxicity available at the time of publication, and may specify detection limits that are not achievable in all matrices. However, for those analytes not listed, or for which the measured concentration or LOD is greater than the method-specified limit, the laboratory must demonstrate that the analyte at the measured concentration or reported LOD does not exceed one tenth the expected chronic value for the most sensitive species tested and/or cultured. The expected chronic value is based on professional judgment and the best available scientific data. The "USEPA Ambient Water Quality Criteria Documents" and the EPA AQUIRE database provide guidance and data on acceptability and toxicity of individual metals and organic compounds.

i) The quality of the food used for testing or culturing must 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 must 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 test method. All test chambers used in a test must be identical.

l) Test organisms shall be fed the quantity and type food or nutrients specified in the test method. They shall also be fed at the intervals specified in the test methods.

m) All organisms in a test must be from the same source. Where available, certified seeds are used for soil tests.

n) All organisms used in tests, or used as broodstock to produce neonate test organisms (e.g., cladocerans and larval fish), must appear healthy, show no signs of stress or disease and exhibit acceptable survival (90% or greater) during the 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 must be non-toxic and cleaned as described in the test methods. Materials must not reduce or add to sample toxicity. Appropriate materials for use in toxicity testing and culturing are described in the referenced manuals.

p) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the test 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 organisms received. These observations shall adequately address the 24-hour time period referenced in Section 2.8.n of this Appendix (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 must be as specified in the test method. Supporting information, such as hatch dates and times, times of brood releases and metrics (e.g., 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 36 hours; samples may be used for renewal up to 72 hours after first use except as prescribed by the method and approved by the regulatory agency having authority for program oversight.

t) All samples shall be chilled to 0 to 6°C during or immediately after collection (see requirements in Section 5.8.3.1) except as prescribed by the method and approved by the regulatory agency having authority for program oversight.

u) Organisms used in a given test must be from the same batch.

v) All tests shall have the minimum number of replicates per treatment as prescribed by the method.

w) The control population of Ceriodaphnia in chronic effluent or receiving water tests shall contain no more than 20% males.

x) The culturing of C. dubia shall be adequate such that blocking by parentage can be established.

y) Dissolved oxygen and pH in aquatic tests shall be within acceptable range at test initiation and aeration (minimal) is provided to tests if, and only if, acceptable dissolved oxygen concentrations cannot be otherwise maintained, or if specified by the test method.

z) Test soils or sediments must be within the geochemical tolerance range of the test organism.

aa) 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 test method). The acceptability of the test shall depend on the experience and professional judgment of the technical director and the permitting authority.

D.3 MICROBIOLOGY TESTING

These standards apply 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.

D.3.1 Sterility Checks and Blanks, Positive and Negative Controls

a) Sterility Checks and 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, inadequate sterilization, or environmental exposure.

1) A sterility blank shall be analyzed for each lot of pre-prepared, ready-to-use medium (including chromofluorogenic reagent) and for each batch of medium prepared in the laboratory. This shall be done prior to first use of the medium.

2) For filtration technique, the laboratory shall conduct one beginning and one ending sterility check for each laboratory sterilized filtration unit used in a filtration series. The filtration series may include single or multiple filtration units, which have been sterilized prior to beginning the series. For pre-sterilized single-use funnels, a sterility check shall be performed on one funnel per lot. The filtration series is considered ended when more than 30 minutes elapses between successive filtrations. During a filtration series, filter funnels must be rinsed with three 20-30 ml portions of sterile rinse water after each sample filtration. In addition, laboratories must insert a sterility blank after every 10 samples or sanitize filtration units by UV light after each sample filtration.

3) For pour plate technique, sterility blanks of the medium shall be made by pouring, at a minimum, one uninoculated plate for each lot of pre-prepared, ready-to-use media and for each batch of medium prepared in the laboratory.

4) Sterility checks on sample containers shall be performed on at least one container for each lot of purchased, pre-sterilized containers. For containers prepared and sterilized in the laboratory, a sterility check shall be performed on one container per sterilized batch with non-selective growth media.

5) A sterility blank shall be performed on each batch of dilution water prepared in the laboratory and on each batch of pre-prepared, ready-to-use dilution water with non-selective growth media.

6) At least one filter from each new lot of membrane filters shall be checked for sterility with non-selective growth media.

b) Positive Controls

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).

1) Each pre-prepared, ready-to-use lot of medium (including chromofluorogenic reagent) and each batch of medium prepared in the laboratory shall be tested with at least one pure culture of a known positive reaction. This shall be done prior to first use of the medium.

c) Negative Controls

Negative culture controls demonstrate that the medium does not support the growth of non-target organisms or does not demonstrate the typical positive reaction of the target organism(s).

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 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.

D.3.2 Test Variability/Reproducibility

For test methods that specify colony counts, such as membrane filter or plated media, duplicate counts shall be performed monthly on one positive sample, for each month that the test is performed. If the laboratory has two or more analysts, each analyst shall count typical colonies on the same plate. Counts must be within 10% difference to be acceptable. In a laboratory with only one microbiology analyst, the same plate shall be counted twice by the analyst, with no more than 5% difference between the counts.

D.3.3 Method Evaluation

a) Laboratories are required to demonstrate proficiency with the test method prior to first use. This shall be achieved by comparison to a method already approved for use in the laboratory, or by analyzing a minimum of ten spiked samples whose quality system matrix is representative of those normally submitted to the laboratory, or by analyzing and passing one PE series provided by an approved proficiency sample provider. The laboratory shall maintain this documentation as long as the method is in use and for at least 5 years past the date of last use.

b) Laboratories shall participate in the PE programs identified by NELAP (Sections 4.1.5.k or 5.9.1). The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data.

D.3.4 Test Performance

a) All growth and recovery media must be checked to assure that the target organism(s) respond in an acceptable and predictable manner (see Section 3.1.b of this Appendix).

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.

D.3.5 Data Reduction

The calculations, data reduction and statistical interpretations specified by each test method shall be followed.

D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared from commercial dehydrated powders or may be purchased ready to use. Media may be prepared by the laboratory from basic ingredients when commercial media are not available or when it can be demonstrated that commercial media do not provide adequate results. Media prepared by the laboratory from basic ingredients must be tested for performance (e.g., for selectivity, sensitivity, sterility, growth promotion, growth inhibition) prior to first use. Detailed testing criteria information must be defined in either the laboratory's test methods, SOPs, Quality Manual, or similar documentation.

b) Reagents, commercial dehydrated powders and media shall be used within the shelf-life of the product and shall be documented according to Section 5.6.3.5.

c) Distilled water, deionized water or reverse-osmosis produced water free from bactericidal and inhibitory substances shall be used in the preparation of media, solutions and buffers. The quality of the water shall be monitored for chlorine residual, specific conductance, 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 for metals and the Bacteriological Water Quality Test (to determine presence of toxic agents or growth promoting substances) shall be performed annually. Results of these analyses shall meet the specifications of the required method and records of analyses shall be maintained for five years. (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 Type I or Type II reagent water.)

d) Media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer's instructions or the test method. Documentation for media prepared in the laboratory shall include date of preparation, preparer's initials, type and amount of media prepared, manufacturer and lot number, final pH of the media, and expiration date. Documentation for media purchased pre-prepared, ready-to-use shall include manufacturer, lot number, type and amount of media received, date of receipt, expiration date of the media, and pH of the media.

D.3.7 Selectivity

a) 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 NELAP Accrediting Authority. Microorganisms may be single use preparations or cultures maintained by documented procedures that demonstrate the continued purity and viability of the organism.

1) Reference cultures may be revived (if freeze-dried) or transferred from slants and subcultured 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 must not be re-frozen and re-used.

2) Working stocks shall not be sequentially cultured more than five times and shall not be subcultured to replace reference stocks.

D.3.8 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. Plants, food, and drink shall be prohibited from the laboratory work area.

b) Laboratory Equipment

1) Temperature Measuring Devices

Temperature measuring devices, such as liquid-in-glass thermometers, thermocouples, and platinum resistance thermometers used in incubators, autoclaves and other equipment, shall be of the appropriate quality to meet specification(s) in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement and they shall be calibrated to national or international standards for temperature (see Section 5.6.2.2). Calibration shall be done at least annually.

2) Autoclaves

i. The performance of each autoclave shall be initially evaluated 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.

ii. Demonstration of sterilization temperature shall be provided by use of continuous temperature recording device or by use of a maximum registering thermometer with every cycle. Appropriate biological indicators shall be used once per month to determine effective sterilization. Temperature sensitive tape shall be used with the contents of each autoclave run to indicate that the autoclave contents have been processed.

iii. Records of autoclave operations shall be maintained 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.

iv. Autoclave maintenance, either internally or by service contract, shall be performed annually and shall include a pressure check and calibration of temperature device. Records of the maintenance shall be maintained in equipment logs.

v. The autoclave mechanical timing device shall be checked quarterly against a stopwatch and the actual time elapsed documented.

3) Volumetric Equipment

Volumetric equipment shall be calibrated as follows:

i. equipment with movable parts such as automatic dispensers, dispensers/diluters, and mechanical hand pipettes shall be verified for accuracy quarterly.

ii. equipment such as filter funnels, bottles, non-class A glassware, and other marked containers shall be calibrated once per lot prior to first use.

iii. the volume of the disposable volumetric equipment, such as sample bottles, disposable pipettes, and micropippette tips, shall be checked once per lot.

4) UV Instruments

UV instruments, used for sanitization, shall be tested quarterly for effectiveness with an appropriate UV light meter or by plate count agar spread plates. 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.

5) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements (see Section 5.5.2.1.d).

6) Incubators, Water Baths, Ovens

i. The stability and uniformity of temperature distribution and time required after test sample addition to re-establish equilibrium conditions in incubators and water baths shall be established. Temperature of incubators and water baths shall be documented twice daily, at least four hours apart, on each day of use.

ii. Ovens used for sterilization shall be checked for sterilization effectiveness monthly with appropriate biological indicators. Records shall be maintained for each cycle that include date, cycle time, temperature, contents and analyst's initials.

7) Labware (Glassware and Plasticware)

i. The laboratory shall have a documented procedure for washing labware, if applicable. Detergents designed for laboratory use must be used.

ii. Glassware shall be made of borosilicate or other non-corrosive material, free of chips and cracks, and shall have readable measurement marks.

iii. 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 annually, and each time the laboratory changes the lot of detergent or washing procedures.

iv. Washed labware shall be tested at least once daily, each day of washing, for possible acid or alkaline residue by testing at least one piece of labware with a suitable pH indicator such as bromothymol blue. Records of tests shall be maintained.

D.4 RADIOCHEMICAL TESTING

These standards apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. These procedures for radiochemical analysis may involve some form of chemical separation followed by detection of the radioactive decay of analyte (or indicative progenies) and tracer isotopes where used. For the purpose of these standards, procedures for the determination of radioactive isotopes by mass spectrometry (e.g. ICP-MS or TIMS) or optical (e.g., KPA) techniques are not addressed herein.

D.4 DOE-1

KPA is addressed in Section 1.7 of this Appendix.

D.4.1 Negative and Positive Controls

D.4 D0E-2

QC Sample Preparation: All samples and QC samples in each Analytical Batch shall be prepared concurrently and in the same manner.

QC Sample Counting: All QC samples shall be counted and analyzed in the same manner as the samples in the Analytical Batch, in the same time frame and using the same instrument calibration parameters, instrument analysis algorithms, etc.

The "same time frame" implies that where multiple detectors are used and are sufficient to count the entire batch at the same time, the entire batch is counted at the same time. If the number of detectors is not sufficient to count the entire batch at the same time, then samples shall be counted consecutively on the available detector(s).

The "same instrument calibration parameters, instrument analysis algorithms, etc.," implies that these parameters for a given instrument shall not be changed for the samples in that batch. It is understood that for multiple detectors, the parameters may not be identical.

a) Negative Controls

1) Method Blank - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the QC measures to be used to assess the batch. The method blank result shall be assessed against the specific acceptance criteria (see Section 5.4.1.2.b.18) specified in the laboratory method manual (see Section 5.4.1.2). When the specified method blank acceptance criteria is not met, the specified corrective action and contingencies (see Sections 5.4.1.2.b.19 and 20) shall be followed and results reported with appropriate data qualifying codes. The occurrence of a failed method blank acceptance criteria and the actions taken shall be noted in the laboratory report (see Section 5.10.3.1.a).

D.4 DOE-3

Batch blanks shall be counted for a sufficient time to meet the required detection limit, except in the case where the achieved MDA is calculated from the standard deviation of a blank population. In this case, the batch blanks shall be counted for the same count time as the samples.

The batch blank matrix shall be the same as the samples, as can be

reasonably achieved and shall be documented in the Case Narrative.

D.4 DOE-4

Blank Acceptance Criteria:

The method blank results shall be less than 2 times the blank Combined Standard Uncertainty (CSU). (Note: CSU is equivalent to Total Propagated Uncertainty (TPU)) For decisions regarding detectability of analytes in the sample, 1.65 times the method blank CSU shall be used to evaluate method blank acceptance.

The Batch Blank MDA shall be less than the RDL.

If these criteria are not met, corrective actions shall be taken (e.g., recount, interferent cleanup, as appropriate), unless all sample results are greater than 5 times the blank activity. If the criteria are still not met, then the samples shall be reanalyzed.

2) In the case of gamma spectrometry, generally a non-destructive analysis, a method blank shall be prepared using a calibrated counting geometry similar to that used for the samples. The container of the appropriate geometry can be empty or filled to similar volume to partially simulate gamma attenuation due to a sample matrix.

D.4 DOE-5

The following batch blank matrices shall be used for all radiochemistry analyses:

Distilled or deionized water, radon free
Characterized solid material representative of the sample matrix

Filters
Physically and chemically identical filter media, analyte free (if supplied to the laboratory by customer)

3) There shall be no subtraction of the required method blank (see Section 4.1.a.1 of this Appendix) result from the sample results in the associated preparation or analytical batch unless permitted by method or program. This does not preclude the application of any correction factor (e.g., instrument background, analyte presence in tracer, reagent impurities, peak overlap, etc.) to all analyzed samples, both program/project submitted and internal QC samples. However, these correction factors shall not depend on the required method blank result in the associated analytical batch.

4) The method blank sample shall be prepared with similar aliquot size to that of the routine samples for analysis and the method blank result and acceptance criteria (see Section 5.4.1.2.b.18) shall be calculated in a manner that compensates for sample results based upon differing aliquot size.

D.4 DOE-6

All samples and QC samples in each preparation (or analytical) batch shall be prepared concurrently and in exactly the same manner.

b) Positive Controls

1) LCSs - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the QC measures to be used to assess the batch. The LCS result shall be assessed against the specific acceptance criteria (see Section 5.4.1.2.b.18) specified in the laboratory method manual (see Section 5.4.1.2). When the specified LCS acceptance criteria is not met, the specified corrective action and contingencies (see Sections 5.4.1.2.b.19 and 20) shall be followed. The occurrence of a failed LCS acceptance criteria and the actions taken shall be noted in the laboratory report (see Section 5.10.3.1.a).

D.4 DOE-7

Counting: The LCS shall be counted for a sufficient time to meet the required detection limit.

Matrix: The matrix shall be the same as the samples, as close as can be reasonably achieved, and the matrix shall be documented in the Case Narrative.

Acceptance Criteria: The relative bias as calculated from the formula:

$$\text{Relative bias} = \frac{observed - known}{known}$$

shall be in the range -0.25 to +0.25 unless otherwise identified in client specifications.

2) Matrix Spike - Shall be performed at a frequency of one per preparation batch for those methods that include a chemical separation process without the use of an internal standard or carrier, and where there is sufficient sample to do so. Although gross alpha, gross beta and tritium measurements do not involve a chemical separation process, matrix spikes shall be performed for these analyses on aqueous samples. The results of this analysis shall be

one of the QC measures to be used to assess the batch. The matrix spike result shall be assessed against the specific acceptance criteria (see Section 5.4.1.2.b.18) specified in the laboratory method manual (see Section 5.4.1.2). When the specified matrix spike acceptance criteria is not met, the specified corrective action and contingencies (see Sections 5.4.1.2.b.19 and 20) shall be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report (see Section 5.10.3.1.a). The lack of sufficient sample aliquot size to perform a matrix spike shall be noted in the laboratory report.

D.4 DOE-8

Matrix Spikes: Matrix spikes shall be added as early in the sample preparation steps as practicable.

Matrix spikes are not required for radiochemical analyses if an isotopic tracer or chemical carrier is used in the analysis to determine chemical recovery (yield) for the chemical separation and sample mounting procedures. Matrix spikes are not required for gross alpha, gross beta, gamma, or non-aqueous tritium analysis.

Matrix spikes shall be run on a separate sample aliquot using the same analyte as that being analyzed whenever possible.

Acceptance Criteria: Matrix spike recoveries shall be within the control limits of 60 – 140%, or as specified by client. Matrix spike samples for which the sample activity is greater than five times the spiking level are not required to meet this criteria.

3) The activity of the LCS shall:

i. be at least 5 times the LOD, and
ii. at a level comparable to that of routine samples when such information is available if the sample activities are expected to exceed 5 times the LOD.

D.4 DOE-9

LCS Selection and Level: The LCS shall be of the same element as the sample analyte and shall be at least 5 times, but not greater than 20 times the RDL with the following exceptions:

- For RDLs of low activity the analyte shall be at a level where the random counting error does not exceed 10% in the counting time required to attain the RDL.

- Analytes for gamma spectroscopy need not be the same as the sample analyte but should fall in the approximate energy region of the spectrum (low, mid-range, and high energy).
- For gross alpha and/or gross beta analysis, the analytes in the LCS shall be the same analytes used for the calibration curve.

4) The activity of the matrix spike analytes(s) shall be greater than five times the LOD.

D.4 DOE-10

Matrix Spike Selection and Level: The matrix spike shall be added at a concentration of at least five, but not greater than 20 times the RDL. In samples having known significant activity of the radionuclides to be analyzed, more than 20 times the RDL may be added to minimize the effect of the sample activity on determination of spike recoveries.

5) The laboratory standards used to prepare the LCS and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration and must meet the requirements for reference standards provided in Section 4.7 a of this Appendix.

6) The matrix spike shall be prepared by adding a known activity of target analyte after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.). Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g., plutonium, Pu-238 and Pu-239, using alpha spectrometry), only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified LOD, each shall be assessed against the specified acceptance criteria.

D.4 DOE-11

Counting: The matrix spike shall be counted for a sufficient time to meet the required detection limit.

Where the original (unspiked) sample contains significantly elevated activity, the matrix spike shall be counted for a duration equal to that of the associated original sample.

7) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope, the LCS shall contain isotopes that represent the low (e.g., americium-241), medium (e.g., cesium-137) and high (e.g., cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples, the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

D.4 DOE-12

The LCS shall be traceable to the National Institute of Standards and Technology (NIST) or accepted international standard, or shall be a working reference material as described in *ASTM C 1128 (current version),* and may be used repeatedly for different analytical batches as long as it is appropriate for the matrix and geometry of the batch.

The analyte need not be the same as the sample analyte, but shall fall in the approximate energy region of the spectrum as the analyte(s) i.e., low, mid-range, or high energy.

Matrix Spike sample is not required for gamma spectrometry analysis.

8) The LCS shall be prepared with similar aliquot size to that of the routine samples for analyses.

c) Other Controls

D.4 DOE-13

Tracers and carriers chemically mimic and do not interfere with the target analyte through radiochemical separations. Isotopic tracers are typically radioactive materials (e.g., Pu-242, Sr-85), while carriers are typically nonradioactive (e.g., natural strontium). They are added to samples to determine the overall chemical yield for the analytical preparation steps. When tracers or carriers are used, each sample (including any batch associated QC samples) shall be spiked separately with the same materials and individual sample yields will be determined. The tracer shall be added to the sample at the very beginning of the sample preparation. For solid samples, the tracer shall be added after grinding, sieving, etc., but prior to any muffling or dissolution of the sample.

1) Tracer - For those methods that utilize a tracer (i.e., internal standard) each sample result shall have an associated tracer recovery calculated and reported. The tracer shall be added to the sample after subsampling if required, but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The tracer recovery for each sample result shall be one of the QC measures to be used to assess the associated sample result acceptance. The tracer recovery shall be assessed against the specific acceptance criteria (see Section 5.4.1.2.b.18) specified in the laboratory method manual (see Section 5.4.1.2). When the specified tracer recovery acceptance criteria is not met, the specified corrective action and contingencies (see Sections 5.4.1.2.b.19 and 20) shall be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report (see Section 5.10.3.1.a).

2) Carrier - For those methods that utilize a carrier for recovery determination, each sample shall have an associated carrier recovery calculated and reported. The carrier shall be added to the sample after subsampling, if required, but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The carrier recovery for each sample shall be one of the QC measures to be used to assess the associated sample result acceptance. The carrier recovery shall be assessed against the specific acceptance criteria (see Section 5.4.1.2.b.18) specified in the laboratory method manual (see Section 5.4.1.2). When the specified carrier recovery acceptance criteria are not met, the specified corrective action and contingencies (see Sections 5.4.1.2.b.19 and 20) shall be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report (see Section 5.10.3.1.a).

D.4 DOE-14

Tracer yield requirements for isotope dilution methods:
(usually alpha spectroscopy)

The chemical yield for isotope dilution methods shall fall within the range 30% - 110% or as specified by the client. Tracer activity and sample count duration shall be adequate to achieve relative uncertainties for the tracer measurement of less than 10% at the 2-sigma level.
Sample results with chemical yields below 30% are quantitative

and considered acceptable if:

- the relative uncertainty associated with the yield correction is less than 10% (2-sigma),
- spectral resolution requirements are met and there are no indications of spectral interferences, and
- detection limit requirements are met.

Requirements for indirect yield measurements: (e.g., radiometric results are corrected for chemical yield using 'indirect' yield measurement techniques such as gravimetric measurement of added carriers or a second radiometric measurement of added tracer)

The chemical yield for each sample determined using an indirect yield measurement method shall fall within the range 40% - 110% or as specified by the client. The technique used for the indirect yield measurement should be sufficient to maintain relative uncertainties associated with the yield correction below 10% at the 2-sigma level.

Reporting yield measurement uncertainties: The uncertainty associated with chemical yield corrections shall be incorporated into the CSU of the associated sample results.

D.4.2 Analytical Variability/Reproducibility

a) Replicate - Shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria (see Section 5.4.1.2.b.18) specified in the laboratory method manual (see Section 5.4.1.2). When the specified replicate acceptance criteria is not met the specified corrective action and contingencies (see Sections 5.4.1.2.b.19 and 20) shall be followed. The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report (see Section 5.10.3.1.a).

D.4 DOE-15

Duplicates: The purpose of the Duplicate sample analysis is to assess laboratory precision by providing information on the laboratory's reproducibility, and the homogeneity of the sample. The Duplicate activity shall not be averaged with the corresponding sample activity when reporting results. Samples identified as Field Blanks shall not be used for Duplicate sample analysis.

Frequency: At least one duplicate sample shall be prepared and analyzed with every Analytical Batch of samples.

Counting: The duplicate shall be counted for a sufficient time to meet the required detection limit. Where the sample contains significantly elevated activity, the matrix spike shall be counted for a duration equal to that of the associated original sample.

Evaluation Criteria: The duplicates are evaluated using two criteria. The duplicate error ratio (normalized absolute difference) (DER) between the sample and laboratory duplicate, given by the following equation shall be used to determine that the results do not differ significantly when compared to their respective one sigma uncertainty.

$$\frac{|\ S - D\ |}{\sqrt{(CSU_S)^2 + (CSU_D)^2}} \leq 3$$

Note: 3 provides a 99+% confidence level.

S = Sample result
D = Duplicate result
CSU_S = Combined Standard Uncertainty of the sample
CSU_D = Combined Standard Uncertainty of the duplicate

Relative Percent Difference (RPD) may also be used to evaluate the duplicates. The RPD shall be less than 25% or other client applied criteria.

When either the DER or the RPD pass, then the duplicate is acceptable.

Duplicates that do not meet the above requirements due to the difficulty of subsampling shall be described in the Case Narrative.

b) For low-level samples (less than approximately three times the LOD), the laboratory may analyze duplicate LCSs or a replicate matrix spike (matrix spike and a matrix spike duplicate) to determine reproducibility within a preparation batch.

D.4.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Initial Demonstration of Capability - (Section 5.4.2.2 and Appendix C) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type (e.g., different detection technique), personnel or method.

b) Performance Evaluation (PE) Samples - The results of such analysis (Sections 4.1.5.k and 5.9.1) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.4.4 Radiation Measurement Instrumentation

Because of the stability and response nature of modern radiation measurement instrumentation, it is not typically necessary to verify calibration of these systems each day of use. However, verification of calibration is required as outlined in Section b below. This section addresses those practices that are necessary for proper calibration and those requirements of Section 5.5.2.2 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

a) Instrument Calibration

1) Given that activity detection efficiency is independent of sample activity at all but extreme activity levels, the requirements of subsections f, h and i of Section 5.5.2.2.1 are not applicable to radiochemical method calibrations except mass attenuation in gas-proportional counting and sample quench in liquid scintillation counting. Radiation measurement instruments are subject to calibration prior to initial use, when the instrument is placed back in service after malfunctioning and the instrument's response has changed as determined by a performance check or when the instrument's response exceeds predetermined acceptance criteria for the instrument QC.

2) Instrument calibration shall be performed with reference standards as defined in Section 4.7.a of this Appendix. The standards shall have the same general characteristics (i.e., geometry, homogeneity, density, etc.) as the associated samples.

D.4 DOE-16

Detection efficiency shall be determined with sources that are NIST- traceable or with sources prepared from NIST-traceable standards.) When sources used for determinations for detection efficiency are prepared from NIST-traceable standards, they shall be "working reference materials" as defined in STD.ASTM C1128 (current version).

For alpha spectrometry, a material balance check shall be done on each source to clearly demonstrate accountability of all activity by mass balance. The material balance check shall be done on the fraction remaining from, the neodymium fluoride precipitation, or the electro-deposition plus all rinses from an adequate cleaning of any vessel used in the process. The estimated error in preparing the source shall be propagated into the error of the efficiency determination.

Check sources shall be used only to verify that efficiencies have not changed. They shall not be used to determine efficiencies.

3) The frequency of calibration shall be addressed in the laboratory method manual (see Section 5.4.1.2.b.13) if not specified in the method. A specific frequency (e.g., monthly) or observations from the associated control or tolerance chart, as the basis for calibration shall be specified.

b) Continuing Instrument Calibration Verification (Performance Checks)

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

1) For gamma spectroscopy systems, the performance checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.

D.4 DOE-17

For systems using sample changers and/or long count times that run more than a day, the energy calibration shall be checked before each analytical batch.

The Full-Width-Half-Maximum (FWHM) resolution of the detector shall be evaluated daily or prior to instrument use. The measured FWHM resolution shall be trended. Corrective actions shall be taken when an intolerable condition becomes evident or when gross changes are identified in the resolution of the detector at the

energies that bound the applicable energy range (ASTM D 3649-98a).

2) For alpha spectroscopy systems, the performance check for energy calibration shall be performed on a weekly basis and the performance check for counting efficiency shall be performed on at least a monthly basis.

D.4 DOE-18

Detector response (counting efficiency) determinations shall be performed when the check source count is outside the acceptable limits of the control chart (reference ANSI N42.23, Annex A5).

It is important to use calibration or QC sources that will not cause detector contamination from recoil atoms from the source.

3) For gas-proportional and liquid scintillation counters, the performance check for counting efficiency shall be performed on a day-of-use basis. For batches of samples that uninterruptedly count for more than a day, a performance check can be performed at the beginning and end of the batch as long as this time interval is no greater than one week. Verification of instrument calibration does not directly verify secondary calibrations, e.g., the mass efficiency curve or the quench curve.

4) For scintillation counters, the calibration verification for counting efficiency shall be performed on a day of use basis.

D.4 DOE-19

For radon scintillation detectors, efficiency shall be verified at least monthly, when the system is in use.

c) Background Measurement

Background measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality objectives. These values may be subtracted from the total measured activity in the determination of the sample activity.

D.4 DOE-20

Except for gamma spectrometry, background check measurements shall be conducted on a day-of-use basis and monitored for trends to ensure that a laboratory maintains its capability to meet required data quality objectives.

Successive long background measurements may be evaluated as background check measurements.

Low levels of contamination not detected in a shorter background counting time may bias the results of sample analyses. The duration of the background check measurement shall be of sufficient duration (i.e., at least as long as the sample count time) to quantify contamination that may impact routine sample measurements.

The background check frequency may be extended to accommodate long sample count times.

If the background check is conducted less frequently than daily, any associated sample results shall not be released for use until a (bracketing) background check is measured and has met all acceptance criteria. A background check for alpha spectroscopy can be a shorter measurement that can be performed on a weekly basis.

A background shall also be collected before and after any counting chamber changes are made, i.e., cleaning, liner replacement, or instrument modification.

1) For gamma spectroscopy systems, background measurements shall be performed on at least a monthly basis.

D.4 DOE-21

For gamma spectroscopy systems, long background measurements (to be used for background corrections) shall be performed on at least a monthly basis. The duration of the background measurement shall be sufficient to quantify contamination that may affect routine sample measurements (the count time for the background shall be at least as long as the sample count time).

2) For alpha spectroscopy systems, background measurements shall be performed on at least a monthly basis.

D.4 DOE-22

The monthly background determinations shall be performed for each Region of Interest (ROI). The duration of the background measurement shall be sufficient to quantify contamination that may affect routine sample measurements.

Backgrounds for alpha spectrometers should be rechecked after being subjected to high-activity samples.

3) For gas-proportional counters, background measurements shall be performed on at least on a weekly basis.

D.4 DOE-23

Long background measurements (to be used for background corrections) shall be performed on a quarterly basis, at minimum,

Backgrounds for gas flow proportional counters should be rechecked after being subjected to high-activity samples.

4) For scintillation counters, background measurements shall be performed each day of use.

D.4 DOE-24

The duration of the background measurement shall be sufficient to quantify contamination that may affect routine sample measurements.

The daily instrument check shall include a check with an unquenched, sealed background vial (which should never be used to correct sample results for background, since it is not in the same configuration as samples).

For background measurements used in quantitation, see Sections 4.10.d or 4.11.d of this Appendix as appropriate.

d) Instrument Contamination Monitoring

The laboratory shall have a written procedure for monitoring radiation measurement instrumentation for radioactive contamination. The procedure

shall indicate the frequency of the monitoring and shall indicate criteria that initiate corrective action.

D.4.5 Minimum Detectable Activity (MDA)/Minimum Detectable Concentration (MDC)/Lower Level of Detection (LLD)

a) Must be determined prior to sample analysis and must be redetermined each time there is a significant change in the test method or instrument type.

b) The procedures employed must be documented and consistent with mandated method or regulation.

D.4 DOE-25

The SOPs that incorporate the equations to calculate the decision level and the minimum detectable concentration (or activity) must be documented and consistent with mandated method or regulation.

MDA Determination

- The MDA is the smallest amount of an analyte in a sample that will be detected with a probability β of non-detection (Type II error), while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). For the purposes of this QSAS and the equations below, the α and β probabilities are assumed to be 0.05. However, other confidence levels may be dictated by the MQOs.

- MDA Factors and Conditions - MDAs are determined based on the normal factors and conditions, which influence the measurement. The MDA is used to evaluate the capability of a method relative to the required RDLs. Sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency shall be optimized to result in sample MDAs less than or equal to the RDLs. If RDLs are not achieved, then the cause shall be addressed comprehensively in the Case Narrative.

- MDA Calculation - The basic MDA calculation shall be based on concepts developed by L. A. Currie, “Limits for Qualitative Detection and Quantitative Determination, *Analytical Chemistry*, March, 1968, Vol. 40, No.3, Pg. 586. The following general equations shall be used to calculate the MDA.

Without Blank Population -

$$MDA = \frac{4.65 * \sqrt{\frac{b}{T}}}{K} + \frac{3}{K * T}$$

where,

b = background count rate in cpm
T = count time in minutes
K = efficiency * e $^{-\lambda t}$ * aliquot fraction * tracer recovery* ABN Efficiency = detector efficiency
T = time from sample collection to mid-point of count time(or nuclide separation time, as applicable) in the same units as half-life
λ = Analyte decay constant = ln2/(half-life)
ABN = abundance

Use of the above equation requires that the background and sample count times are equivalent. When sample and background counts are different, the following adjustment to the equation is required.

$$MDA = \frac{3.29 * \sqrt{\frac{b}{T_S} + \frac{b}{T_B}}}{K} + \frac{3}{K * T_S}$$

where,
T_S = count time of the sample in minutes
T_B = count time of the background in minutes

The above equations for MDA have the units of dpm/sample. Any other units will require appropriate conversion.

Site specific requirements may be provided for other MDA formulations.

With Blank Population -

$$MDA = \frac{3.29 * s_b}{KT_S} + \frac{3}{KT_S}$$

s_b = standard deviation of the blank population where the blank population is in net blank counts in count time T_S

Use of blank populations for calculation of MDAs requires the selection of an implementation method, which includes but is not limited to:

Identification of blanks to be used in the population:

- The number of blanks to use in the population
- How the blank population changes
- Limitations on the deletion of blanks

The method of implementation shall not introduce any statistical bias.

The appropriate blank subtraction shall be the mean blank value of the blank population.

The implementation of blank populations for calculation of MDAs shall be described in detail in an SOP.

MDA Optimization - The laboratory shall optimize analysis parameters in order to achieve analyte MDAs less than or equal to the RDLs, except when sample activities are significantly greater than the RDL. Samples with elevated activities shall be handled according to the following requirements:

- The appropriate aliquot size shall be determined based on the activity level in the sample. The aliquot shall be large enough to generate data, which meet the following criteria:

- The measurement uncertainty shall not be greater than 10% (1 sigma) of the sample activity.

- The MDA for the analysis shall be a maximum of 10% of the sample activity.

If sample-specific MDAs are calculated and reported, that shall be clearly stated in the data package.

The definition of the MDA presupposes that an appropriate detection threshold (i.e., the decision level) has already been defined. The α probabilities assumed for the decision level shall also be used for the calculation of the MDA.

Decision Level Calculation

- **Decision Level (DL)**: In the context of analyte detection, the minimum measured value (e.g., of the instrument signal or the analyte concentration) required to give confidence that a positive (nonzero) amount of analyte is present in the material analyzed. The DL is sometimes called the critical level (Lc) or critical value (MARLAP). It is

the quantity of analyte at or above which an *a posteriori* decision is made that a positive quantity of the analyte is present. For this document, the probability of a Type I error (probability of erroneously reporting a detectable nuclide in an appropriate blank or sample) is assumed to be set at 0.05; however, other confidence levels may be dictated by the MQOs.

- DL Factors and Conditions – DLs are determined *a posteriori* based on sample-specific sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency.

- DL Calculation - The basic DL calculation shall be based on concepts developed by L. A. Currie, "Limits for Qualitative Detection and Quantitative Determination, *Analytical Chemistry*, March, 1968, Vol. 40, No.3, Pg. 586.The following general equations shall be used to calculate the decision level.

The DL can either be based on the CSU of the sample, or the standard deviation determined from a set of appropriate blanks.

Without Blank Population -

When determined from the CSU of the sample, the DL evaluates the level where there is 95% confidence (or other specified level of confidence) that the true result is greater than zero and can be estimated by the following equation:

$$DL = 1.65 \times CSU_R$$

where:

- Lc is the DL (dpm/unit);

- CSUR is the combined standard uncertainty (random component only) of the result, R (dpm/unit); and

- 1.65 is the abscissa of the standardized normal distribution for 95% confidence.

With Blank Population -

When determined from the standard deviation of a set of appropriate blanks, the DL evaluates the level where the blank results will not exceed more than 5% of the time (or other specified level of confidence) and may be estimated by the following equation:

$$DL = \frac{(t \times S_B) + \overline{R_B}}{E \times R \times IDF \times W}$$

where:

- DL is the decision level in disintegrations per minute per unit volume or weight (dpm/unit);
- S_B is the standard deviation of a set of appropriate blank net count rate after background subtraction for blanks counted for the same length of time as the sample;
- R_B is the average blank count rate in counts per minute (cpm);
- t is the student t factor for appropriate degrees of freedom and confidence level;
- E is the fractional detector efficiency (c/d) for the sample;
- R is the fractional chemical yield for the sample;
- IDF is the ingrowth or decay factor for the sample; and
- W is the weight or volume of sample.

DLs are used as the default detection threshold. Alternatively, the client may use/specify detection thresholds that meet project/site-specific MQOs.
The client may request or use detection project thresholds or MQOs

D.4.6 Data Reduction

a) Refer to Section 5.4.7.2, "Computers and Electronic Data Related Requirements."

D.4 DOE-26

Negative Numbers: All negative activities shall be reported as such. If the sum of the activity and the measurement activity at +3-sigma is a negative number, the cause shall be investigated and evaluated to determine if it is systematic or random. If the cause is systematic, it shall be corrected. If the cause is random, it shall be documented in the Case Narrative. Recurrent problems with significant negative results suggest that the background subtraction and/or blank subtraction, if applicable,

are in error or that the estimate or error is low. Investigation of such problems and documentation of the resolution is required and shall be discussed in the Case Narrative.

References:

DOE / EH - 0173T "Environmental Regulatory Guide for Radiological Effluent Monitoring and Environmental Surveillance, January 1991

Multi-Agency Radiological Laboratory Analytical Protocols Manual NRC NUREG-1576, EPA 402-B-04-001C, NTIS PB2004-105421 July 2004 Section 18.6.5

b) Measurement Uncertainties - each result shall be reported with the associated measurement uncertainty. The procedures for determining the measurement uncertainty must be documented and be consistent with mandated method and regulation.

D.4 DOE-27

Measurement Uncertainties: Each result shall be reported with the associated measurement uncertainty as a combined standard uncertainty. The SOP for determining the measurement uncertainty must be consistent with mandated method and regulation.

Combined Standard Uncertainty: All measurement uncertainties shall be propagated and reported with each result. The formula for calculating the combined standard uncertainty of a result shall be documented in the appropriate SOP. The Combined Standard Uncertainty shall include both systematic and random error. **Combined Standard Uncertainty is always 1 sigma. Results should be reported at the 95% confidence level, which is 2 sigma.**

The uncertainty of a count may be estimated as the square root of counts except when there are zero (0) counts. In the case of zero (0) counts, the uncertainty of the count is assumed to be the square root of one count.

Systematic Error shall include, but is not necessarily limited to:

(1) The errors from all measurement devices, such as, but not limited to pipettes and balances.

(2) The uncertainty of know values of tracer solutions, calibration uncertainties, etc.

Random Error shall include, but is not necessarily limited to, the total

random counting error associated with each sample and appropriately propagated when more than one variable is used to determine the result.

D.4.7 Quality of Standards and Reagents

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

1) Reference standards that are used in a radiochemical laboratory shall be obtained from the NIST, or suppliers who participate in supplying NIST standards or NIST-traceable radionuclides. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to ensure the quality of their products.

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

D.4 DOE-28

Standards shall be verified prior to initial use.

Preparations of standards solutions used for a period of time exceeding one year shall be verified annually, at a minimum, and documented in a logbook.

At least three verification measurements of a standard shall be used to determine the mean value and standard deviation of the verification results.

The mean value shall be within 5% of the decay corrected certified value.

The two sigma value used for the 95% confidence interval of the mean shall not exceed 10% of the mean value of the three verification measurements.

If all criteria are met, the certified value shall be used.

3) Laboratories should consult with the supplier if the laboratory's verification of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory shall not use a value other than the decay corrected certified value. The laboratory shall have a written procedure for handling, storing and establishment of expiration dates for reference standards.

D.4 DOE-29

Corrections for radioactive decay and/or ingrowth of progeny shall be performed for radionuclide standards.

b) All reagents used shall be analytical reagent grade or better.

D.4 DOE-30

Water purity shall be at least distilled or deionized water.

D.4.8 Constant and Consistent Test Conditions

The laboratory shall maintain a radiological control program that addresses analytical radiological control. The program shall address the procedures for segregating samples with potentially widely varying levels of radioactivity. The radiological control program shall explicitly define how low level and high level samples will be identified, segregated and processed in order to prevent sample cross-contamination. The radiological control program shall include the measures taken to monitor and evaluate background activity or contamination on an ongoing basis.

D.4 DOE-31

The detection/quantification requirements for contamination control sampling should be consistent with the lowest level of sample analyte or MDA equivalent. Samples shall be segregated by activity levels in sample receipt, processing areas, and storage areas.

D.4 DOE-32

D.4.9 Isotopic Determinations by Alpha Spectrometry

a) Tracer - Shall be used for isotope specific analysis by alpha spectrometry. Initial sample preparation shall include treatment to ensure that tracer and analyte will undergo similar reactions during processing. All tracers used for alpha spectrometry shall be tested by the laboratory for contribution in the ROIs of the analytes of interest. All tracers shall be of the same element or of an element with the same chemistry for the separations. If a significant contribution is found, the method for correction shall be Site accepted prior to use.

b) Background Correction - The gross counts in each target analyte and tracer ROI shall be corrected for the particular detector's background contribution in those same ROIs.

c) Blank Correction - shall not be routinely performed.

d) Conditions Requiring Reanalysis

1) Sample- and Analyte-Specific Conditions: Any one of the following are additional conditions that require reanalysis for a particular sample and analyte.

i. If the tracer recovery for the sample does not fall within 30% - 110%, reanalysis is required, beginning with preparation.

iii. If the FWHM for the tracer peak exceeds 100 keV and/or the peak energy does not fall within ± 50 keV of the known peak energy, reanalysis is required.

iv. If the target analyte and tracer peaks are not resolved because the target analyte activity is significantly larger than the tracer activity, the sample shall be reanalyzed with a smaller aliquot such that resolution of tracer and analyte peaks is accomplished.

v. If the sample analyte spectrum contains significant interferences with the analyte and/or tracer ROIs, reanalysis is required.

2) Analytical Batch Conditions: If the tracer chemical recovery for the Batch Blank does not fall with 30% - 110%, reanalysis of the entire Analytical Batch, beginning with the preparation, is required if sufficient sample is available.

e) Instrument Calibration - Calibration of each alpha spectrometry detector used to produce data shall include channel vs. energy calibration, detector response

f) (Efficiency) determination and background determination for each ROI. Alpha spectrum regions of interest shall be selected with consistency from analyte to analyte.

g) Energy Calibration

1) The energy calibration for each detector shall be performed. A curve shall be fit for Energy (Y-axis) versus Channel (X-axis) and the equation with the slope and Y-intercept for the fit shall be documented.

2) The slope of the equation shall be <15 keV/channel.

3) The energy calibration shall be performed using at least three isotopes within the energy range of 3 to 6 MeV.

4) The final peak energy positions of all observed isotopes shall be within ±40 keV of the expected peak energy.

h) Background Requirements

1) The background total counts (or counts per unit time) for each target analyte and tracer isotope ROI shall be analyzed on each detector and documented.

2) The background for each ROI shall be sufficiently low to ensure that required detection limits are met

3) The limits of acceptability for each background ROI shall be documented. These shall be set such that RDLs can be obtained for backgrounds at the limit of acceptability.

4) Background count times shall be equal to or longer than sample count times.

i) Detector Response Determination Requirements

Detector response (efficiency) is not used in the calculation of results when tracers are used in the analysis, but only used to calculate the estimated yield, which is also not used, except as a general method performance indicator.

1) The response (efficiency) counts for the ROI shall be background corrected using the same ROI for the background unless the background is less than 0.5% of the total counts in the ROI.

2) The response (efficiency) shall be determined on at least 3,000 net counts in the ROI (after background correction).

3) Check source counts to verify detector response (efficiency) shall be determined on at least 2,000 counts.

4) The detector response and detector response error shall be documented.

5) The detector response check as determined by the check source and/or pulser count and the associated error and limits of acceptability for the check source result shall be

documented.

j) Spectrum Assessment

1) ROIs shall be clearly indicated either graphically or in tabular form on alpha printouts. **Spectra with ROIs shall be saved and made available for review upon request.**

2) The FWHM resolution for each sample and QC sample tracer peak shall be ≤100 keV.

3) The tracer peak energy for each sample and QC sample shall be within ±50 keV of the expected energy.

4) Each sample and QC sample spectrum shall be assessed for correctly chosen ROIs, acceptable spectral resolution, acceptable energy calibration and interferences with the analyte and tracer ROIs.

D.4.10 Radon Scintillation (Lucas Cell)

a) Procedures for sample analyses by Lucas Cell shall incorporate and adhere to ASTM D3454-05, Stand Test Method for Radium-226 in Water. Where the word "should" is used in ASTM D3454-05, performance shall be in accordance with the statement unless otherwise provided in this document. Reference is to the current version of the method. When references change, an implementation schedule should be determined.

b) The operating voltage plateau for the detector shall not exceed a slope of 2%/100V.

c) A new Lucas Cell shall be calibrated every month for the first six months of use and then annually after the initial six months of use.

d) Background measurements for quantitation in each cell shall be carried out prior to each sample measurements.

e) When consistent with MQO, Rn-222 ingrowth times may be shortened to the degree permitted by EPA Method 903.1

D.4.11 Liquid Scintillation Counting

a) Tritium in Water - Water samples for tritium analysis and all associated QC samples shall be distilled prior to analysis unless specified otherwise by the client. The applicable preparation SOP shall specify

the fraction to be collected. The same fraction shall be collected for samples and all associated QC samples.

b) Counting Vial Preparation - Samples shall be counted in vials equivalent to or superior to low potassium glass vials or high density polyethylene vials. Samples in polyethylene vials shall be counted within a time period not to exceed the manufacturer's specification for the cocktail used in the analysis. Analysis documentation shall contain sufficient information for this to be verified. Vials shall be prepared according to manufacturer's specification for the cocktail. The vials shall be "dark adapted" for a minimum of 30 minutes or according to the cocktail manufacturer's specifications before counting. The prepared vials shall be inspected to verify that the sample loaded properly in the cocktail.

c) Laboratory SOPs for methods using liquid scintillation counting shall incorporate and adhere to ANSI N42.15-1997, American National Standard Check Sources for and Verification of Liquid Scintillation Systems. References are for the current version. When references change, an implementation schedule will be determined.

d) Instrument Background - The instrument background vial for all tritium matrices shall be prepared with low-tritium or "dead" water. The instrument background vial shall be prepared with the same water to cocktail ratio as the samples are prepared. The type of water used to prepare the instrument background vial shall be explicitly noted on the preparation and counting documentation. The instrument background shall be run with each sample batch. Unless calculated from a running average of background counts or a background quench curve, the most recent background count shall be used to calculate sample activities and MDAs. This is not a performance check rather a background subtraction sample in a configuration equivalent to that of associated samples in the batch. It is used to generate the background subtraction data for the batch (using the results associated directly with that batch, results of a rolling mean, or background quench curve). The effect of quench on background shall be evaluated and corrected using a background quench curve if it is significant.

e) For analysis methods using quench curves to determine individual sample detection efficiency or background, the quench curves shall be generated at least yearly and verified after any instrument maintenance.

f) If the calibration method is constant quench, the detection efficiency shall be checked at least weekly when in use or with each counting batch.

g) Sample-Specific Conditions - The following are conditions that require reanalysis for a particular sample and analyte, beginning with the preparation or recounting, as appropriate:

1) If the constant quench method of calibration is used, the quench of each sample analyzed shall fall within +/-5% relative to the average efficiency at that quench level. If this condition is not met, the sample must be reanalyzed beginning with vial preparation.

2) If the sample quench does not fall within the range of the quench curve, the samples shall be reanalyzed such that the sample quench is in the range of a quench curve.

h) Spectrum Assessment - For analytes requiring separations other than distillation:

1) Sample spectra shall be retained for each sample and QC sample including identification of ROIs.

2) Each sample and QC sample spectrum shall be assessed for correctly chosen ROIs, acceptability of peak shape, and interferences due to non-target analytes or luminescence.

D.4.12 Gas Flow Proportional Counting

a) Planchets - shall be thoroughly cleaned before use to ensure that there are no interfering residues or contamination. All planchets shall be prepared not to exceed sample weights in excess of the calibrated ranges of established self-absorption curves. Sample weights shall be documented and stable prior to counting. Planchets exhibiting physical characteristics notably different from the self-absorption standards (e.g., evidence of corrosion) shall not be counted unless remediation efforts such as additional sample preparation and remounting, flaming prove unsuccessful. Any non-routine counting situations shall be documented in the Case Narrative.

b) Instrument Calibration - shall be performed in accordance with the requirements in ANSI N42.25, Calibration and Usage of Alpha/Beta Proportional Counters. Where the word “should” is used in ANSI N42.25, calibration shall be performed in accordance with the statement. References are for the current version. When references change, an implementation schedule will be determined.

c) Calibration Sources and Standards –

1) The standard reference material used to prepare sources for

determining detector efficiencies and self-absorption curves shall be NIST-traceable. The calibration sources shall provide adequate counting statistics over the period for which the source is to be counted.

2) However, the source shall not be so radioactive as to cause pulse pileups or dead time that is significantly different from that to be expected from routine analyses.

3) The geometry of the calibration sources used for efficiency and self-absorption/crosstalk curves shall be the same as that of the prepared sample and QC sample planchets. The depth and shape (flat, flanged, ringed, etc.), in addition to the diameter, are factors that shall be the same for calibration sources as for samples.

4) The sources used for the determination of self-absorption and cross talk should be of similar isotope content to that of the analytical samples. Am-241; Po-210; or Th-230 shall be used for alpha and Cs-137 or Sr-90/Y-90 for beta. The crosstalk isotopes shall be the same as the efficiency/self-absorption curve isotopes.

d) Self-Absorption and Crosstalk Curves

1) Self-absorption curves are required for both alpha and beta counting.

2) A crosstalk curve shall be established for alpha to beta crosstalk versus residue weight.

3) Beta to alpha crosstalk is not significantly affected by planchet residue weight, and is generally constant over the applicable weight range. Therefore this crosstalk correction does not require residue weight consideration.

4) The data used to generate self-absorption and crosstalk curves shall consist of at least seven points, well distributed throughout the mass range.

5) Each alpha and beta calibration standard shall be counted to an accumulation of at least 10,000 counts minimum for the initial calibration and 5,000 counts minimum for the calibration verification.

6) Whenever a new detector voltage plateau or efficiency is determined, a new cross-talk curve must be measured.

e) Check Source Requirements

1) The alpha and beta response and corresponding crosstalk of each detector used to count analytical samples or QC samples shall be checked daily with separate alpha and beta emitting sources. The only exception to this requirement is when performing analyses with extended count times. In this case, check source measurements may be performed between sample sets.

2) Following gas bottle changes, check sources and backgrounds shall be analyzed before samples are counted.

3) Check source data shall be documented and retained.

D.4.13 Gamma Spectrometry

a) Sample Counting Requirements

1) SOPs for sample analysis by gamma spectrometry shall incorporate and adhere to ANSI N42.14-1991, Calibration and Use of Germanium Spectrometers for the Measurement of Gamma Ray Emission Rate of Radionuclides, and/or ANSI N42.12-1994, Calibration and Usage of Thallium-Activated Sodium Iodide Detector Systems for Assay of Radionuclides. References are for the current version. When references change, an implementation schedule will be determined.

2) The gamma detector system shall consist of any detector suitable for measuring the gamma isotopes of interest in the typical energy range of approximately 0.059 to 1.836 MeV with regard to attaining RDLs, bias and precision requirements. Ge detectors of either intrinsic (pure) germanium or lithium drifted germanium are preferred; however for some specific requirements, another detector type, such as sodium iodide, may be more appropriate.

3) Detectors shall be calibrated for the specific geometry and matrix considerations used in the sample analysis. The Laboratory should have the capability to seal soil samples in airtight cans or equivalent in order to allow ingrowth of radon for accurate analysis of Ra-226 or its progeny by gamma spectroscopy when requested.

4) Spectral Data Reference - Identification of the reference used for the half-life, abundance and peak energy of all nuclides shall be documented. The laboratory shall document, review and provide configuration control for gamma spectrometry libraries. Assumptions made for libraries (i.e., half-lives based on supported/unsupported assumptions, inferential determinations

(e.g., Th-234 = U-238 because supported)) shall be documented and narrated.

b) Efficiency Calibration Requirements

1) Each gamma spectrometry system shall be efficiency calibrated for the sample geometry and matrix with NIST-traceable standards or prepared from NIST-traceable sources.

i) Germanium Detectors: Refer to ANSI N42.14 for guidance on isotope specific efficiency and efficiency as a function of energy calibrations. The efficiency calibration measurements shall be at least seven peaks which cover the typical energy range of approximately 0.059 to 2 MeV. At least 10,000 net counts (total counts minus the Compton continuum and ambient background) shall be accumulated in each full-energy gamma-ray peak of interest used for the efficiency equation (ASTM D 3649-98a).
Sodium Iodide Detectors: Refer to ANSI N42.12.

ii) Efficiencies shall be determined when there is a change in resolution, geometry, or system configuration (ASTM D 3649-98a).

2) Current software that does not require a physical calibration standard to obtain efficiencies for various matrices and geometries may be used to count samples where a standard calibration source of known matrix and geometry cannot be specified. This type of calibration technique is preferred for matrices such as waste or debris. When such software is used, the laboratory shall supply detailed information and documentation regarding the selection of parameters used to specify the efficiency calibration and sample models. Each sample selected for analysis using this type of calibration shall have a unique set of model parameters associated with it. When such models are used, the closest model to the actual sample shall be selected. The model selected for each sample shall be presented in the Case Narrative and shall include a discussion of actual and predicted peak ratios for isotopes with multiple gamma energies present in the sample.

c) Energy Calibration Requirements

Each gamma spectrometry system shall be energy

calibrated with NIST-traceable standards or prepared from NIST-traceable sources.

i) Germanium Detectors: Refer to ANSI N42.14, Section 5.1 for guidance on calibrating gamma-ray energy as a function of channel number at a fixed gain. The energy calibration measurements shall be made using at least three peaks which cover the energy range from 0.059 to approximately 2 MeV. Additional peaks shall be used as deemed appropriate by the laboratory.

ii) At least 10,000 net counts (total counts minus the Compton continuum and ambient background) shall be accumulated in each full-energy gamma-ray peak of interest (ASTM D 3649-98a).

iii) Energy calibration shall be linear and accurate to 0.5 keV.

iv) Sodium Iodide Detectors: Refer to ANSI N42.12, Section 4.3.2."

d) Performance Evaluation

Germanium Detectors: Refer to ANSI N42.14, Section 7.
Sodium Iodide Detectors: Refer to ANSI N42.12, Section 4.3.5.

e) Spectrum Assessment: Each sample and QC sample spectrum shall be assessed for acceptability of key peak width and shape, and interference due to superimposed peaks or other sources. Any major contributor to the spectrum that is an unidentified peak shall be discussed in the Case Narrative.

D.4.14 Conditions Requiring Reanalysis or Recount

If reanalysis is not possible, the client shall be contacted for specific guidance or requirements.

a) General Conditions

1) If the RDLs could not be achieved because of laboratory errors or oversights such as inadequate count times, inadequate aliquot size, inappropriate dilution, low detector efficiencies, high detector backgrounds, etc., then the sample shall be reanalyzed under more optimal conditions.

2) If the RDLs could not be achieved because of problems associated

with the sample such as inadequate sample provided, elevated radioactivity levels, sample matrix interferences such as high amounts of suspended solids, multiphase liquids, etc., then such problems shall be explained in the Case Narrative.

b. Sample and Analyte-Specific Conditions: Any one of the following are additional conditions that require reanalysis for a particular sample and analyte:

1) If, for any reason, sample or batch QC integrity becomes suspect (e.g., spillage, mis-identification, cross-contamination), all potentially affected samples shall be reanalyzed from a point before that at which the integrity came into question. If new batch QC must be prepared for reanalysis, samples for reanalysis shall be restarted at the normal point of initiation for the batch QC.

2) All samples failing the criteria for tracers or carriers as defined in Section 4.1.c of this Appendix.

3) All samples associated with expired standards.

c. Analytical Batch Conditions: Except where noted otherwise, any one of the following conditions requires reanalysis of the entire Analytical Batch, beginning with the preparation:

1) Batches that failed the Batch Blank criteria, as defined in Section 4.1.a of this Appendix.

2) Batches that failed the LCS criteria, as defined in Section 4.1.b of this Appendix.

3) Batches that failed the Matrix Spike criteria, as defined in Section 4.1.b of this Appendix.

d) Conditions requiring a re-count – If the RDL was not achieved due to inadequate count duration, low detector efficiencies, or high detector backgrounds, the sample shall be re-counted under more optimal conditions, and the reasons for the re-count shall be documented in the Case Narrative of the laboratory report.

D.5 AIR TESTING

These standards shall apply to samples that are submitted to a laboratory for the purpose of analysis. They do not apply to field activities such as source air emission measurements or the use of continuous analysis devices.

D.5.1 Negative and Positive Controls

a) Negative Controls

1) Method Blanks – Shall be performed at a frequency of at least one per batch of 20 environmental samples or less per sample preparation method. The results of the method blank analysis shall be used to evaluate the contribution of the laboratory provided sampling media and analytical sample preparation procedures to the amount of analyte found in each sample. If the method blank result is greater than the limit of quantitation and contributes greater than 10% of the total amount of analyte found in the sample, the source of the contamination must be investigated and measures taken to eliminate the source of contamination. If contamination is found, the data shall be qualified in the report.

2) Collection Efficiency - Sampling trains consisting of multiple sections (e.g., filters, sorbent tubes, impingers) that are received intact by the laboratory, shall be separated into “front” and “back” sections if required by the client. Each section shall be processed and analyzed separately and the analytical results reported separately.

b) Positive Controls

1) LCS – Shall be analyzed at a rate of at least one per batch of 20 or fewer samples per sample preparation method for each analyte. If a spiking solution is not available, a calibration solution, whose concentration approximates that of the samples, shall be included in each batch and with each lot of media. If a calibration solution must be used for the LCS, the client will be notified prior to the start of analysis. The concentration of the LCS shall be relevant to the intended use of the data and either at a regulatory limit or below it.

c) Surrogates - Shall be used as required by the test method or if requested by the client.

d) Matrix spike – Shall be used as required by the test method, or if requested by the client.

D.5.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates – Shall be analyzed at a minimum of one in 20 samples per sample batch. The laboratory shall document their procedure to select the use of appropriate types of spikes and duplicates. The selected samples(s) shall be rotated among client samples so that various sample matrix problems may be noted and/or addressed. Poor performance in the spikes and duplicates may indicate a problem with the sample composition and shall be reported to the client.

D.5.3 Method Evaluation

In order to ensure the accuracy of the reported result(s), the following procedures shall be in place:

a) Demonstration of Capability – (Sections 5.2.6 and 5.4.2.2) shall be performed prior to the analysis of any samples and with a significant change in instrument type, personnel, quality system matrix, or test method.

b) Calibration – Calibration protocols specified in Section 5.5.2 shall be followed.

c) Proficiency Test Samples – The results of such analyses (Sections 4.1.5.k or 5.9.1) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.5.4 Limit of Detection

The requirements of Section 1.2.1 of this Appendix shall apply.

D.5.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.5.6 Quality of Standards and Reagents

a) The source of standards shall comply with Section 5.6.2.2.

b) The purity of each analyte standard and each reagent shall be documented by the laboratory through certificates of analyses from the manufacturer/vendor, manufacturer/vendor specifications, and/or independent analysis.

c) In methods where the purity of reagents is not specified, analytical reagent grade or higher quality, if available, shall be used.

D.5.7 Selectivity

The laboratory shall develop and document acceptance criteria for test method selectivity, such as absolute and relative retention times, wavelength assignments, mass spectral library quality of match, and mass spectral tuning.

D.5.8 Constant and Consistent Test Conditions

a) The laboratory shall ensure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) The laboratory shall document that all sampling equipment, containers and media used or supplied by the laboratory meet required test method criteria.

c) If supplied or used by the laboratory, procedures for field equipment decontamination shall be developed and their use documented.

d) The laboratory shall have a documented program for the calibration and verification of sampling equipment (such as pumps, meter boxes, critical orifices, flow measurement devices and continuous analyzers), if this equipment is used or supplied by the laboratory.

D.6 ASBESTOS TESTING

D.6 DOE-1

The laboratory shall be American Industrial Hygiene Association (AIHA) accredited for asbestos analysis.

The following requirements apply to laboratories undertaking the examination of asbestos samples. These requirements are 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. If NIST SRMs specified below are unavailable, the laboratory may substitute an equivalent reference material with a certificate of analysis.

D.6.1 Negative Controls

D.6.1.1 Transmission Electron Microscopy

D.6.1.1.1 Water and Wastewater

a) Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each 24 shall be tested for background level. When using glass bottles, four bottles from each 24 shall be tested. An acceptable bottle blank level is defined as ≤ 0.01 MFL > 10 μm. (EPA /600/R-94/134, Method 100.2, Section 8.2)

b) 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. (EPA /600/R-94/134, Method 100.2, Section 11.8)

D.6.1.1.2 Air

a) 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 20 samples analyzed. [40 CFR Part 763, Appendix A to Subpart E (AHERA), Table 1]

b) Maximum contamination on a single blank filter shall be no more than 53 structures/mm^2. Maximum average contamination for all blank filters shall be no more than 18 structures/mm^2. (AHERA, III.F.2)

D.6.1.1.3 Bulk Samples

a) Contamination checks using asbestos-free material, such as the glass fiber blank in SRM 1866 (Page C-3, NIST Handbook 150-3, August 1994) shall be performed at a frequency of one for every 20 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.

b) The laboratory must maintain a list of non-asbestos fibers that can be confused with asbestos (Section 7.5, Page C-8, NIST Handbook 150-3, August 1994). The list must include crystallographic and/or chemical properties that disqualify each fiber being identified as asbestos (Section 2.5.5.2.1, Identification, Page 54, EPA/600/R-93/116).

c) The laboratory should have a set of reference asbestos materials from which a set of reference diffraction and X-ray spectra have been developed.

D.6.1.2 Phase Contrast Microscopy

At least two 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 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 should be unknown to the counter until all counts have been completed. If a field blank yields greater than seven fibers per 100 graticule fields, possible contamination of the samples shall be reported.

D.6.1.3 Polarized Light Microscopy

a) Friable Materials - At least one blank slide must be prepared daily or with every 50 samples analyzed, whichever is less. The blank slide is prepared by mounting a subsample of an isotropic verified non-Asbestos Containming Material (ACM) (e.g., fiberglass in SRM 1866) in a drop of immersion oil (n_D should reflect usage of various n_Ds) 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 must be scanned to detect any asbestos contamination. A similar check must be made after every 20 uses of each piece of homogenization equipment. An isotropic verified non-ACM must 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 non-ACM non-friable material must be prepared and analyzed with every 20 samples analyzed. This non-ACM must go through the full preparation and analysis regimen for the type of analysis being performed.

D.6.2 Test Variability/Reproducibility

D.6.2.1 Transmission Electron Microscopy

Quality assurance 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 quality assurance 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 reanalyzed). The proportion of QC samples can later be lowered gradually, as control indicates, to a minimum of 10%.

D.6.2.1.1 Water and Wastewater

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings. QA analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks must be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from interlaboratory proficiency testing. Relative Standard Deviations (RSD) for amphibole asbestos decreased from 50% at 0.8 MFL to 25% at seven million fibers per liter (MFL) in interlaboratory PE, while RSD for chrysotile was higher, 50% at six 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 1.5 times of Poisson standard deviation. This shall be performed at a frequency of one per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)

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 2.0 times of Poisson standard deviation. This shall be performed at a frequency of one per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)

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 per 20 samples. Qualified analysts must maintain an average of $\geq$ 80% true positives, $\leq$ 20% false negatives, and $\leq$ 10% false positives.

D.6.2.1.2 Air

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

The laboratory and TEM analysts must 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 NIST certificate. These limits are derived from the allowable false positives and false negatives given in Section 6.2.1.2.c, Verified Analysis, below. SRM 1876b shall be analyzed a minimum of once per year by each TEM analyst.

The laboratory must 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 one μm in length in known standard materials traceable to NIST, such as NIST bulk asbestos SRM 1866.

Interlaboratory analyses shall be performed to detect laboratory bias. The frequency of interlaboratory verified analysis must correspond to a minimum of one per 200 grid square analyses for clients.

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

a) Replicate – A second, independent analysis shall be performed in accordance with Section 6.2.1.1.a of this Appendix. (AHERA, Table III)

b) 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 2.0 times of Poisson standard deviation. This shall be performed at a frequency of one per 100 samples. (AHERA, Table III)

c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section 6.2.1.1.c of this Appendix. (AHERA, Table III)

D.6.2.1.3 Bulk Samples

Determination of precision and accuracy should follow guidelines in NISTIR 5951, Guide for Quality Control on the Qualitative and Quantitative Analysis of Bulk Asbestos Samples: Version 1. Because bulk samples with low (< 10%) asbestos content are the most problematic, a laboratory's QC program should focus on such samples. 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 out of 50 samples must be reanalyzed by the same analyst. For single analyst laboratories, at least one out of every 10 samples must be reanalyzed by the same analyst.

b) Inter-Analyst Precision - At least one out of 15 samples must be reanalyzed by another analyst. Inter-analyst results will require additional reanalysis, 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 must participate in round robin testing with at least one other laboratory. Samples must be sent to this other laboratory at least four times per year. These samples must be samples previously analyzed as QC samples. Results of these analyses

must be assessed in accordance with QC requirements. As a minimum, the QC requirements must address misclassifications (false positives, false negatives) and misidentification of asbestos types.

D.6.2.2 Phase Contrast Microscopy

a) Inter-Laboratory Precision – Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory QA program that, as a minimum, includes participation of at least two other independent laboratories. Each laboratory shall participate in round robin testing at least once every six months with at least all the other laboratories in its inter-laboratory QA 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.

D.6.2.3 Polarized Light Microscopy

Refer to Section 6.2.1.3 of this Appendix.

D.6.3 Other Quality Control Measures

D.6.3.1 Transmission Electron Microscopy

D.6.3.1.1 Water and Wastewater

a) Filter preparations shall be made from all six asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between one and 20 structures (> 10μm) per 0.01 mm^2. One of these preparations shall be analyzed independently at a frequency of one per 100 samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with Turner and Steel (NISTIR 5351).

b) NIST SRM 1876b must be analyzed annually by each analyst. Results shall be evaluated in accordance with limits published for that SRM. Note: This SRM is not strictly appropriate for waterborne asbestos, but analysts can demonstrate general TEM asbestos competence by producing results within the published limits of this (the only recognized TEM counting standard) SRM.

D.6.3.1.2 Air

a) Filter preparations shall be made from all six asbestos types in accordance with Section 6.3.1.1.a of this Appendix.

b) NIST SRM 1876b must be analyzed annually in accordance with Section 6.3.1.1.b of this Appendix.

D.6.3.1.3 Bulk Samples

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite). Standards for the six asbestos types listed are available from NIST (SRMs 1866 and 1867). These materials can also be used as identification standards for AEM (Section 3.2.1, Qualitative Analysis, Page 57, EPA/600/R-93/116).

D.6.3.2 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 should re-label slides before the second count. A test for Type II error (NIOSH 7400, Issue 2, 15 August 1994, Section 13) shall be performed to determine whether a pair of counts by the same analyst on the same slide should 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. It shall not be necessary to use this statistic on blank recounts.

b) All individuals performing airborne fiber analysis must have taken the NIOSH Fiber Counting Course for sampling and evaluating airborne asbestos dust or an equivalent course.

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 AIHA, or equivalent.

D.6.3.3 Polarized Light Microscopy

a) Friable Materials - Because accuracy cannot be determined by reanalysis of routine field samples, at least one out of 100 samples must be a standard or reference sample that has been routinely resubmitted to determine analyst's precision and accuracy. A set of these samples should be accumulated from PE samples with predetermined weight compositions, or from standards generated with weighed quantities of asbestos and other bulk materials (Perkins and Harvey, 1993; Parekh et al., 1992; Webber et al., 1982). At least half of the reference samples submitted for this QC must contain between one and 10% asbestos.

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

D.6.4 Method Evaluation

In order to ensure the accuracy of reported results, the following procedures shall be in place:

a) Demonstration of Capability – (Refer to Section 5.4.2.2) shall be performed initially (prior to the analysis of any samples) and with any significant change in instrument type, personnel, or method.

b) Performance Audits – (Refer to Section 5.4.2.j or 5.5.3.4) The results of such analyses shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.6.5 Asbestos Calibration

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

D.6.5.1 Transmission Electron Microscopy

Analytical Electron Microscopy (AEM) equipment requirements will not be discussed in this document.

D.6.5.1.1 Water and Wastewater

All calibrations listed below (unless otherwise noted) must be performed under the same analytical conditions used for routine asbestos analysis and must 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 must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000x and 20,000x. A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.1)

b) Camera Constant – The camera length of the TEM in the Selected Area Electron Diffraction (SAED) mode must be calibrated before SAED patterns of unknown samples are observed. The diffraction specimen must 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 must 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 (EPA /600/R-94/134, Method 100.2, Section 10.2). 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 must be displayed on control charts that show trends over time.

c) Spot Size – The diameter of the smallest beam spot at crossover must be less than 250 nm as calibrated quarterly. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.3)

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 ≥ 1 μm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 15 seconds.

e) EDXA System

1) The x-ray energy versus 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.

2) 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.

3) The k-factors for elements found in asbestos (Na, Mg, Al, Si, Ca, and Fe) relative to Si shall be calibrated semiannually, or any time 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 must be displayed on control charts that show trends over time.

4) 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 must be displayed on control charts that show trends over time.

5) 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.

6) 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-Cellulose-Ester (MCE) filters. Calibration data must 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 one opening/sample. The variation in the calibration measurements (2s) is <5% of the mean calibration value.

D.6.5.1.2 Air

All calibrations must be performed in accordance with Section 6.5.1.1 of this Appendix, with the exception of magnification. Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 15,000x to 20,000x (AHERA, III.G.1.c). A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

D.6.5.1.3 Bulk Samples

All calibrations must be performed in accordance with Section 6.5.1.2 of this Appendix.

D.6.5.2 Phase Contrast Microscopy

a) 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.

b) The phase-shift LOD 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 (refer to NIOSH 7400, Issue 2, 15 August 1994, Section 10b). This procedure ensures that the minimum detectable fiber diameter (< ca. 0.25μm) for this microscope is achieved.

c) 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, d_c (mm), of the circular counting area and the disc diameter must 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 must be re-measured (or re-calibrated) to determine field area (mm^2). Re-calibration 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 mm^2 to 0.00817 mm^2. The actual field area shall be documented and used.

D.6.5.3 Polarized Light Microscopy

a) 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. (Section 2.2.5.2.3, EPA/600/R-93/116, July 1993)

b) 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.

D.6.6 Analytical Sensitivity

D.6.6.1 Transmission Electron Microscopy

D.6.6.1.1 Water and Wastewater

An analytical sensitivity of 200,000 fibers per liter (0.2 MFL) is required for each sample analyzed (EPA /600/R-94/134, Method 100.2, Section 1.6). 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).

D.6.6.1.2 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 (AHERA, Table I).

D.6.6.1.3 Bulk Samples

a) The range is dependent on the type of bulk material being analyzed. The sensitivity may be as low as 0.0001% depending on the extent to which interfering materials can be removed during the preparation of AEM specimens. (Section 2.5.2, Range, Page 51, EPA/600/R-93/116)

b) There should be an error rate of less than 1% on the qualitative analysis for samples that contain chrysotile, amosite, and crocidolite. A slightly higher error rate may occur for samples that contain anthophyllite, actinolite, and tremolite, as it can be difficult to distinguish among the three types. (Section 3, Page 10, NIST Handbook 150-3, August 1994)

D.6.6.2 Phase Contrast Microscopy

The normal quantitative working range of the test 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. (NIOSH 7400, Issue 2, 15 August 1994)

D.6.6.3 Polarized Light Microscopy

The laboratory shall utilize a test 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 test method or applicable regulation.

D.6.7 Data Reduction

D.6.7.1 Transmission Electron Microscopy

D.6.7.1.1 Water and Wastewater

a) The concentration of asbestos in a given sample must be calculated in accordance with EPA /600/R-94/134, Method 100.2, Section 12.1. Refer to Section 5.4.7, “Computers and Electronic Data Related Requirements,” of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample (EPA /600/R-94/134, Method 100.2, Section 12.2.2).

D.6.7.1.2 Air

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized, e.g., AHERA. Refer to Section 5.4.7, “Computers and Electronic Data Related Requirements,” of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

D.6.7.1.3 Bulk Samples

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 5.4.7, "Computers and Electronic Data Related Requirements," of this document for additional data reduction requirements.

b) Measurement Uncertainties - PE for floor tiles analyzed by TEM following careful gravimetric reduction (New York ELAP Certification Manual, Item 198.4) has revealed an interlaboratory 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.

D.6.7.2 Phase Contrast Microscopy

a) Airborne fiber concentration in a given sample must be calculated in accordance with NIOSH 7400, Issue 2, 15 August 1994, Sections 20 and 21. Refer to Section 5.4.7, "Computers and Electronic Data Related Requirements," of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the intra-laboratory and inter-laboratory relative standard deviation with each set of results. (NIOSH 7400, Issue 2, 15 August 1994)

c) Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of the filter area covered with particulate should be reported as "uncountable" or "probably biased". Other fiber counts outside the 100-1300 fibers/mm^2 range should be reported as having "greater than optimal variability" and as being "probably biased."

D.6.7.3 Polarized Light Microscopy

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 5.4.6, "Computers and Electronic Data Related Requirements," of this document for additional data reduction requirements.

b) Method Uncertainties - Precision and accuracy must 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 (refer to EPA/600/R-93/116, July 1993, Table 2-1) should be generated for different concentrations of asbestos.

D.6.8 Quality of Standards and Reagents

D.6.8.1 Transmission Electron Microscopy

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

1) Reference standards that are used in an asbestos laboratory shall be obtained from the 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 ensure the quality of their products.

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

b) All reagents used shall be analytical reagent grade or better.

c) 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.

D.6.8.2 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 PE samples, such as PAT and AAR samples available from the AIHA, may be utilized as reference samples (refer to Section 6.2.2.b of this document) 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 NIOSH 7400, Issue 2, 15 August 1994).

D.6.8.3 Polarized Light Microscopy

Refer to Section 6.8.1 of this Appendix.

D.6.9 Constant and Consistent Test Conditions

The laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

QUALITY SYSTEMS FOR ANALYTICAL SERVICES

APPENDIX E

Non-Destructive Assay (NDA)

Note: DOE requirements are identified in gray boxes. In cases where DOE requirements differ from the NELAC requirements, DOE requirements supersede the NELAC requirements.

This appendix addresses quality assurance and control measures to be implemented by the NDA measurement organization. There are two subsections, one concerning quality assurance requirements that must be performed and documented, and another addressing quality control measures with criteria for acceptable performance and associated action limits.

E.1 Quality Assurance

E.1.1 Nondestructive Assay System Calibration

This section delineates requirements for establishment of a traceable NDA measurement system "initial calibration", confirmation of the "initial calibration" and the continuing verification of such. Procedures shall be developed and implemented for NDA measurement system calibration methods and processes. Per the purpose of this Appendix, the term calibration is referred to and defined in three separate ways: 1) initial calibration, 2) calibration confirmation, and 3) calibration verification.

The "initial calibration" is that fundamental calibration that addresses and accounts for the response of an NDA measurement system to radioactive materials present in the waste containers or process components of interest (measurement items). The "calibration confirmation" is a thorough corroboration of the "initial calibration" using traceable working reference materials (WRMs) and representative waste matrix/ process component configurations. The "calibration verification" is a periodic verification of the "initial calibration" to ensure on-going long-term data quality compliance through the period of NDA operations.

Procedural steps for calibration are not specified here. However, those elements that must be considered during the "initial calibration," "calibration confirmation", and "calibration verification" are enumerated. This allows the NDA measurement organization autonomy in devising and implementing techniques and analytical procedures for these three calibration definitions. Through these three mechanisms, the NDA measurement organization shall demonstrate the calibration and associated uncertainty is compliant with applicable client and/or end-user requirements initially and throughout the contract period.

E.1.1.1 Initial NDA System Calibration

An NDA measurement system "initial calibration" shall be performed to ensure the measurement system response provides valid data of known and documented quality. Calibrations shall be performed using traceable WRMs obtained from suppliers maintaining a nationally recognized reference base and an accredited measurement program. Full documentation of the calibration technique, process, and results is required. For cases where there is an insufficient number and denomination of traceable radioactive material standards to support the "initial calibration", the NDA organization can develop alternate calibration strategies based on available resources. Alternate strategies shall be clearly documented and technically justifiable.

The development and establishment of an "initial calibration" shall address the following as applicable:

a) SOPs shall be in place to specify steps/activities necessary to develop and determine the "initial calibration" including but not limited to, specification of traceable radioactive sources or their alternates, geometrical positioning of sources, traceable source/matrix media configurations, acquisition of NDA system response data, computational methods, analysis of response data to determine a robust calibration, calibration acceptance criteria, calibration applicability and qualifiers and calibration uncertainty.
b) The "initial calibration" shall be performed through the use of traceable working reference materials, unless exceptions have been stipulated and documented. For mass calibrations (i.e,, calibrations that use a direct measurement of the same isotopes, matrices, and containers that will subsequently be measured in unknown items), the radioactive material mass and matrix characteristics must span and bracket the range of anticipated values for the measurement items. For calibrations based on instrument response modeling, sufficient information shall be provided in the method description and calibration regimen to assure that the calibration measurements and model appropriately spans and brackets the anticipated analysis space (e.g., provide mechanisms to account for anticipated geometries, radioactive material mass, chemical composition, and matrix characteristics). For enrichment determinations using the enrichment meter technique, the initial calibration must span the range of enrichments in anticipated unknown item measurements.
c) The measurement uncertainty associated with the application of the "initial calibration" shall be established using a sound and technically defensible technique. Methods for the estimation of total measurement uncertainty (TMU) shall be developed and documented. Where applicable, the calibration uncertainty shall include terms for mass, matrix characteristics and configurations and radioactive material properties. These methods shall consider, at a minimum, uncertainty components, the calibration uncertainty model (method of uncertainty component propagation), estimates of uncertainty introduced by differences between item characteristics and calibration modeling assumptions. For example, if the model assumes a homogeneous distribution of the isotope of interest, the uncertainty

introduced if items are not homogeneous using a worst case distribution as determined through a documented engineering judgment including supporting data must be determined.

d) The NDA measurement method capability related to each initial calibration must be defined and documented. As applicable, this capability includes waste matrix types, process equipment types, geometries, configurations, radioactive material types, matrix density range, hydrogenous material range, radioactive material mass range, radioactive material compound, and other parameters affecting instrument response. The intent of defining the capability is to delineate those source/matrix configurations where the calibration is applicable and where it is not.

e) Where surrogate materials are used to simulate waste matrices, their configuration(s) must be nominally representative of the actual waste item population. The design of surrogate matrix configurations must be documented. Surrogate materials used to produce a given matrix configuration shall be carefully specified, procured and the resultant physical properties and configuration documented.

f) If NDA method manuals, national standards, or a mandated NDA calibration methods do not specify the number of traceable WRMs to span the mass/activity and radioactive material compound(s) characteristics of the waste/process component, a minimum number must be determined and technically justified. NDA organization must document this number and their denominations in a calibration SOP or other applicable document. This requirement does not necessarily apply to NDA methods that rely on modeling. However, the method used to assure that the calibration and model appropriately spans and brackets the anticipated analysis space (e.g., provide mechanisms to account for anticipated geometries, radioactive material mass, chemical composition, and matrix characteristics) as per item (b) above must be technically justified and documented. The For NDA methods that do not necessarily require calibration with source material similar in nature to the waste or process items (e.g., neutron counting), those source(s) used are still required to be traceable. However, accounting of the efficiency variation because of the composition of the actual radioactive material shall be assessed and corrected for (e.g., Californium (^{252}Cf) fission neutron spectrum counter efficiency versus uranyl fluoride (UO_2F_2) neutron spectrum efficiency.)

g) The “initial calibration” process shall be clearly documented including the calibration measurement configurations, data acquisition parameters, acquired data, data reduction methods, resultant calibration factors or expressions, statistical analyses and uncertainties. Records containing information pertinent to the calibration process shall be retained including but not limited to:

1) WRM and/or surrogate waste matrix configurations used to acquire instrument response data, calibration determination techniques,
2) SOP(s) used,
3) data acquisition parameters,
4) NDA system identification,
5) analytical software used,
6) traceable standard identifications,
7) analytical support equipment information,

8) electronic file storage locations.

Records shall be sufficient to allow reproduction of the "initial calibration".

h) The initial calibration shall be re-established when repairs or changes are made to the measurement system that are likely to affect one or more calibration parameters. Examples that may require repeating the initial calibration include, but not are limited to:
   1) major NDA system repairs or modifications,
   2) replacement of vital NDA measurement system components (e.g., collimator, multi-channel analyzer (MCA), neutron generator),
   3) change in collimator depth and/or aperture not accounted for in a model, and
   4) significant software modification and/or changes.

E.1.1.2 Calibration Confirmation

A confirmation of the "initial" NDA measurement system calibration shall be performed. In this context, confirmation means the "initial calibration" shall be assessed and determined to be correct and true by the objective collection of evidence supporting the calibration was properly established.

a) The "calibration confirmation" process is to produce objective evidence demonstrating the applicability and correctness of the "initial calibration" relative to the waste forms and process components of interest. The recommended method is to assemble test item(s) consisting of traceable source/matrix configuration(s) nominally representative of the waste form and/or process components to be characterized. They cannot be the same configurations used to establish the "initial calibration". They must contain a known and traceable radioactive element/isotope, mass/activity and/or enrichment in a known and representative matrix configuration. The confirmation test item(s) are then measured using the "initial calibration" of the NDA system. The number of differing test item configurations used to confirm the calibration is to be determined by the NDA organization and documented. The reported "calibration confirmation" measurement result must agree, with criteria as established by the NDA organization, with the known element/isotope, mass/activity and/or enrichment of the confirmation test item(s). The NDA organization acceptance criteria shall not exceed the criteria as presented in Section E.1.1.3 unless technically justified and documented.
b) The radioactive sources used for "calibration confirmation" purposes shall, to the extent practicable, be representative of the actual radioactive material compositions and chemical compounds as found in the measurement item inventory of interest.
c) Radioactive material standards used for "calibration confirmation" are to be traceable to a nationally recognized reference base (e.g., National Institute of Standards and Technology [NIST] or New Brunswick Laboratory [NBL]). The traceable standards used for "calibration confirmation" shall not be related to (from the same feedstock or lineage) those used to perform the "initial calibration". Noncompliance with this requirement, due to lack of a sufficient variety of traceable standards, can be temporarily waived provided an adequate alternate confirmation strategy is devised.

d) Calibration confirmation acceptance is assessed through the degree of agreement between the known "calibration confirmation" test item value and that as per the NDA confirmation measurement result. The NDA organization is to determine and document representative "calibration confirmation" source/matrix surrogate configuration(s). The NDA organization may also develop "calibration confirmation" bias and precision acceptance criteria specific to the NDA system and measurement items under consideration. Recommended "calibration confirmation" acceptance criteria are delineated in Section E.1.1.3.

e) Calibration confirmation results outside NDA organization defined acceptance criteria require implementation of corrective action(s) as applicable. Calibration confirmation results are not to exceed the maximum allowable acceptance criteria of Section E.1.1.3 unless the NDA organization has specifically determined and documented greater limits with the requisite technical justification.

f) For the case where a corrective action was required and subsequently implemented, the "calibration confirmation" process is to be repeated. Acceptable results must be obtained and documented before the NDA system is considered operational. Where a "calibration confirmation" failure was determined to be due to a minor issue (e.g., wrong constant, wrong efficiency file, or an inappropriate test item), the entire "calibration confirmation" measurement regimen may not need to be repeated. This is acceptable provided it is the true cause of the failure. All corrective actions and their effects, supporting data, results, etc., shall be documented and retained.

g) In the case where the "calibration confirmation" was acceptable for certain types or categories of radioactive material/waste matrix configurations, but unacceptable for other categories with distinctly different source/matrix properties, conditional acceptance of the "calibration confirmation" can be made. The NDA organization, however, must clearly identify which categories of source/matrix configurations are approved for NDA measurement and which are not. The technical basis for accepting certain source/matrix categories shall be documented and available for review. Recalibration or corrective action efforts should be implemented and documented for source/matrix categories not meeting acceptance criteria for "calibration confirmation".

h) The "calibration confirmation" process shall be performed following an initial calibration or when there are indications warranting a re-assessment of the "initial calibration", e.g., the source/matrix configuration of measurement items varies relative to the source/matrix configurations used to develop the "initial calibration". Additional causes for a performing a "calibration confirmation" include:
   1) major NDA system repairs or modifications,
   2) replacement of NDA measurement system components, e.g., detector, neutron generator or supporting electronic components that have the potential to affect data quality,
   3) re-calibration,
   4) significant changes to the NDA system software, and,
   5) relocation of the system (applies primarily to fixed stationary systems).

i) Records must be retained to permit reconstruction of any NDA measurement system "calibration confirmation"(e.g., NDA method, measurement system configuration, confirmation date, primary radioactive isotope(s), mass or concentration and response, calibration factor(s), or equations/coefficients used to convert NDA instrument response to mass/concentration). Documentation must explicitly connect the "calibration confirmation" data/records to the "initial calibration".

E.1.1.3 Calibration Confirmation Acceptance Criteria

a) Bias and precision limits are used to determine the acceptability of "calibration confirmation" measurements. The specified limits should be "upper limits" to be applied to all NDA measurement techniques over all matrix configurations. The recommended "calibration confirmation" limits are not specifically tied to end-user requirements, rather they are nominal performance levels expected of NDA systems. Failure to comply with these bias and precision limits is used as an indicator that more capable measurement techniques need to be developed.
b) NDA measurement system bias and precision should be determined through the acquisition of replicate measurements using matrix container and/or process component mock-ups combined with traceable WRMs. The source/matrix configurations are to be representative of the actual measurement item population of interest. The number of different source/matrix test configurations and replicate measurements of each shall be determined by the NDA organization and documented. The "calibration confirmation" bias is to be determined in terms of %Bias [(mean measured value - known value)/known value]*100 or %R (mean measured value/known value)*100. The bias shall not be outside the limits as per Table E-1 at the 95% confidence level.
c) Precision is reported as percent relative standard deviation (% RSD). The %RSD shall not exceed the value listed in the last row of Table E-1 for twenty replicate measurements of the "calibration confirmation" source/matrix test item(s). Equivalent %RSD limits for a number of different replicate values are tabulated in table E-2.

Table E-1. Calibration Confirmation Acceptance Limits

Confirmation Range	%Bias	%R
bias (lower limit)	-30	70
bias (upper limit)	30	130
precision	20% RSD at the 95% confidence level for 15 replicates	

Table E-2. Upper Limits for %RSD vs. Number of Replicates

Number of Replicates	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Max %RSD[a]	1.8	6.6	10.0	12.3	14.0	15.2	16.2	17.1	17.7	18.3	18.8	19.3	19.7	20.0

a – the values listed are derived from the measured standard deviation of the replicate measurements using

$$\frac{s}{\mu} \times 100\% < \sqrt{\frac{(0.292)^2 \times \chi^2_{0.05,n-1}}{n-1}} \times 100\%$$

where s is the measured standard deviation, n is the number of replicates, μ is the known or true value, $\chi^2_{0.05,n-1}$ is the critical value for the upper 5% tail of a one sided chi-squared distribution, with n-1 degrees of freedom, and the 0.292 constant corresponds to a 95% upper confidence bound on the true system precision limit of 29.2%.

NDA service providers may develop alternate methods and limits for bias and precision. Such alternate methods and limits must be technically defensible and clearly documented.

Failure to comply with the bias and precision requirements for "calibration confirmation" requires development of a corrective action plan (CAP). The CAP shall include detail on the nature of the failure, its suspected causes, methods to evaluate potential causes, and activities proposed to identify and rectify the deficiency. The CAP results shall be documented and show why the failure occurred and what actions were taken to prevent a re-occurrence. The calibration confirmation shall be performed again after the corrective actions in the CAP have been implemented and the results documented.

E.1.1.4 Calibration Verification

"Calibration verification" is a measure designed to provide continual and long-term information on the stability of the "initial calibration" while minimizing the impact on NDA operational schedules and resources. The "calibration verification" test item(s) must meet the bias acceptance criteria delineated in Section E.1.1.3. A "calibration verification" shall be performed at least once every five operational days for each measurement system and calibration in use. A five day operational period is defined as a rolling tally of five days where NDA operations were in effect, not necessarily consecutive. The start point for the five day operational period is from the start of approved operations or the first operational day after the previous rolling five day tally was completed. The five day operational "calibration verification" requirement may be extended to a maximum of thirty operational days provided the NDA organization can demonstrate and technically justify the long term stability of the NDA system per established acceptance criteria.

Calibration verification test items are typically selected from or assembled from the traceable standards and matrix containers or process component mock-ups used in the "calibration confirmation" process. The "calibration verification" test item is to be submitted to NDA operations in a "blind" manner, where applicable, and processed through the measurement routine as though it were an actual measurement item. The "calibration verification" test items are to be selected and/or configured and submitted such that during a 12-month period the operational space of the NDA system "initial calibration" is spanned. The "calibration verification" is a point check in the calibration realm. It is not required that each waste matrix type comprising the operational space of the NDA system be tested. However, it is expected that the "calibration verification" configurations vary over the operational space. The NDA organization is responsible for specification, assembly and selection of "calibration verification" test items and meeting the applicable rolling operational day period, (i.e., minimum five days, maximum thirty days).

Acceptable performance for a "calibration verification" measurement result in terms of bias, trending measures and so forth shall be determined and documented by the NDA organization. It is recommended that the "calibration confirmation" acceptability requirements of Section E.1.1.3 be considered in this process. A CAP for out-of-control "calibration verification" results is to be prepared by the NDA organization. The CAP shall include a provision requiring the evaluation of measurement item data potentially affected by the failed "calibration verification" measure. The "calibration verification" protocol, monitoring, acceptance criteria, action levels, etc., are to be clearly documented and readily available for review. The calibration verification data is to be control charted and monitored for trends over time.

The NDA organization can utilize other methods of "calibration verification" provided they are technically justifiable and documented.

E.1.1.5 NDA Method Detection Limit

A methodology shall be in place to determine NDA measurement system detection limit for those radionuclides specified per the client/end-user requirements. It shall be re-determined each time there is a significant change in the measurement method or matrix configuration. Instruments performing low-level waste discrimination measurements must have a minimum detectable activity (MDA)/lower limit of detection (LLD) sufficient to meet the acceptance criteria. The methodology for determination of the MDA/LLD is to be documented by the NDA organization.

The LLD is that level of radioactivity which, if present, yields a measured value greater than the critical level (Lc) with a 95% probability, where the Lc is defined as that value which measurements of the background will exceed with 5% probability (the LLD may be defined in a different manner to comply with specific client needs). Because the LLD is a measurement-based parameter, it is not feasible to calculate LLDs for radionuclides that are not determined primarily by measurement, e.g., ^{99}Tc. In such cases, the NDA organization shall derive the equivalent of an LLD (i.e., a reporting threshold for a radionuclide(s) when technically justified). This value may be based on decay kinetics, scaling factors, or other scientifically based relationships and must be adequately documented in site records.

The minimum detectable activity is that activity of an analyte in a sample that will be detected with a probability β of non-detection (Type II error) while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). For the purposes of this document, the alpha (α) and beta β) probabilities are both set at 0.05 unless otherwise specified.

E.1.1.6 Infinite Thickness

For a given radioactive material thickness (deposit or buildup), a thickness may be reached beyond which there is no increase in counts for an increase in thickness. At this point, infinite thickness has been reached. This phenomenon is typically only observed in gamma-ray counting. The NDA organization shall have a documented process for identifying infinite thickness when performing measurements. Some common techniques include:

a) Transmission Factor - ASTM C1133-89, 'Standard Test Method for NDA of Special Nuclear Material in Low Density Scrap and Waste by Segmented Passive Gamma-Ray Scanning,' ASTM, 1989.
b) Peak ratio - Software such as Multi-Group Analysis for Uranium.

E.1.1.7 NDA Measurement Uncertainty

NDA organizations shall have and apply methods and procedures for estimating total measurement uncertainty (TMU) for all reported values. The NDA organization shall perform a preliminary identification of uncertainty components and produce measurement uncertainty estimates for the waste population to be characterized prior to generating characterization data for the client/end-user. An estimate of the measurement uncertainty for the measurement item inventory of interest is to be performed and documented. The estimate shall be based on knowledge of the measurement method performance and make use of previous experience and validation data from similar measurement apparatus and configurations when available. The estimated measurement uncertainties must be evaluated per client and/or end-user needs and requirements. The method used to calculate TMU for the purpose of demonstrating compliance with client and/or end-user requirements must be documented and technically justified.

The NDA organization shall have a method to determine total measurement uncertainty for each NDA system employed including:

a) Develop a document or plan that delineates the approach to TMU determination, defines measurement uncertainty components, and determines a method for acquiring data/information on components of variance and processing of acquired data and information to arrive at technically defensible TMU for the measurement item population of interest.
b) Procedure or applicable document that provides specific direction on the acquisition of NDA system measurement data for use in deriving the TMU.
c) Produce documentation that clearly describes the processing of acquired data, accounting for all significant variables, and the application of methods to determine the TMU.
d) Clearly define how the TMU is expressed (e.g., 95% confidence level, percent, one-sigma, etc.)
e) The TMU determination method must be clearly documented; NDA organizations that utilize commercial off-the-shelf data analysis and uncertainty software are still accountable to produce clear documentation of the TMU approach, components of variance, and technique for arriving at the TMU value.

E.1.1.8 NDA Measurement Traceability

The calibration of NDA instrumentation and support measurement devices (e.g., weight scale), used for NDA characterization purposes shall have traceable calibrations established and documented before being put into service. Traceability is the ability to relate individual measurement results through an unbroken chain of calibrations to a nationally recognized reference base (e.g., NIST, r NBL, etc.). For NDA measurements, traceable materials include radioactive WRMs, certified weights for scale calibrations and thickness measurement methods.

a) The NDA organization shall have a program and procedures for establishing a traceable calibration as well as QC checking of its NDA instrumentation and support equipment. This program shall include a system for selecting, procuring, using, and controlling traceable reference standards for NDA measurement instrumentation and support equipment. For cases where traceable working reference materials are not yet available, the NDA organization may propose alternate methods that are technically defensible and clearly documented.
b) Traceable sources used for calibration shall be traceable for all attributes used for the calibration (e.g., a ^{252}Cf source shall be certified in its neutron yield and isotopic composition used to calculated the decay rate, and a mixed nuclide source used to perform an efficiency calibration of a gamma-ray detector shall be certified for the yield of each gamma ray energy used in the calibration and the decay properties of the contributing nuclides).
c) The NDA organization shall have a procedure(s) for the specification, procurement and acceptance of WRMs. The WRM certifications shall be acquired and maintained, and traceable to a nationally recognized reference base (e.g., NIST, NBL).

d) The NDA service provider shall retain records for all WRMs including the manufacturer/vendor, the manufacturer's Certificate of Traceability, the date of receipt, and a certificate expiration date.
e) Traceable standards shall be verified at a minimum of every five years. Standards with an expiration date less than five years shall be verified at a period equal to the time expiration time interval. Verification of a standard is accomplished through an assessment of its usable attribute to the NDA application (e.g., ^{235}U 185.7 keV gamma-ray emission rate and neutron emission rate). The area number of means by which a standard can be deemed verified as acceptable for use.
   1) The standard can be sent to a qualified facility maintaining measurement systems traceable to a certified reference material (CRM) for a determination of the standard attribute of interest. In this case the standard is simply given an updated attribute value and returned to the NDA organization with a revised or new certificate.
   2) Another method is to cross compare the standard with another traceable standard possessing the same attribute in a calibrated and operational measurement system. An evaluation of the results can produce a verification of the standard that is about to or has expired. The NDA organization must determine the acceptable uncertainty in the verified value relative to the NDA characterization process at hand.

   The verification method used and standard verification acceptability criteria shall be documented. The results of the verification are to be documented and maintained as a QA record.
f) WRM Certificates of Traceability shall contain information and data that clearly details traceability to a CRM.
g) Checks needed to maintain confidence in the status of WRMs shall be carried out according to defined procedures and schedules.
h) The NDA service provider shall have procedures for the safe handling, transport, storage and use of WRMs in order to prevent contamination or deterioration and protect their integrity.

E.1.1.9 NDA Measurement System Software

Software quality assurance (SQA) requirements must be implemented by NDA organizations that utilize software as part of NDA waste characterization, developed in-house or acquired.

When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage, or retrieval of NDA measurement data, the NDA organization shall have documentation or SOPs for software related activities. This documentation includes but is not limited to the following as applicable:

a) For software acquired from a commercial vendor or other third party, evidence of software quality control (QC), verification and validation (V&V) and other pertinent data shall be acquired and maintained by the NDA organization. Software verification is the process of evaluating software to determine whether the products of a given development phase satisfy the conditions imposed at the start of that phase (IEEE-STD-610). Software validation is the process of evaluating software during or at the end of the development process to determine whether it satisfies specified requirements. (IEEE-STD-610)

b) For software developed or modified in-house by the NDA organization, software development planning and QA controls shall be identified in documented plans. The following activities shall be addressed in such plans/procedures:
   1) Software development and testing,
   2) Software V&V,
   3) Software configuration control, and
   4) Software operation and maintenance.

c) Computer software developed by the NDA organization shall be documented per applicable software development quality standards. Such standards usually require documentation, including:

   1) Software specification document,
   2) Software design document,
   3) Software test plan, and
   4) Software V&V document (Note: Commercial off-the-shelf software [e.g., word processing, database and statistical programs in general] used within its designed application range are usually considered to be sufficiently validated). However, NDA organization developed software and/or modifications to commercial software must be validated. Installation and operability checks shall also be performed.

d) Software change procedures shall include requirements for the requesting, testing, quality assurance, approving, and implementation of changes.
e) Data including but not limited to, decay constants, branching ratios, material attenuation values, neutron yields, and master gamma libraries used in the reduction of processing of NDA measurement data to a reportable quantity, whether electronic or hardcopy, shall be placed under a control system so only authorized individuals have access.
f) Working data or source files (e.g., nuclear data libraries, master gamma libraries, geometry files, and efficiency files) shall be controlled by the NDA organization to prevent unauthorized access or inadvertent changes and controlled to document changes by authorized users to allow for re-creatability of the data used.
g) When commercial software is used that has the capability of performing user-defined calculations or macros (e.g., spreadsheet), all user-defined components shall be verified before initial use and after changes. Documentation of such shall be readily available for review. Appropriate protections must be included to preclude inadvertent changes to user-defined equation or macros. Printouts from any spreadsheet should include that information used to calculate the result;
h) Software version control methods must be in place to document the software version currently used as well as data reports with the date and time of generation and the software version used to generate the data report. Software that includes user-defined calculations and/or macros shall also track revisions to the user-defined customization using version information.
i) and confidentiality of data entry or collection, data storage, data transmission and data processing.

j) Computers and automated equipment are to be maintained to ensure proper function and must have appropriate environmental and operating conditions necessary to maintain the integrity of NDA measurement data and information.
k) Procedures are to be established and implemented for the maintenance of security of data, including the prevention of unauthorized access to and the unauthorized amendment of, computer records.
l) An inventory of all applicable software used to generate NDA characterization data shall be maintained that identifies the software name, version, classification and exemption status (DOE 0 414.C or latest version), operating environment, and the person and organization responsible for the software.
m) Maintain a historical file of software, software operating procedures, software changes, and software version numbers.

E.1.1.10 Acceptable Knowledge

NDA methods typically directly quantify one or more of the prevalent radionuclides known to be present in the waste and process component items. Other radionuclides may be present, some of which are not readily quantifiable through the NDA method being employed. NDA measurement campaigns often require that radionuclide not directly measureable by NDA methods be quantified and/or the minimum detectable activity determined and reported.

For radionuclides to be reported per contractual requirements, but not quantifiable through existing NDA techniques, isotopic ratios or radionuclide scaling factors based on acceptable knowledge (AK) of the facility process are commonly employed. The radionuclides and isotopes that are quantifiable through the NDA methods are used in conjunction with AK derived ratios and scaling factors to quantify the radionuclides not directly quantifiable. To use AK to determine such ratios and scaling factors, the NDA organization must technically justify the AK data and its use with NDA measurement information. The AK ratios or scaling factors must be appropriate to the generation point of the waste, process component, etc.

a) AK Documentation
The use of AK information concerning the radiological composition of a waste type or process component must be documented either in an AK summary report for that waste type/component or other controlled document. Should this information be contained in AK package(s) prepared to meet other general waste characterization requirements, it need not be duplicated in other controlled documents that address the radiological properties of the waste stream. However, all relevant information must be included in the AK record.

All ratios or scaling factors used must be technically sound and based on known, documented relationships or correlations. Uncertainties reported when using ratios and scaling factors are used must include the uncertainty in the ratio or scaling factor.

The type and quantity of supporting documentation may vary by waste stream and shall be compiled in a written record that includes a summary identifying all sources of information used to delineate the waste stream's isotopic distribution or radionuclide scaling factors. The basis and rationale for the delineation shall be clearly summarized in an AK report and traceable to referenced documents. Assumptions made in this rationale shall be identified. The following information should be included as part of the AK written record:

1) Map of the site with the areas and facilities involved in waste generation and process equipment identified,
2) Facility mission description as related to radionuclide-bearing materials and their management (e.g., routine production, fuel research and development, and experimental processes),
3) Description of the specific site locations (such as the area or building) and operations relative to the isotopic composition of the uranium bearing wastes and process components they generated,
4) Waste identification or categorization schemes used at the facility relevant to the waste material's isotopic distribution (e.g., the use of codes that correlate to a specific isotopic distribution and a description of the isotopic/radionuclide composition of each waste stream),
5) Information regarding the waste's physical and chemical composition that could affect the isotopic distribution (e.g., processes used to remove ingrown daughters or alter its expected contribution based solely on radioactive decay kinetics), and
6) Statement of all numerical adjustments applied to derive the material's isotopic distribution (e.g., scaling factors, decay/in-growth corrections and secular equilibrium considerations).

Documentation must be sufficient to enable independent calculation of the scaling factor or ratio of interest.

b) Supplemental AK Information

Supplemental AK information should be obtained dependent on availability. The amount and type of this information cannot be mandated, but information should be collected as appropriate to support contentions regarding the waste's isotopic distribution. This information is used to compile the waste's AK written record. Supplemental AK documentation that may be used includes but is not limited to information from the following sources:

1) Safeguards and security, materials control and accountability, and other nuclear materials control systems or programs and the data they generated,
2) Reports of nuclear safety or criticality, accidents/excursions involving the use of special nuclear material (SNM), or nuclear material,

3) Waste packaging procedures, waste disposal, building or nuclear material management area logs or inventory records, and site databases that provide information on SNM or nuclear materials,
4) Test plans, research project reports, or laboratory notebooks that describe the radionuclide content of materials used in experiments,
5) Information from site personnel (e.g., documented interviews), and
6) Historical analytical data relevant to the isotopic distribution of the waste stream.

c) AK Discrepancy Resolution

If there is any form of discrepancy between AK information related to isotopic ratios or composition, the NDA organization is responsible for having the sources of the discrepancy evaluated to determine information credibility. Information that is not credible or information that is limited in its applicability to the NDA characterization effort will be identified as such, and the reasons for dismissing it will be justified in writing. Limitations concerning the information will be documented in the AK record and summarized in the AK report. In the event the discrepancy cannot be resolved, the site will perform direct measurements for the impacted population of containers or process items. If discrepancies "result in a change to the original determinations, the AK summary will be updated.

E.1.1.11 NDA Data Reporting, Review, and Verification

a) NDA Measurement Data Reporting

The NDA organization is to document individual NDA measurement item results in a standard report format. For each NDA measurement item (waste container/ process component) there shall be a separate report. The NDA measurement item reports shall contain or reference the location of information sufficient to fully describe all input data, NDA measurement configuration information, acquisition parameters, analysis technique, software version, QC data, etc. to allow reconstruction of the reported results.

1) Title and contact information, including:
   a) Report title (e.g., "NDA Measurement Item Report"),
   b) Name of NDA organization,
   c) Client contact name for which report is to be delivered and NDA service provider point of contact responsible for ensuring the submittal of the report in the approved manner, and
   d) Identification of project name, site, or facility NDA measurement items are associated with.

2) Measurement item identification and QC information:
   a) Measurement item identification/designator and other identifiers/designations as applicable (e.g., the clients own identifier),
   b) Date(s) of NDA data acquisition,
   c) Analysis, background, and QC file names,
   d) Measurement item description,
   e) NDA field worksheet file name, log name, or other identifier,
   f) Gross/net weight, if applicable,
   g) NDA measurement live time, and
   h) Location of NDA measurement system, site name, facility name, building name, and other identifying information.

3) Primary radionuclide measurement results:
   a) Primary NDA measurement quantitation method (e.g., gamma, neutron),
   b) Primary radioisotopes and their associated TMU s in appropriate units, (for example, gram, activity, activity concentration, MDA, and % uncertainty),
   c) Total radionuclide mass, activity, concentration, and associated TMU,
   d) ^{235}U fissile gram equivalent and associated TMU (gram), and
   e) Other primary quantities such as uranium enrichment weight percent (wt%) and associated wt% TMU.

4) NDA acquisition and analysis information:
   a) NDA detector or system identification,
   b) Name of ancillary data and/or information sheets associated with the NDA measurement item. These are often called NDA Field Worksheets and contain information pertinent to the analysis of the acquired data such as container fill height and measurement configuration (e.g., detector to item distance and operator signature/date),
   c) Identification of real time radiography examination files, if applicable,
   d) The acquisition software identification and version, and
   e) Analysis software identification and version.
5) Comment/Narrative section:
   a) Name or reference to procedures used to acquire the NDA measurement data analyze the data, and acquire supporting data/information used in analysis,
   b) Name or reference to QC procedures utilized in the acquisition and processing of the data,
   c) Identification or reference to WRM and check source(s) used for calibration and/or QC activities,

d) Identification of or reference to calibration procedures and records and/or location, and,
e) If not specified elsewhere, definition of the quoted uncertainties (i.e., one σ, two σ). When TMU is reported differently on the batch cover sheet of the IMS, the method of expressing TMU shall be specified on the NDA measurement item report sheet or the applicable procedures referenced.

The NDA measurement item report is to have the analyst signature and date and the independent technical reviewer signature and date.

b. NDA Data Review

All NDA measurement data must be reviewed and approved by qualified personnel prior to being reported. At a minimum, the data and analysis must be reviewed by an independent technical reviewer (a second qualified person). This reviewer shall be an individual other than the data generator (analyst) who is qualified to have performed the initial work. The independent technical reviewer shall verify, at a minimum, the following information:

a) NDA measurement system QC results are within established control limits and, if not, the data have been appropriately dispositioned using the nonconformance process. This shall include a complete summary of qualitative and/or quantitative data for all items with data flags or qualifiers;
b) “calibration verification” measurements were performed and reviewed as acceptable;
c) NDA system data acquisition and reduction were conducted in a technically correct manner in accordance with current methods (verification of procedure and revision);
d) Calculations performed outside of software that is in the software QA program have been reviewed by a valid calculation program, a periodic spot check of verified calculation programs (not required with every report) and/or 100 percent check of all hand calculations;
e) Proper constants such as half lives, branching ratios, attenuation values, neutron yields, gamma libraries were used;
f) Data were reported in the proper units and correct number of significant figures;
g) Values that are not verifiable to within rounding or significant difference discrepancies must be rectified prior to completion of independent technical review;
h) The data have been reviewed for transcription errors;
i) Calibrations have been documented;
j) Standards used are traceable to nationally recognized certificates.

c. NDA Data Verification

Data verification is a systematic confirmation by examination and provision of evidence that specified requirements have been met data to ensure that the required data quality characteristics have been obtained. The verification process ensures that applicable quality controls have been properly implemented and data validity per program requirements has been met. Verification activities are usually performed at the batch level where all QA elements ranging from NDA measurement reports to compliance with applicable regulations are collected, collated, and prepared for submittal. NDA measurement data reports are to be provided to the client on a batch basis as determined with and agreed to by the client.

1) Batch data reports are to be prepared for each measurement batch on standard form (hard copy or electronic equivalent). Batch data reports shall at a minimum include the following:
   a) NDA organization name, NDA measurement system identification, batch number, NDA measurement item identifications included in the batch, date and signature release by authorized personnel;
   b) Table of contents
   c) QC data, backgrounds, replicate data, and control charts, etc., for the relevant batch time period;
   d) Data verification per the NDA service provider QA Plan, and as per applicable procedures.

2) Batch reports must be reviewed and approved by qualified personnel before being submitted. Only appropriately trained and qualified personnel shall be allowed to perform data verification/review. Verification reviews shall ensure:
   a) The QC documentation for the batch report is complete and includes as applicable a list of containers in the set or batch and applicable set or batch QC results.
   b) Data were collected as described in the planning documents and are complete and correct. All batch data reports must be approved by the project manager or designee. The project manager shall verify at a minimum the following information:
      (1) Data generation-level verification have been performed by a second qualified person and appropriate signature release,
      (2) Batch review checklists are complete,
      (3) Batch reports are complete and data are properly reported (e.g., data are reported in the correct units and with the correct number of significant figures), and
      (4) Data comply with program objectives.

Results of the review may require that qualifiers be placed on the use of the data. Verification methods shall be planned and documented. The documentation shall include the acceptance criteria used to determine if the data are valid. For noncompliant data, corrective action procedures shall be implemented.

E.1.1.12 NDA Measurement Performance Evaluation

The NDA organization shall demonstrate that its NDA methods, calibrations and uncertainty estimates are applicable to the matrix/process components. Part of this demonstration of proficiency is the participation in performance evaluation (PE) programs as scheduled and conducted by specified qualification and approval agencies, if available. Elements of the performance evaluation process include:

a) NDA organization shall demonstrate successful participation in applicable PE program(s). The NDA organization shall demonstrate continued proficiency throughout its' the term of operation. The testing will be single-blind and representative of the matrix types and configurations, and analytes (^{235}U, ^{238}U, etc.) to be characterized;
b) Unacceptable NDA results for PE test sample(s), as determined per PE program criteria, will require the NDA organization to implement corrective action procedures and submit a corrective action plan to the PE program or applicable oversight agency. Results of the corrective action plan shall be documented and available for review. .
c) Documentation of successful capability demonstration such as a Certification Statement or letter of concurrence from the qualifying agency must be acquired .and retained by the NDA organization. All associated supporting data necessary to reproduce the PE measurement results as contained in the Certification Statement or equivalent document must be retained by the NDA organization.
d) Once the initial capability demonstration is successfully completed, continuing demonstration of method performance is to be accomplished through the periodic "calibration verification" measurements as well as all applicable QC requirements.

E.2 NDA Quality Control

The purpose of a measurement control program is to test and ensure the stability of the measurement process and to gain additional information on measurement uncertainties where practicable. The measurement control program provides for the administration, evaluation, and control of measurement processes. The design of the measurement control program is to ensure the NDA measurement process provides data of sufficient quality (i.e., the measurement system is in control per defined criteria). The NDA organization can then make and document qualifying statements about the suitability and validity of measurement data as generated for the client and/or end-user.

QC measurements are to be performed in conjunction with and related to a batch of NDA measurement items. A batch is a grouping of similar measurement items to which a set of QC criteria is applied to demonstrate acceptability of the results. The batch size is specified to be 20 items such that when one replicate is performed per batch, a 5% check of the data is achieved. In addition to the replicate requirement are pre- and post-batch QC checks (e.g., background and energy calibration checks). A batch can be fewer than 20 items as for the case where there are fewer than 20 similar measurement items available for analysis or other driving circumstances, such as throughput requirements.

For each measurement item batch, QC measures are to be performed before commencement of a batch and at the end of the batch. An analytical batch may span a period of more than one day, but the requirement to perform QC checks per day is not superseded. The replicate QC measure does not have to be performed twice per batch, but rather once. Performance checks shall bracket the NDA measurements which comprise the batch. Out of control performance checks for a given NDA instrument shall cause the batch data to be considered suspect. Corrective actions shall be in place to evaluate the measurement item results for the affected batch.

E.2.1 QC Procedures

The NDA organization shall have procedures implementing applicable QCs for monitoring the validity of NDA measurements and the analytical results. The NDA QA program shall specify qualitative and quantitative acceptance criteria for the QC checks. The NDA QC measures and acquired information/data shall be documented or logged in such a way that trends are detectable. Statistical techniques shall be applied to the evaluation of acquired QC data and action levels specified. Procedures shall also be in place to implement the corrective action process when QC criteria are not satisfied. The QC program shall be periodically reviewed. In addition, the NDA service provider shall address the following:

a) Development of a QC plan with clearly defined roles and responsibilities. The QC program should assure objectivity and independence of action. The person assigned responsibility for the QC program shall be knowledgeable of the measurement system being controlled, statistical QC, and the process being monitored. The organization should provide sufficient separation of functions to avoid any conflict of interest.
b) Acquisition and maintenance of suitable WRMs and check sources to monitor measurement system performance during NDA characterization operations. Records concerning specification and acquisition of standards and sources, including an assessment of their uncertainties and procurement shall be documented and retained.
c) QC checks shall include a means to evaluate the variability and/or repeatability of NDA measurement results.
d) Determination of measurement parameters and acceptance criteria necessary to ensure the accuracy of the NDA method using daily performance checks and analysis of performance check data (e.g., control charts, trending analysis, and replicate measurements).
e) QC protocols as specified in the NDA organization method manual and/or procedure(s) shall be followed.
f) QC measurement parameter action levels shall be established and documented.

g) Written procedures shall be developed and documented to address out-of-control conditions and the subsequent re-qualification of the instrument.

E.2.2 NDA QC Requirements

Procedures cited in various ASTM, ANSI standards, NRC standard practices, and guidelines as referenced in Appendix A are recommended for use at all NDA measurement facilities. QC requirements must at a minimum include the following:

a) Background Measurements must be performed and recorded for neutron and gamma systems for each system in use at least once per day and twice for each batch. The once per day background measurement can serve as the beginning or ending background measurement required for the batch. The two background measurements for each batch shall bracket the start and end of the batch, one at the beginning of the batch and one at the end of the batch, unless technical justification to do otherwise is developed and documented. The count time for neutron and gamma background checks shall be at least as long as the measurement count time unless otherwise specified and documented by an appropriately qualified individual. The background measurement shall be evaluated before daily NDA measurements commence. Depending on environmental conditions, the background frequency may need to be increased to ensure data quality. Increases in the frequency of background measurements shall be determined and documented by an appropriately qualified individual (Note: Enrichment measurement systems that employ an infinite-thickness analysis technique do not require a background performance check). The recorded background data is to be monitored using control charts or tolerance charts to ensure the background environment is within statistical control. Contributions to background because of radiation from nearby radiation producing equipment, standards, or wastes must be controlled to the extent practicable or more frequent background checks must be performed.

b) Instrument Performance Measurement checks must be acquired for each NDA measurement system in use at least once per day and twice for each data batch. For each performance check two measurements shall be used to bracket the batch, one before and one after the batch measurements are completed. Performance checks include detection efficiency checks, matrix correction checks, and for spectrometric instruments, energy calibration and energy resolution checks. The NDA organization is to establish acceptable performance check ranges or limits as applicable. An out-of control energy calibration check may cause measurement item results to be suspect since the last successful energy calibration check. Energy calibration checks can be performed at a greater frequency than once per day. Performance checks, as applicable, shall also be acquired on support equipment. The recorded performance measurement checks is to be monitored using control charts or tolerance charts to ensure the instrument performance is within statistical control.

c) Replicate Measurements are used to determine the repeatability of a measurement system that represents the intrinsic instrument variability. Repeatability variance is a short-term variance usually dominated by counting statistics. The replicate measurement is acquired by randomly selecting one measurement item that has been processed through the NDA system for the batch. This measurement item is then to be re-measured using the same NDA system, software, and acquisition/reduction parameters. Data analysis is to be performed independently for the two measurements. The second measurement of the item is to be performed any time before the start of the next data set or batch. This repeat measurement is then the replicate for that batch. A minimum of one replicate measurement is required for each batch. For a randomly selected replicate measurement item that corresponds to a measurement below the lower limit of quantitation (LLQ), the 95% uncertainty ranges of the pair of measurements must overlap.

When two replicates are utilized to assess repeatability, the data should be evaluated using the Relative Percent Difference (RPD) as follows:

$$\frac{|D-S|}{S} \times 100\% \leq 25\%$$

Where:

S = initial randomly selected measurement item

D = duplicate result for measurement item S

An acceptable RPD shall be less than or equal to 25% or other criteria specifically requested by the client. A control chart of the RPD shall be maintained for trending analysis. Procedures shall be established for the collection, processing and periodic evaluation of replicate data. Alternate methods for determining repeatability and assessing its acceptability may be implemented by the NDA organization provided they are technically justifiable, documented and available for review. The replicate data is to be monitored using control charts or tolerance charts to ensure the instrument reproducibility is within statistical control.

Check sources used for QC checks should be traceable, long-lived and provide adequate counting statistics for a relatively short count time. If the check source is not traceable, it should be correlated with a traceable source or well known, characterized and documented.

All performance data shall be monitored on an as-recorded basis and over time using control charts and trending techniques. Most monitoring techniques assume that measurement data are distributed normally and that observations are independent. The assumption of normality should be assessed prior to implementation of a control regimen. The NDA organization is responsible for determining acceptance criteria for as-recorded and long term data trending. Recommended control chart limits and actions levels are contained in Table E-2. Corrective action plans or procedures shall be in place to manage out-of-control results and the associated measurement item data.

E.3 QC Action Levels and Response

Quality control measurements shall be performed on a periodic basis as prescribed above and evaluated relative to established acceptance criteria. Quality control measurements shall also be reviewed and evaluated over time to determine continued acceptability of the assay system and to monitor trends. If daily quality control checks yield results that are outside the acceptable range(s), the required responses in Table E-3 must be followed. The NDA service provider may implement more restrictive control limits and other administrative limits as applicable. All control limits and associated actions are to be documented and maintained.

Refer to Table E-3 Range of Applicability, page E-25

Table E-3 Range of Applicability

Category	Acceptability Range[a]	Required Response
Acceptable Range	$\lvert Data \rvert$ [b] $\leq 2\sigma$[c]	No action required
Warning Range	2σ[c] $< \lvert Data \rvert \leq 3\sigma$[c]	The performance check shall be rerun no more than two times. If the rerun performance result is within 2σ, then the additional performance checks shall be documented and work may continue. If the system does not fall within the ± 2σ after two rerun performance checks, then the required response for Action Range shall be followed.
Action Range	$\lvert Data \rvert > 3\sigma$[c]	Work shall stop and the occurrence shall be documented and appropriately dispositioned (e.g., initiating a nonconformance report). The NDA system shall be removed from service pending successful resolution of the failure cause. All assays performed since the last acceptable performance check, are suspect, pending satisfactory resolution. At a minimum, a "calibration verification" is required prior to returning the system back to service.

a - American National Standards Institute. *Nondestructive Assay Measurement Control and Assurance*, ANSI N15.36.
b - absolute value
c - the standard deviation is only based on the reproducibility of the data check measurements themselves. This is not TMU.