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Quality Systems Manual for

Environmental Laboratories

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Environmental Laboratories

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Preface

The Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) Data Quality Workgroup (DQW) participants developed the DoD/DOE Quality Systems Manual for Environmental Laboratories (QSM). The QSM provides requirements for accreditation to the DoD Environmental Laboratory Accreditation Program (ELAP) and the DOE Consolidated Audit Program Accreditation Program (DOECAP-AP).

The QSM incorporates requirements from International Organization for Standardization (ISO)/International Electrotechnical Commission (IEC) 17025:2017, *General requirements for the competence of testing and calibration laboratories*, NELAC Institute (TNI) EL-V1-2016-Rev.2.1, and DoD/DOE requirements. Conformance to the requirements contained in this standard is mandatory for any laboratory that is seeking or maintaining accreditation in accordance with the DoD ELAP and/or the DOECAP-AP.

To alleviate issues of copyright and provide a standard that is freely available to all, the QSM is presented in a format which must be used in conjunction with the ISO/IEC 17025:2017 Standard. Module 2 of the QSM presents additional DoD/DOE and TNI requirements as text in the order in which topics are addressed in the ISO/IEC 17025:2017 Standard. For requirements that come from the TNI 2016 Standard, a reference to their location has been included in Module 2. Other modules follow the TNI 2016 structure as applicable, although numbering has changed in places (e.g., initial number has been removed in all modules). Information that may be beneficial to a laboratory is marked as a “Note.” Notes do not contain requirements and do not form an integral part of this standard. To the extent possible, DoD and DOE requirements have been consolidated. Requirements that are unique to either DoD or DOE are marked as such.

The QSM is international in scope and is applicable to any laboratory seeking accreditation to this standard regardless of size or complexity. Nothing in this document relieves any laboratory from complying with more stringent contract specifications, host-nation final governing standards, or federal, state, tribal, and local regulations. Laboratories may continue work on existing contracts under the project-required version of the QSM. Laboratories shall be assessed to this version of the QSM during their routine accreditation cycle, beginning six months after the release date. All laboratories shall be assessed to this version of the QSM no later than two years and six months after the release date.

The DoD and DOE reserve the right to periodically issue new requirements or clarifications to current requirements as addenda posted on DoD Environment, Safety, and Occupational Health Network and Information Exchange (DENIX) and the DOE website.

This manual was created in the spirit of cooperation between agencies for the purpose of consolidating and improving quality systems. The DoD and DOE expert committee members wish to thank the many volunteers and reviewers that provided insight and guidance into the resolution of complex scientific issues that are now a part of this manual. Additional appreciation is extended to TNI for allowing use of TNI EL-V1-2016-Rev.2.1 language to be included within the body of this document.

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Acronym List

AA	Atomic Absorption
AAR	Asbestos Analysts Registry
AB	Accreditation Body
ACM	Asbestos-Containing Material
AFFF	Aqueous Film Forming Foam
AIHA	American Industrial Hygiene Association
AIHA LAP	AIHA Laboratory Accreditation Programs, LLC
ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BSC	Background Subtraction Count
CAA	Central Accumulation Area
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CHP	Chemical Hygiene Plan
CLCS	Conductivity Limit Check Sample
CPM	Counts per Minute
CRM	Certified Reference Material
CS	Calibration Source
CSU	Combined Standard Uncertainty
CV	Coefficient of Variation
CVAA	Cold Vapor Atomic Absorption
DDT	4,4'-Dichlorodiphenyltrichloroethane
DENIX	DoD Environment, Safety, and Occupational Health Network and Information Exchange
DER	Duplicate Error Ratio
DFTPP	Decafluorotriphenylphoshpine
DL	Detection Limit
DMR-QA	Discharge Monitoring Report Quality Assurance
DOC	Demonstration of Capability
DoD	Department of Defense
DOE	Department of Energy
DOECAP	Department of Energy Consolidated Audit Program
DOECAP-AP	Department of Energy Consolidated Audit Program Accreditation Program
DOE-DQW	Department of Energy Data Quality Workgroup
DOT	Department of Transportation
DPM	Disintegrations per Minute
EC	Escherichia coli
EDQW	Environmental Data Quality Workgroup
EDXA	Energy Dispersive X-Ray Analysis
EICP	Extracted Ion Current Profile
EIS	Extracted Internal Standard
ELAP	Environmental Laboratory Accreditation Program

EMPC	Estimated Maximum Possible Concentration
EPA	Environmental Protection Agency
FB	Field Blank
FoPT	Fields of Proficiency Testing
FoA	Field of Accreditation
FWHM	Full Width at Half Maximum
GC	Gas Chromatography
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/FID	Gas Chromatography/Flame Ionization Detector
GC/MS	Gas Chromatography/ Mass Spectrometry
HAZWOPER	Hazardous Waste Operations and Emergency Response
HPLC	High Performance Liquid Chromatography
HPLC/UV	High Performance Liquid Chromatography/Ultraviolet
HSE/NPL	Health and Safety Executive/National Physical Laboratory
IB	Instrument Blank
IC	Ion Chromatography
ICC	Instrument Contamination Check
ICAL	Initial Calibration
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
IC/MS	Ion Chromatography/Mass Spectrometry
IC/MS/MS	Ion Chromatography/Mass Spectrometry/Mass Spectrometry
ICS	Interference Check Solution
ICV	Initial Calibration Verification
ID	Identification
IEC	International Electrotechnical Commission
IH	Industrial Hygiene
IRCS	Internal Recovery and Calibration Standard
IS	Internal Standard
ISC	Instrument Sensitivity Check
ISE	Ion Selective Electrode
ISM	Incremental Sampling Methodology
ISO	International Organization for Standardization
IT	Information Technology
LC	Liquid Chromatography
LC/MS	Liquid Chromatography/ Mass Spectrometry
LC/MS/MS	Liquid Chromatography/ Mass Spectrometry/ Mass Spectrometry
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LLCCV	Low-Level Continuing Calibration Verification
LLLCS	Low-Level Laboratory Control Sample
LLOQ	Lower Limit of Quantitation
LOD	Limit of Detection
LOQ	Limit of Quantitation
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols
MB	Method Blank
MCE	Mixed cellulose ester
MD	Matrix Duplicate
MDA	Minimum Detectable Activity
MDC	Minimum Detectable Concentration

MDL	Method Detection Limit
MDLb	Method Detection Limit blank
ME	Marginal Exceedance
MPN	Most Probable Number
MQO	Measurement Quality Objective
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NELAC	National Environmental Laboratory Accreditation Conference
NFPA	National Fire Protection Association
NIOSH	National Institute for Occupational Safety and Health
NIS	Non-Extracted Internal Standard
NIST	National Institute of Standards and Technology
NMI	National Metrology Institute
NOAEC	No-Observed-Adverse-Effect Concentration
NOEC	No-Observed-Effect Concentration
OPR	Ongoing Precision and Recovery
PAH	Polycyclic Aromatic Hydrocarbon
PAT	Proficiency Analytical Testing
PCB	Polychlorinated biphenyl
PCM	Phase Contrast Microscopy
PDS	Post Digestion Spike
PFAS	Per- and Poly-fluoroalkyl Substances
PLM	Polarized Light Microscopy
PMSD	Percent Minimum Significant Difference
PPE	Personal Protective Equipment
PT	Proficiency Testing
PTRL	Proficiency Testing Reporting Limit
QA	Quality Assurance
QC	Quality Control
QCAL	Method Calibration - Quench Curve
QSM	Quality Systems Manual
%R	Percent Recovery
RMB	Radiation Measurement Batch
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RE	Relative Error
RI	Refractive Index
RML	Radioactive Materials License
ROI	Region of Interest
RMP	Reference Material Producer
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRT	Relative Retention Time
RSD	Relative Standard Deviation
%RSE	Relative Standard Error
RSO	Radiation Safety Officer
RT	Retention Time
S/N	Signal to Noise Ratio
SAED	Selected Area Electron Diffraction
SDWA	Safe Drinking Water Act
SI	Système International d'unitès (International System of Units)

SIC	Spectral Interference Check
SIM	Selected Ion Monitoring
SRM	Standard Reference Material
SRT	Standard Reference Toxicants
STS	Sample Test Source
SVOC	Semivolatile Organic Compounds
TCLP	Toxicity Characteristic Leaching Procedure
TDU	Thermal Desorption Unit
TEM	Transmission Electron Microscopy
TEQ	Toxicity Equivalents
TNI	The NELAC Institute
TSCA	Toxic Substances Control Act
TSDF	Treatment, Storage, and Disposal Facility
USDOE	United States Department of Energy
USEPA	United States Environmental Protection Agency
UV	Ultraviolet
VER	Calibration Verification used in EPA 1613
VOA	Volatile Organic Analysis
WET	Whole Effluent Toxicity

Module 1

Proficiency Testing

1.0 Introduction

This module provides specific proficiency testing (PT) requirements for laboratories seeking accreditation to this standard. Laboratories seeking accreditation for testing shall also meet the requirements presented in Module 2, and any applicable testing modules of this standard. In addition to the reporting requirements identified in this module, the laboratory shall also meet any applicable PT reporting requirements from its DoD ELAP accrediting body (AB) and/or DOECAP-AP AB. Participation in a PT program as specified in this module is a requirement for accreditation, except for industrial hygiene testing. See Module 8 for industrial hygiene PT requirements. This module does not supersede any regulatory requirements.

2.0 Scope

The requirements in this module align with Volume 1 Module 1 of The NELAC Institute (TNI) EL-V1-2016-Rev.2.1 standard. Compliance with Volume 1 Module 1 of TNI EL-V1-2016-Rev.2.1 for a field of accreditation (FoA) that is also designated as a TNI field of proficiency testing (FoPT) is sufficient to meet the requirements of this module. Additional requirements have been added for laboratories seeking accreditation for a FoA that is not also designated as a TNI FoPT.

3.0 Terms and Definitions

Definitions are considered requirements. Definitions are included in Module 2, Section 3.0. Additional definitions related to this module are listed below.

Field of Accreditation: Matrix, technology/method, and analyte combination for which the accreditation body offers accreditation.

Field of Proficiency Testing: Matrix, technology/method, analyte combination for which the composition, spike concentration ranges, and acceptance criteria have been established by TNI's Proficiency Testing Program Executive Committee.

Primary Accreditation Body: The accreditation body responsible for assessing a laboratory's total quality system and tracking PT performance for fields of accreditation.

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

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

Proficiency Testing Provider: A person or organization accredited to ISO 17043.

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

Proficiency Testing Reporting Limit: A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The proficiency testing reporting limits are specified in the TNI FoPT tables.

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

PT Study Closing Date:

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

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

PT Study Opening Date:

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

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

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

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

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

Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of the 2016 TNI standard, but that does not have a pre-determined opening date and closing date.

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

4.0 Requirements for Accreditation

4.1 General Requirements

4.1.1 The laboratory shall participate in PT studies for each FoA.

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

4.1.2 For methods where the laboratory analyzes a suite of compounds (e.g., EPA methods 8260, 8270, 8081, 8082), and all the FoA compounds are not included in the PT Studies, the requirements for the additional FoA compounds shall be met by the successful analysis of a FoPT study for that method, unless there are separate PT studies specifically for the analytes not included in the PT studies (e.g., 1,4-dioxane).

4.1.3 Aqueous PT is an acceptable substitute for the AFFF matrix.

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

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

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

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

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

Note: Participation in any of the prohibited activities above is cause for revocation of accreditation.

4.1.5 When a regulatory program has additional PT requirements not covered by this module, then the laboratory shall follow those requirements.

4.2 Sample Handling, Preparation, and Analysis Requirements

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

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

4.3 Reporting Requirements

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

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

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

4.3.4 Except for drinking water analytes referenced in 40 CFR Part 141, a laboratory may choose to analyze and report a single method to represent a technology in a single PT study for a particular analyte. If the laboratory analyzes and reports PT studies by "technology," the score obtained for the reported method shall be applied to all methods in that technology for which the laboratory seeks to obtain or maintain accreditation in that matrix. If a laboratory reports PT results for multiple methods using the same analytical technology, an evaluation of "not acceptable" for one method shall be applied to all methods reported with that technology.

4.3.5 If a laboratory chooses to analyze and report a single method to represent a technology, and multiple combinations of preparation/analytical methods are used for analysis of field samples, the laboratory shall follow a documented schedule and rotate the combinations used for analysis of field samples each PT study. Every combination shall be used a minimum of once every three years for each matrix.

4.4 Record Retention

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

4.5 Requirements When TNI FoPT is Available

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

4.5.2 Where corresponding FoPTs exist, the laboratory shall participate in these PT studies for each FoA.

4.5.3 The laboratory shall obtain scheduled PT studies or supplemental studies for the individual FoPT from a PT Provider accredited to Volume 3 of the TNI 2016 Standard by a proficiency testing provider accreditor.

4.5.4 The laboratory shall evaluate the analytical result for each chemistry and radiochemistry FoA to the proficiency testing reporting limit (PTRL) as established by

the TNI FoPT Tables.

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

4.5.6 For chemistry PT results where the concentrations are below the LOQ, the laboratory may re-scale its initial calibration curve to bracket the concentration of the PT sample result.

4.5.7 The laboratory shall report chemistry PT study results to the PTRL as established by the TNI FoPT tables, or if the laboratory LOQ is below the PTRL, the laboratory may report results down to its normal LOQ.

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

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

4.5.9.a If the result is a numeric value above or equal to the PTRL, the laboratory shall report the value. If the PTRL is less than the laboratory's LOQ, the laboratory shall report the result. Qualification of the result is not required.

4.5.9.b If the result is a numeric value below the PTRL, the laboratory shall report the result as one of the following:

4.5.9.b.i < PTRL; or

4.5.9.b.ii the numeric value if the result is between the LOQ and the PTRL; or

4.5.9.b.iii < LOQ if the result is below the LOQ and the PTRL.

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

4.5.9.c.i < PTRL; or

4.5.9.c.ii < LOQ.

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

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

4.6 Requirements When No TNI FoPT is Available for a FoA

When there is no TNI FoPT table available for a FoA, but there is a United States of America (USA) or Canada-based ISO/IEC 17043 accredited PT provider for that FoA,

the laboratory shall procure, analyze, and report the PT sample(s) in accordance with criteria established by the PT provider. The same requirements for PT Study Frequency for Accreditation, Corrective Action, Complaint Resolution, and Reinstatement of Accreditation after Suspension or Revocation shall apply. If a commercial PT is not available at the required frequency, the minimum frequency is annually.

4.7 No Available ISO/IEC 17043 Accredited PT Sample or PT Provider (Chemical and Radiochemical Testing Only)

When PT samples for a chemical or radiochemical testing FoA cannot be obtained from any USA or Canada-based PT Provider that is ISO/IEC 17043 accredited, and the analyte/matrix/method/technology combination is required for a scope of accreditation, the laboratory shall meet proficiency testing requirements by performing one of the three following options:

4.7.1 Using an ISO/IEC 17043 accredited provider from outside the USA or Canada and reporting the PT sample(s) in accordance with criteria established by the PT provider. The same requirements for PT Study Frequency for Accreditation, Corrective Action, Complaint Resolution, and Reinstatement of Accreditation after Suspension or Revocation shall apply. If a commercial PT is not available at the required frequency, the minimum frequency is annually.

4.7.2 Using a non-ISO/IEC 17043 accredited provider and reporting the PT sample(s) in accordance with criteria established by the PT provider. The same requirements for PT Study Frequency for Accreditation, Corrective Action, Complaint Resolution, and Reinstatement of Accreditation after Suspension or Revocation shall apply. If a commercial PT is not available at the required frequency, the minimum frequency is annually. Use of a non-accredited PT provider requires approval of the Primary AB.

4.7.3 Determining precision and bias in accordance with the following requirements:

4.7.3.a The laboratory shall submit in writing to its DoD ELAP AB and/or DOECAP-AP AB, a list of items on its scope of accreditation for which no suitable commercial PT is available. **(DOE-only requirement)** The laboratory shall also submit this information in writing to all impacted DOE Customers.

4.7.3.b The laboratory shall have procedures for performing precision and bias studies in accordance with this section.

4.7.3.c The laboratory shall maintain records of precision and bias demonstrations for each analyte/matrix/method/technology combination on its scope.

4.7.3.d Precision and bias studies shall be performed twice per year, meeting the same time requirements as those for commercial proficiency testing. The laboratory shall evaluate precision and bias in the relevant quality system matrices and shall process the samples through the entire measurement system for each analyte of interest.

4.7.3.e Precision and bias studies shall include results from no fewer than eight samples performed in the time period since the last study. These samples shall be at

concentrations less than or equal to the mid-range and not less than the concentration used to verify the Limit of Detection (LOD). Where standard solutions/low-level spiking solutions are not available to prepare quality controls for analysis and the laboratory uses a comparable compound for quality control (e.g., "cold" Selenium for Se-79), the laboratory shall collate the results of those analyses to meet this requirement. The laboratory's procedure shall describe how many samples will be chosen, how these samples are chosen for inclusion and shall include a description of how the laboratory ensures the selection is not biased toward evaluating only samples that will be rated "Acceptable." Appropriate methods for choosing samples include, but are not limited to, the following:

4.7.3.e.i preparation and analysis of eight or more replicate samples prepared specifically for the semiannual study;

4.7.3.e.ii evaluation of at least the last eight quality control samples analyzed in routine work (e.g., LCS, and LOD or LOQ verification); or

4.7.3.e.iii evaluation of a random selection of eight quality control samples analyzed in routine work over the six months since the last study was performed.

4.7.3.f Results from the analysis of these samples shall be evaluated for recovery and precision.

4.7.3.f.i For each data set, calculate the average percent recovery and the percent relative standard deviation of the data set.

4.7.3.f.ii Compare the results to acceptance criteria.

4.7.3.g Acceptance criteria shall be those provided in the reference method, if available. If the reference method does not provide criteria, the laboratory shall use criteria provided in a similar method or develop its own criteria based on statistical limits.

4.7.3.h If results of the precision and bias study do not meet the acceptance criteria, the laboratory shall implement its nonconforming work procedure.

4.7.3.i The results of each precision and bias study shall be reported to the laboratory's Primary AB and to any customer that requests the results.

5.0 PT Study Frequency Requirements for Accreditation

5.1 Initial Accreditation

5.1.1 Chemical Testing, Radiochemical Testing, Asbestos, and Microbiology

5.1.1.a The laboratory shall achieve a history of two successful (acceptable scores) PT studies out of the most recent three attempts for each FoA for which the laboratory seeks accreditation.

Note: If the laboratory has two consecutive acceptable PT scores, a third study is not needed.

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

5.1.1.c The opening date of the second study shall be at least seven calendar days after the closing date of the first study.

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

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

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

5.2 Continued Accreditation

5.2.1 Chemical Testing, Radiochemical Testing, Asbestos, and Microbiology

5.2.1.a The laboratory shall maintain a history of two successful (acceptable scores) PT studies out of the most recent three attempts for each FoA for which the laboratory holds accreditation. Failure to do so may result in suspension of the affected FoA. The laboratory’s accreditation for a FoA can be revoked for failure of three consecutive PT studies, either by failure to participate in the required PT study or by failure to obtain acceptable results.

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

5.2.1.b.i The closing dates of subsequent PT studies for a particular FoA shall be no more than seven months apart.

5.2.1.b.ii The opening date of PT studies for a particular FoA shall be at least seven calendar days after the closing date of a PT study for the same FoA.

5.2.1.b.iii A laboratory that analyzes and reports PT study results with an opening date of subsequent PT studies for the same FoA that are closer than seven days from the closing date of the previous PT study are invalid for the purposes of compliance with this module and are not counted toward the laboratory’s PT

history of the most recent three attempts.

5.2.2 Whole Effluent Toxicity (WET) Testing

5.2.2.a To maintain accreditation, the laboratory shall participate in one WET PT study per calendar year for each FoA for which the laboratory is accredited.

5.2.2.b This annual requirement may be met by annual participation in the Environmental Protection Agency (EPA) Discharge Monitoring Report-Quality Assurance (DMR-QA) studies for WET, or

5.2.2.c If the laboratory is not participating in an EPA DMR-QA study for WET, the closing dates of subsequent WET testing PT studies shall be no more than 14 months apart.

5.2.3 A laboratory that fails to analyze and report PT studies for a particular FoA for which it seeks to maintain accreditation within the specified frequency for that FoA is charged with a failed PT study.

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

6.0 Requirements for Corrective Action

6.1 A laboratory that fails to successfully analyze a PT study for a particular FoA shall implement its nonconforming work procedure.

6.2 The laboratory shall provide the nonconforming work investigation records to the Primary AB within 30 calendar days of a request from the Primary AB.

6.3 Failure to submit requested records to the AB within 30 calendar days of the request from the Primary AB is due cause for suspension of accreditation for a particular FoA.

6.4 Records for WET corrective actions shall include:

6.4.a a copy of the raw data used for the study; and

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

7.0 Requirements for Complaint Resolution

7.1 The laboratory shall submit questions about PT samples or performance evaluations made by the PT Provider to the PT Provider. If the PT Provider is not able or is unwilling

to resolve the question to the satisfaction of the laboratory, the laboratory shall refer those questions to the PT Provider's Proficiency Testing Provider Accreditor, if available.

7.2 The laboratory shall submit questions about the AB's PT evaluation to its Primary AB.

8.0 Requirements for Reinstatement of Accreditation after Suspension or Revocation

8.1 A laboratory seeking to have its accreditation reinstated for a FoA after suspension shall meet the requirements for continued accreditation.

8.2 A laboratory seeking to have its accreditation reinstated for a FoA after revocation shall meet the requirements for initial accreditation.

8.3 A laboratory seeking to have its accreditation reinstated for a FoA after suspension due to not supplying a requested corrective action report shall meet the requirements for continued accreditation.

Module 2

Quality Systems General Requirements

1.0 Scope

All requirements in ISO/IEC 17025:2017 Section 1 are applicable.

2.0 Normative References

All references in ISO/IEC 17025:2017 Section 2 are applicable.

3.0 Terms and Definitions

Definitions are considered requirements. All references in ISO/IEC 17025:2017 Section 3 are applicable.

Additional definitions are listed below.

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

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

Accreditation (Clarification): In this Standard, accreditation refers to accreditation in accordance with the DoD ELAP and the DOECAP-AP as applicable.

Accreditation Body: Entities recognized in accordance with the DoD ELAP and the DOECAP-AP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies*. The AB shall be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.

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.

Aliquot: A discrete, measured, representative portion of a sample taken for analysis.

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.

Analysis: A combination of sample preparation and instrument determination.

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

Analyte (Clarification): The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.

Analytical Batch: Samples (e.g., whole samples, extracts, digestates or concentrates) and quality control standards which are analyzed together as a group. An analytical batch/sequence can include prepared samples originating from various quality system matrices and can exceed 20 samples. An analytical batch/sequence shall use the same instrument calibration and shall be bracketed by calibration verifications within acceptance criteria.

Analytical Method: A formal process that identifies and may quantify the substance, organism, physical parameter, property, or chemical constituent of interest (target analytes) in a sample.

Analytical Sequence: See Analytical Batch.

Annually: A period not to exceed 13 months.

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

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

Authoritative Source: An authoritative source for reference materials is an organization such as a manufacturer, government organization, or designated repository that can provide reference materials with known or appropriate identity and purity for laboratory use. Use of such materials may be mandated by customer or regulator or may be the best materials available in lieu of availability of Certified Reference Materials from an ISO 17034-accredited provider or Standard Reference Materials from a National Metrology Institute.

Bias: The systematic or persistent distortion of a measurement process which causes errors in one direction; the difference between the mean value of the measurements and the true value of the measurand.

Blank: A sample that has not been exposed to the analyzed sample stream 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.

Blank (Clarification): Blank samples are negative control samples, which typically include field blank samples (e.g., Trip Blank, Equipment (i.e., rinsate) Blank, and Temperature Blank) and laboratory blank samples (e.g., Method Blank, Reagent Blank, Solvent Blank, Instrument

Blank, Calibration Blank, and Storage Blank).

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

a. In calibration of auxiliary equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

b. In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity or prepared by the laboratory using auxiliary equipment that has been calibrated or verified to meet specifications.

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

Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.

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

Certified Reference Material: Reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a National Metrology Institute.

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

Confirmation: Verification of the identity of a component using 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, or alternative detectors.

Confirmation (Clarification): Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.

Consensus Standard: A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.

Continuing Calibration Verification: The verification of the initial calibration. Required before sample analysis and at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Correction: Action taken to eliminate a detected non-conformity.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation to prevent recurrence.

Customer: Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.

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

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

Definitive Data: Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.

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

Demonstration of Capability (Clarification): A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).

Derived Waste: Derived waste is waste generated in the laboratory from samples, reagents, and residue from sample analysis. The derived waste can be non-hazardous, hazardous, and/or radiological (e.g., digestates, extracts, and aliquots).

Detection Limit: The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

Digestion: A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in a sample to a more easily measured form.

Document: Written component of the laboratory management system (e.g., policy, procedure, and instruction).

Environmental Data: Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.

Extracted Internal Standard: Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and preparation batch QC samples before the first step of sample extraction and to standards and instrument blanks before analysis. Used for isotope dilution methods.

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which

the accreditation body offers accreditation.

Field Sample: A sample provided by the customer, prepared and/or analyzed either in its entirety or as a subsample. Examples of field samples include extracts, digestions, field duplicates or replicates not used for laboratory quality control, or dilutions of a field sample, extract, or digestion.

Holding Time: The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as applicable.

Improper Action: Intentional or unintentional deviation from contract-specified or method-specified analytical practices that have not been authorized by the customer.

Incremental Sampling Methodology: Structured composite sampling and processing procedure that is designed to reduce data variability and provide a reasonably unbiased estimate of mean contaminant concentrations in a volume of particulate material (e.g., soil and sediment).

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

Initial Calibration Verification: Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to verify accuracy of the initial calibration.

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

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

Legal Chain of Custody Procedure: Procedure employed to record the possession of samples from the time of sampling through the retention time specified by the customer or program. These procedures are performed at the special request of the customer and include the use of a Chain of Custody Form that records the collection, transport, and receipt of samples by the laboratory.

Limit of Detection: The lowest concentration empirically determined to consistently provide a response meeting identification and minimum signal requirements for a specific analyte-matrix-method combination. This is an estimation of the smallest concentration of an analyte that shall be present in order to be detected with a result at or above the DL with 99% confidence (false negative, Type II, error of 1%). The LOD is the lowest concentration for reliably reporting a non-detect of an analyte.

Limit of Quantitation: The smallest concentration that produces a quantitative result with known and recorded precision and bias. The LOQ shall be set at or above the concentration of the lowest non-zero initial calibration standard and within the calibration range.

Linear Dynamic Range: Concentration range where the instrument provides a linear response.

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

Matrix: The substrate of a test sample.

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

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

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

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

Measurement Uncertainty: The expression of the statistically derived range of values that contain the true value of a measurand within a certain confidence level. The uncertainty generally includes many components which may be evaluated by statistical analysis of measured quantity values obtained under defined measurement conditions or by standard deviations evaluated from assumed probability distributions based on experience or other information. For the purpose of this standard, the laboratory's analytical uncertainty, may be reported as measurement uncertainty.

Method: A generic term for a process (e.g., Reference Method, Non-reference Method, Sampling Method, Analytical Method, and Preparation Method.)

Method Detection Limit: The value determined following the procedure defined in 40 CFR Part 136 Appendix B, Rev. 2, which is used to establish the Detection Limit.

Method Blank: A sample of a matrix similar to the preparation 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.

Mid-Range: The concentration equal to 50% of the highest calibration standard concentration.

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

Multi-level Calibration: Calibration using standards with differing concentrations to determine an instrument response across a calibration range.

Multi-point Calibration: Calibration using standards with the same concentration to determine an averaged instrument response.

National Institute of Standards and Technology: A federal agency of the US Department of Commerce's Technology Administration that is designated as the United States National Metrology Institute.

National Metrology Institute: An organization that maintains its country's national standards and provides traceability to the International System of Units.

Operator Aid: A technical posting (such as poster, operating manual, or notepad) that assists workers in performing laboratory activities. All operator aids shall be controlled documents (i.e., a part of the laboratory management system).

Non-reference method: A method that is not published in *Standard Methods for the Examination of Water and Wastewater*, or *Multi-Agency Radiological Laboratory Analytical Protocols Manual*, or by recognized entities such as USEPA, USDOE, ASTM, or NIOSH.

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

Precision: The degree of variation of 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.

Preparation Batch: Environmental samples provided by the customer that are prepared and analyzed together with the same process and personnel, using the same lots of reagents. A preparation batch is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria. The maximum time between the start of processing of the first and last sample in the preparation batch shall be 24 hours.

Preparation Method: A formal process that prepares a matrix for subsequent analysis.

Preservation: Any conditions under which a sample shall be kept to maintain chemical, physical, and/or biological integrity before analysis.

Procedure: A document that details the instruction for an operation, analysis, or action, with thoroughly prescribed techniques and steps. Procedures are officially approved as the methods for performing certain routine or repetitive tasks.

Qualitative Analysis: Analysis designed to identify the components of a substance or mixture.

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

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

Quality Control Sample: A sample used to assess the performance of all or a portion of a measurement system (e.g., preparation, cleanup, analysis). Examples of quality control samples include blanks, Certified Reference Materials, quality system matrices fortified by spiking, field samples fortified by spiking, or duplicates or replicates intended to demonstrate that a measurement system or activity is in control.

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

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

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

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

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

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

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

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

Non-Aqueous Liquid: Any organic liquid with less than15% settleable solids.

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

Solids: Includes soils, sediments, sludges, and other matrices with greater than15% settleable solids.

Quality System Matrix (Clarification): The matrix definitions in the TNI standard shall be used for purposes of preparation batch and quality control requirements and may be different from a field of accreditation matrix.

Quantitation Range: The range of values (concentrations) in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution, and final volume) lies within the calibration range.

Quantitative Analysis: Analysis designed to determine the amounts or proportions of the components of a substance.

Raw Data: The records generated during sampling and analysis. These records include, but are not limited to, field notes, electronic data, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Record: The output of implementing and following management system procedures (e.g., test data in electronic or handwritten forms, files, and logbooks).

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

Reference Method: A reference method is a published method issued by an organization generally recognized as competent to do so. When the ISO language refers to a “standard method,” that term is equivalent to “reference method.” When a laboratory is required to analyze an analyte by a specified method due to a regulatory requirement, the specified method is recognized as a reference method for that analyte. An analyte/method combination is recognized as a reference method if it can be analyzed by another reference method using the same technology in the same matrix.

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

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

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

SI (International System of Units): The SI is an internationally recognized reference system that supports comparability of vital chemical measurements across a broad range of industries and sectors. Results from a series of measurements can be linked to the SI through an unbroken chain of comparisons, i.e., calibrations. At the apex of such a calibration hierarchy

are accurate mass determinations of calibrants and purity assessments that realize SI units (National Institute of Standards and Technology).

Signal to Noise Ratio: S/N is a measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of noise on the relative error of a measurement increases.

Storage Blank: A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A Storage Blank is used to record contamination attributable to sample storage at the laboratory.

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 quality control purposes.

Target Analytes: Analytes or chemicals of primary concern identified by the customer.

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

Third Party: An entity who may be indirectly involved but is not a principal party to environmental testing (i.e., reference material provider, accrediting body, data validator, etc.).

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

4.0 General Requirements

All requirements in ISO/IEC 17025:2017 Section 4 are applicable.

4.1 Impartiality

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 4.1.

4.1.6 The laboratory shall establish and maintain a documented program to detect and deter improper, inappropriate, or prohibited actions. Laboratory personnel shall refrain from improper, inappropriate, or prohibited actions.

4.1.6.a This program shall be reviewed annually by management. Records of this review shall be maintained. Management shall indicate their commitment to the program by signature.

4.1.6.b The program shall include requirements for the following:

4.1.6.b.i annual training of all laboratory personnel on their obligations under the program;

4.1.6.b.ii signed commitment of all laboratory personnel to their obligations under the program, including to act impartially and to refrain from improper, inappropriate, or prohibited actions;

4.1.6.b.iii periodic, in-depth monitoring for improper, inappropriate, or prohibited actions; and

4.1.6.b.iv investigations into potential or suspected improper, inappropriate, or prohibited actions.

4.1.6.c The requirements for periodic, in-depth monitoring for improper, inappropriate, or prohibited actions shall include a schedule of items to be reviewed. Records shall be maintained to demonstrate compliance with the schedule.

4.1.6.d The requirements for investigation shall include a procedure for reporting of potential or suspected improper, inappropriate, or prohibited actions in the laboratory, and a process whereby laboratory management is informed of the issues.

4.1.6.e Laboratory management shall ensure a receptive environment in which all employees may privately discuss potential issues or report items of concern. Management shall maintain confidentiality to the extent practicable. Laboratory management shall ensure no retaliation, interference, coercion, or intimidation of employees reporting concerns or potential issues.

4.1.6.f Laboratory management shall evaluate any reports of potential or suspected improper, inappropriate, or prohibited actions. Where laboratory management determines the need for further investigation, appropriate personnel with technical and quality assurance (QA) capability shall be assigned to perform the investigation. Findings of improper, inappropriate, or prohibited actions are considered nonconforming work. Records of evaluations and investigations shall be maintained, including any notifications made to customers receiving any affected data.

4.1.6.g The laboratory shall report any occurrences of improper, inappropriate, or prohibited actions to its AB within 15 business days of discovery. Discovery includes identification of such practices by laboratory staff or customer stakeholders. The laboratory shall submit records of associated corrections taken or proposed corrective actions to its AB within 30 business days of discovery.

4.1.6.h Specific examples of improper, inappropriate, or prohibited actions include, but are not limited to:

4.1.6.h.i fabricating, falsifying, or misrepresenting data, including creating data for an analysis that was not performed or for a sample that was not collected (dry lab);

4.1.6.h.ii using external analysts, equipment, and/or laboratories to perform analyses when not allowed by contract, or intentionally concealing such use;

4.1.6.h.iii recording improper date/time, including resetting the internal clock on an

instrument to make it appear that a sample was analyzed within a compliant time frame, or recording a false date/time or changing a date/time to make it appear that holding times were met;

4.1.6.h.iv unwarranted manipulation of samples or analytical conditions, including unjustified dilution of samples, manipulating GC/MS tuning data to produce an ion abundance that appears to meet specific QC acceptance criteria, or changing the instrument conditions for sample analysis from the conditions used for standard analysis;

4.1.6.h.v unwarranted manipulation of software, including forcing calibration or QC data to meet acceptance criteria, removing computer operational codes such as the "M" qualifier, changing LIMS parameters to avoid displaying appropriate qualifiers, inappropriately subtracting background, or improperly manipulating the chromatographic or spectrophotometric baseline;

4.1.6.h.vi turning off, or otherwise disabling, electronic instrument audit/tracking functions;

4.1.6.h.vii misrepresenting or misreporting QC samples, including representing spiked samples as being digested or extracted when a digestion or extraction was not performed;

4.1.6.h.viii substituting previously generated runs for a non-compliant calibration or QC run to make it appear that an acceptable run was performed;

4.1.6.h.ix failing to prepare or analyze Method Blanks and the laboratory control sample (LCS) in the same manner that samples were prepared or analyzed, using dedicated labware, instrumentation, or laboratory space for the preparation and analysis of QC samples, analyzing QC samples (including storage blanks) outside of required analytical time frames ("off clock"), using additional instrument blanks or increased rinse times before and/or after QC sample analysis when not similarly applied to field samples;

4.1.6.h.x tampering with QC samples and results, including over/under spiking and adding surrogates after sample extraction;

4.1.6.h.xi performing multiple calibrations or QC runs (including CCVs, LCSs, spikes, duplicates, and blanks) until one meets acceptance criteria, rather than taking needed corrective action, or not maintaining records for other unacceptable data;

4.1.6.h.xii deleting or failing to record QC data outside acceptance criteria to conceal the fact that calibration or other QC analyses were outside acceptance criteria;

4.1.6.h.xiii improper calibrations;

4.1.6.h.xiv discarding points in the initial calibration to force the calibration to appear

acceptable, or changing the values in a calibration table to make quantitated QC results appear to meet acceptance criteria;

4.1.6.h.xv discarding points from a detection limit study, other than instances of gross failure, to force the calculated detection limit to be higher or lower than the actual value, or performing a detection limit study using different sample volumes or masses than routine samples;

4.1.6.h.xvi using an initial calibration that does not correspond to the actual run sequence to make continuing calibration data look acceptable when in fact it was not;

4.1.6.h.xvii performing improper manual integrations, including peak shaving, peak enhancing, or baseline manipulation to meet QC acceptance criteria or to avoid nonconforming work;

4.1.6.h.xviii concealing a known analytical or sample problem;

4.1.6.h.xix removing failed QC analyses from the record of an analytical sequence;

4.1.6.h.xx purposely excluding known QC failures from the case narrative;

4.1.6.h.xxi observing out-of-compliance equipment conditions, then adjusting the equipment into compliance and recording only the compliant observation (e.g., temperatures of refrigerators in active use);

4.1.6.h.xxii recording the temperature of the surface of a “blue ice” block rather than the temperatures of the sample containers;

4.1.6.h.xxiii concealing a known improper or unethical behavior or action;

4.1.6.h.xxiv failing to report the occurrence of a prohibited action, or known improper or unethical act to the appropriate laboratory or contract representative, or to an appropriate government official; or

4.1.6.h.xxv reporting data from a modified method without customer approval, including, but not limited to, changing the stoichiometry or detection system of a method, reducing the number of extractions, or reducing acid concentrations for digestions.

4.2 Confidentiality

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 4.2.

4.2.5 The laboratory shall establish procedures to protect its customers’ confidential information. These procedures shall address records storage, transmission of results, and define personnel authorized to access the records.

5.0 Structural Requirements

All requirements in ISO/IEC 17025:2017 Section 5 are applicable.

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 5.2.

5.2.1 (DOE-Only Requirement) The laboratory's Technical Manager, however named, shall be laboratory personnel available for technical consultation for laboratory operations for the fields of accreditation which they manage within a time-frame adequate to address needs of the laboratory without negative impacts to results. The Technical Manager duties and responsibilities shall include:

5.2.1.a (DOE-Only Requirement) ensuring the quality of data generated by the laboratory through participation in laboratory management review, review of QA records and quality control (QC) data, review of data packages, and authorizing reports;

5.2.1.b (DOE-Only Requirement) defining the minimum qualifications, experience, and skills necessary for all positions in the laboratory;

5.2.1.c (DOE-Only Requirement) ensuring that all laboratory technical staff have demonstrated capability in the activities for which they are responsible;

5.2.1.d (DOE-Only Requirement) providing for on-going training opportunities for all technical staff and ensuring on-going competence demonstrations;

5.2.1.e (DOE-Only Requirement) ensuring adequate supervision of all personnel employed by the laboratory; and

5.2.1.f (DOE-Only Requirement) appointing a qualified member of staff to temporarily perform these functions in the event of an extended absence greater than 15 calendar days.

5.2.2 (DOE-Only Requirement) The Quality Manager, however named, who, irrespective of other duties and responsibilities, shall have defined responsibility and authority for ensuring that the management system related to quality is implemented and followed at all times. Where staffing is limited, the Quality Manager may also be the Technical Manager. The roles and responsibilities of technical management and the Quality Manager, including their responsibility for ensuring compliance with this standard, shall be defined in the quality manual. Furthermore, the laboratory's Quality Manager and any designees shall:

5.2.2.a (DOE-Only Requirement) have direct access to the highest level of management at which decisions are made on laboratory policy or resources;

5.2.2.b (DOE-Only Requirement) serve as the focal point for QA and QC and be responsible for the oversight and/or review of QC data;

5.2.2.c (DOE-Only Requirement) function independently from laboratory operations for which they have QA oversight;

5.2.2.d (DOE-Only Requirement) evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

5.2.2.e **(DOE-Only Requirement)** arrange for or conduct internal audits annually;

5.2.2.f **(DOE-Only Requirement)** notify laboratory management of deficiencies in the quality system; and monitor corrective actions;

5.2.2.g **(DOE-Only Requirement)** 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; and

5.2.2.h **(DOE-Only Requirement)** review (or oversee the review of) the quality manual at least annually and update it if needed.

6.0 Resource Requirements

All requirements in ISO/IEC 17025:2017 Section 6 are applicable.

6.2 Personnel

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 6.2.

6.2.4.a Records of communications identified in ISO/IEC 17025:2017 Clause 6.2.4 shall be maintained.

6.2.7 Each employee training record shall contain a certification that the employee has read, understands, and is using the latest version of the management system documents relating to his/her job responsibilities.

6.2.8 **(DOE-Only Requirement)** The Technical Manager, however named, shall have educational and experience qualifications developed, recorded, and required by the laboratory management.

6.2.9 **(DOE-Only Requirement)** The Quality Manager, however named, shall have records of training and/or experience in QA and QC procedures and the laboratory's quality system, and have a general knowledge of the analytical methods for which data review is performed.

6.2.10 Personnel dealing with radioactive samples shall have records they are trained in radioactive sample receipt, radioactive waste management, radioactive materials shipping and handling (49 CFR Part 172), and radioactive material control, as applicable to their duties.

6.2.11 Upon employment, laboratory employees shall have initial training in computer security awareness and shall have ongoing refresher training on an annual basis. Records of the training shall be maintained.

6.2.12 Data integrity training shall be provided as a formal part of new employee orientation and shall also be provided on an annual basis for all current employees. The initial data integrity training and the annual refresher training shall have a signature attendance sheet or other records demonstrating all staff have participated and

understand their obligations related to data integrity.

6.2.13 Data integrity training shall encompass requirements for complete records supporting all reported data, including data with QC outliers, and requirements to refrain from improper, inappropriate, and prohibited actions. Employees shall be informed that evidence of participation in improper, inappropriate, or prohibited actions shall result in an investigation. The outcome of such an investigation could have serious consequences for involved personnel including immediate termination, debarment, or civil/criminal prosecution. A record of the topics covered in such training shall be provided to all trainees. At a minimum, the following topics shall be addressed:

6.2.13.a the relationship of laboratory-generated data to public health concerns and the need for known and documented quality;

6.2.13.b review of data integrity procedures;

6.2.13.c how and when to report data integrity issues;

6.2.13.d requirements for keeping analytical records;

6.2.13.e requirements for reporting qualified data;

6.2.13.f prohibited actions; and

6.2.13.g potential consequences of engaging in improper, inappropriate, or prohibited actions.

6.3 Facilities and Environmental Conditions

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 6.3.4.

6.3.4.d Standards and reference materials shall be stored separately from samples, extracts, and digestates.

6.3.4.e **(DOE-Only Requirement)** The laboratory shall have a safety inspection program in place that includes routine inspections of laboratory areas for safety-related concerns.

6.4 Equipment

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 6.4.

6.4.13.i Records retained for equipment shall include instrument configuration and settings.

6.4.14 The following requirements apply specifically to equipment used for auxiliary activities. Such equipment may include balances, weight/mass sets, temperature measuring devices, volumetric measuring and dispensing devices, refrigerators, freezers, incubators, water baths, ovens, water purification systems, timers, radiological survey equipment, etc. Where these items are used as the measurement

system for a method, the item shall meet any method-defined calibration requirements.

6.4.14.a The laboratory shall implement its nonconforming work procedure when a performance check for auxiliary equipment is outside acceptance criteria.

6.4.14.b Calibration and verification shall be performed in the expected use range using calibrated equipment. Where metrological traceability is required, these calibrations shall be traceable to the International System of Units (SI) through an accredited calibration laboratory or a United States National Metrology Institute (NMI) (e.g., National Institute of Standards and Technology (NIST)).

6.4.15 The results of calibration and verification of auxiliary equipment 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. Calibration and verification records, including those of established correction factors, shall be maintained. In the absence of more stringent method-specific requirements, the minimum requirements are as follows (Table 6-1):

Table 6-1 Minimum requirements for calibration and verification of auxiliary equipment.

Performance Check	Minimum Frequency	Acceptance Criteria
Balance verification check Use two standard weights that bracket the measured masses	Daily before use	Top-loading balance: ± 0.2% or ± 0.02 g, whichever is greater. Analytical balance: ± 0.1% or ± 0.5 mg, whichever is greater.
Balance Calibration	Annually	Unexpired and Endorsed Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory or a NMI.
Calibration of standard masses Use weights traceable to the International System of Units (SI) through a NMI	Every 5 years	Unexpired and Endorsed Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory or a NMI.

Performance Check	Minimum Frequency	Acceptance Criteria
Verification of working standard masses (*i.e.*, masses used for daily balance verification) Option 1: comparison to calibrated reference weights not in daily use. Option 2: check on balance immediately (same day) after required balance calibration from accredited calibration provider.	Annually	± 0.1% or ± 0.2 mg, whichever is greater
Monitoring of refrigerator/freezer temperatures Metrological Traceability not required for sample and standard storage	Daily (i.e., 7 days per week) When personnel or an automated system are not available to record daily, use MIN/MAX thermometers or data loggers to monitor. Evaluate the data from devices upon return to the laboratory. The laboratory shall implement its nonconforming work procedure within 24 hours of detecting any excursion noted on MIN/MAX thermometers or data loggers with longer than 2 hours between measurements. For data loggers recording more frequently, action shall be taken for any excursion of > 2 ˚C or any excursion > 2 hours.	Refrigerators: 0 ˚C to 6 ˚C Freezers: ≤ -10 ˚C

Performance Check	Minimum Frequency	Acceptance Criteria
Monitoring of other equipment requiring temperature checks Metrological Traceability is required when it impacts the uncertainty of the result (e.g., methods for flashpoint, ignitability, pH, and conductivity, and when monitoring incubator temperatures)	Daily before use, or as required by reference method, whichever is more frequent	Per reference method, requirements document, or laboratory procedure, whichever is more stringent
Thermometer verification check Use a calibrated reference thermometer (not required for sample and standard storage thermometers) Perform multiple measurements at each of two temperatures that bracket the measured temperatures; if the range of use is ≤ 10 ˚C (e.g., 0 °C to 6 °C), verification may be at a single temperature within the range of use	Liquid in glass and electronic: Before first use and annually Hand-held infrared: Before first use and quarterly	Per reference method, requirements document, or laboratory procedure, whichever is more stringent Apply correction factors or replace thermometer as appropriate
Calibration of Reference Thermometer	Every 5 years	Unexpired and Endorsed Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory or a NMI
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean ± 2% of nominal volume Precision: RSD ≤ 1% of nominal volume (based on 10 replicate measurements)

Performance Check	Minimum Frequency	Acceptance Criteria
Non-volumetric labware, (i.e., labware used for critical volumetric measurements that is not Class A or Class B, for example, volumetrically-marked digestion vessels.) Required only when used for volumetric measurements impacting the uncertainty of the result	By lot before first use and upon evidence of deterioration	Bias: Mean ± 3% of nominal volume Precision: RSD ≤ 3% of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette For variable volume pipettes, verify at the volume of use or using two volumes that bracket the range of use	Daily before use	Bias: Mean ± 2% of nominal volume Precision: RSD ≤ 1% of nominal volume (based on minimum of 3 replicate measurements)
Glass gas-tight syringe	Upon evidence of deterioration	Per reference method, requirements document, or laboratory procedure, whichever is more stringent Discard syringe if deterioration is evident
Drying oven temperature check (applicable only when used as part of an analytical procedure)	Daily before and after use	± 5% of set temperature
Water purification system	Daily before use	Per reference method, requirements document, or laboratory procedure, whichever is more stringent

Performance Check	Minimum Frequency	Acceptance Criteria
Radiological survey equipment The battery is checked and a background reading is taken; and operation verified with a radiological source	Daily before use	Per laboratory procedure
Timer Metrological traceability is required when it impacts the validity of the result.	Annually	Per laboratory procedure, ensure timer is fit for propose.
All other auxiliary equipment Metrological traceability is required when it impacts the validity of the result.	Calibrate or verify at least annually	Per laboratory procedure

Note: The table above does not replace the requirement for the laboratory to maintain traceability per its respective Accreditation Body requirements.

6.5 Metrological Traceability

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 6.5.2.

6.5.2.d The laboratory shall use Certified Reference Materials specifically identified as such in an accompanying Certificate of Analysis from a Reference Material Producer (RMP) accredited to ISO 17034 or Standard Reference Materials from a NMI. If such standards are used, verification of initial calibrations from a second source is not required. If no such material is available from an accredited RMP based in the USA or Canada, then the laboratory shall use standards from an authoritative source and verify all initial calibrations with a standard from an authoritative independent second source.

6.5.2.e When establishing traceability through reference materials, the certified values provided on the certificates are only considered traceable to the SI for reference materials used before the expiration date indicated on the certificate. This stated expiration date also applies to any solutions prepared from the primary reference material. If the manufacturer or vendor provides an extended expiration date on a certificate for a particular lot of the product, the extended date may be used.

6.5.2.f All storage containers of prepared standards, reference materials, and reagents shall bear an expiration date and unique identifier that provides traceability to the preparation record.

6.5.2.g For original containers, if an expiration date is provided by the manufacturer or vendor, it shall be recorded on the container. If an expiration date is not provided by the manufacturer or vendor, it is not required. (TNI 2016 V1M2 5.6.4.2.b)

6.6 Externally Provided Products and Services

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 6.6.2.

6.6.2.e The laboratory shall have procedures describing how services and supplies affecting laboratory activities are selected, purchased, received, and stored.

6.6.2.f Records for services and supplies that may affect the quality of environmental tests shall include the following, as applicable:

6.6.2.f.i date of receipt;

6.6.2.f.ii expiration date;

6.6.2.f.iii purchase source;

6.6.2.f.iv unique identifier (e.g., lot, serial, source, batch numbers);

6.6.2.f.v calibration and verification records;

6.6.2.f.vi accreditation or certification scopes/certificates; and

6.6.2.f.vii **(DOE-Only Requirement)** date opened.

6.6.2.g If the laboratory provides sample containers to its customers, then the laboratory shall maintain records demonstrating that each lot of those containers, along with any included preservatives, are free of likely contaminants exceeding ½ the LOQ for the associated analysis (e.g., vendor certificate or result from laboratory testing).

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 6.6

6.6.4 The laboratory shall only place subcontracted work with a subcontractor who is accredited for that work. The laboratory shall maintain records of the subcontractor's accreditation.

6.6.5 Reported results shall clearly indicate the subcontractor that performed the testing. If not provided with the laboratory's report, the laboratory shall provide a copy of the subcontractor's report to the customer upon request.

6.6.6 Laboratories shall receive approval for use of subcontracted laboratories from the customer before any samples are analyzed and maintain records of approval.

6.6.7 The requirements for subcontracting laboratories also apply to the use of any laboratory under the same corporate umbrella, but at a different facility or location.

6.6.8 All subcontracted or outsourced management system elements (such as data

review, data processing, project management, and IT support), or outsourced personnel shall meet the requirements of accreditation.

7.0 Process Requirements

All requirements in ISO/IEC 17025:2017 Section 7 are applicable.

7.1 Review of Request, Tenders and Contracts

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.1.1.c.

7.1.1.c.i The laboratory shall maintain records of customer approval for use of an external provider to perform any of the following:

7.1.1.c.i.a sampling or subsampling;

7.1.1.c.i.b sample preparation;

7.1.1.c.i.c sample analysis;

7.1.1.c.i.d data reduction;

7.1.1.c.i.e data review; or

7.1.1.c.i.f reporting.

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.1.5.

7.1.5.a Waivers from QSM requirements shall be obtained in writing from the customer-identified technical point of contact on a project-specific basis and shall include project-specific technical justification for the waiver. Records of approval for the waiver shall be maintained by the laboratory and included in all affected data packages.

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 7.1.

7.1.9 Customer clarification or feedback shall be sought immediately for the following situations:

7.1.9.a methods which require modifications to ensure achievement of customer objectives contained in planning documents (e.g., difficult matrix, poor performing analyte);

7.1.9.b project planning documents (e.g., Quality Assurance Project Plan or Sampling and Analysis Plan) are missing or requirements (e.g., action levels, detection and quantification capabilities) in the documents require clarification; or

7.1.9.c the laboratory has encountered problems with sampling that may impact results (e.g., improper preservation of sample).

7.2 Selection, Verification and Validation of Methods

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.2.1.2.

7.2.1.2.a The laboratory shall maintain documents that accurately reflect all current laboratory activities.

7.2.1.2.a.i Collectively, these documents shall provide instruction for implementing management system requirements and test method requirements where the absence of instruction could jeopardize the defensibility of results.

7.2.1.2.a.ii These documents may be from external sources or internally prepared. For example, the documents may be equipment manuals provided by the manufacturer, published reference methods, or internal procedures. External documents that contain sufficient information to perform the activity do not need to be supplemented or rewritten as internal procedures.

7.2.1.2.b The laboratory's test method documents shall include instructions for all accredited methods.

7.2.1.2.b.i In cases where modifications to published reference methods have been made by the laboratory, these modifications shall be clearly identified and described in the method instructions.

7.2.1.2.b.ii In cases where the published reference method provides options, is ambiguous, or provides insufficient detail, the choices and/or clarifications made by the laboratory shall be clearly identified and described in the method instructions.

7.2.1.2.c These instructions shall include or reference the following topics where applicable:

Note: The following topics are not intended to provide any document formatting requirements.

7.2.1.2.c.i These topics shall be included in the instructions:

7.2.1.2.c.i.a identification of the method;

7.2.1.2.c.i.b applicable matrix or matrices;

7.2.1.2.c.i.c scope and application, including analytes to be analyzed;

7.2.1.2.c.i.d summary of the method;

7.2.1.2.c.i.e interferences;

7.2.1.2.c.i.f safety measures for hazards specific to the test method beyond general safety measures covered below;

7.2.1.2.c.i.g equipment and supplies;

7.2.1.2.c.i.h reagents and standards;

7.2.1.2.c.i.i sample collection, preservation, shipment, and storage;

7.2.1.2.c.i.j complete list of quality controls to be analyzed and preparation instructions for those quality controls;

7.2.1.2.c.i.k type of calibration to be analyzed and calibration instructions;

7.2.1.2.c.i.l prescribed techniques and steps;

7.2.1.2.c.i.m data analysis and calculations; and

7.2.1.2.c.i.n references.

7.2.1.2.c.ii These topics shall be addressed but may be included by reference to other documents or records:

7.2.1.2.c.ii.a definitions;

7.2.1.2.c.ii.b limits of detection and quantitation;

7.2.1.2.c.ii.c calibration evaluation and acceptance criteria;

7.2.1.2.c.ii.d data assessment and acceptance criteria for quality controls;

7.2.1.2.c.ii.e actions for handling out-of-control or unacceptable data;

7.2.1.2.c.ii.f general laboratory safety;

7.2.1.2.c.ii.g **(DOE-Only Requirement)** cleaning labware; and

7.2.1.2.c.ii.h **(DOE-Only Requirement)** approaches to address background corrections when quantitation requires adjustments or intended algorithms are overridden, when applicable.

7.2.1.2.d All technical instructions (e.g., sample preparation, analytical procedures, sample storage, or sample receipt) shall be reviewed for accuracy and adequacy at least annually and updated if necessary. All such reviews shall be conducted by personnel having the pertinent background, recorded, and made available for assessment.

Note: Non-technical instructions that are not required elements of the quality manual (e.g., personnel disciplinary policies, timekeeping procedures, or payroll) are considered administrative instructions and do not require an annual review.

7.2.1.2.e **(DOE-Only Requirement)** The laboratory shall track authorized departures from procedures and periodically evaluate if formal procedure revision is appropriate.

7.3 Sampling

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.3.1.

7.3.1.a Subsampling procedures shall address recording the presence of extraneous materials (e.g., rocks, twigs, vegetation) present in samples. To avoid preparing non-representative subsamples, the laboratory shall not "target" within a relatively small mass range (e.g., 10.00 ± 0.01 g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The handling of multiphase samples shall be addressed in procedures, as applicable. The laboratory's subsampling procedures shall comply with recognized consensus standards (e.g., ASTM standards, or EPA's *Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples* (EPA/600/R-03/027)) where applicable.

7.4 Handling of Test or Calibration Items

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.4.1.

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

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.4.3.

7.4.3.a The laboratory shall have a procedure for communicating to all affected laboratory personnel when samples that require non-routine analysis, additional sample preparation steps, or customer-required deviations are received. Records of these communications shall be maintained.

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 7.4.

7.4.5 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 (i.e., a laboratory ID code). This system shall include identification for all samples, sub-samples, preservations, sample containers, tests, and subsequent extracts and/or digestates. (TNI 2016 V1M2 5.8.5.a)

7.4.6 This laboratory ID code shall maintain an unequivocal link with the unique field ID code assigned to each sample. (TNI 2016 V1M2 5.8.5.b)

7.4.7 The laboratory ID code shall be placed as a durable mark on the sample container. (TNI 2016 V1M2 5.8.5.c)

7.4.8 The laboratory ID code shall be entered into the laboratory records and shall be the link that associates the sample with related laboratory activities such as sample preparation. (TNI 2016 V1M2 5.8.5.d)

7.4.9 The laboratory shall have procedures for sample acceptance. These shall include requirements for:

7.4.9.a sample identification, location, date and time of collection, collector's name, sample matrix, and any preservation included;

7.4.9.b proper sample labeling to ensure readability and unique identity;

7.4.9.c proper sample container material and volume;

7.4.9.d calculating holding times;

7.4.9.e sample preservation verification;

7.4.9.f actions needed if samples are damaged, contaminated or improperly preserved;

7.4.9.g how data are qualified for any deviations from requirements; and

7.4.9.h sample rejection.

7.4.10 Sample temperature measurement shall be verified through the use of one or more temperature blanks for each shipping container, if provided. If a temperature blank is not available, other temperature measurement techniques shall be used.

7.4.11 Chemical preservation shall be checked at the time of sample receipt for all samples, unless it is not technically acceptable to check preservation upon receipt (e.g., VOA and Oil and Grease samples). If any of the following conditions exist, chemical preservation shall be rechecked:

7.4.11.a continued preservation of the sample is in question (e.g., the sample may not be compatible with the preservation); or

7.4.11.b deterioration of the preservation is suspected.

7.4.12 If the sample does not meet the sample receipt acceptance criteria, the laboratory shall either retain correspondence and/or records of conversations concerning the final disposition of rejected samples or maintain detailed records of any decision to proceed with the analysis of samples not meeting acceptance criteria. (TNI 2016 V1M2 5.8.7.2)

7.4.13 The condition of samples that do not meet sample receipt acceptance criteria shall be recorded on the chain of custody or transmittal form, and laboratory receipt records, and sample results shall be appropriately qualified on the final report. (TNI 2016 V1M2 5.8.7.2)

7.4.14 The laboratory shall utilize a permanent record such as a logbook or electronic database to record receipt of all sample containers. This sample receipt log shall record the following: (TNI 2016 V1M2 5.8.7.3)

7.4.14.a customer/project name;

7.4.14.b date and time of laboratory receipt;

7.4.14.c unique laboratory ID code; and

7.4.14.d identity of the person making the entries.

7.4.15 During the login process, the following information shall be unequivocally linked to the log record or included as a part of the log. If such information is recorded elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request and available to individuals who will process the sample. The placement of the laboratory ID code on the sample container is not considered a permanent record. (TNI 2016 V1M2 5.8.7.3.b)

7.4.15.a The field ID code, which identifies each sample, shall be linked to the laboratory ID code in the sample receipt log. (TNI 2016 V1M2 5.8.7.3.b.i)

7.4.15.b The date and time of sample collection shall be linked to the sample and to the date and time of receipt in the laboratory. (TNI 2016 V1M2 5.8.7.3.b.ii)

7.4.15.c The requested analyses (including applicable approved method numbers) shall be linked to the laboratory ID code. (TNI 2016 V1M2 5.8.7.3.b.iii)

7.4.15.d Any comments resulting from inspection for sample rejection shall be linked to the laboratory ID code. (TNI 2016 V1M2 5.8.7.3.b.iv)

7.4.16 All records, such as memos, chain of custody, or transmittal forms that are transmitted to the laboratory by the sample transmitter, shall be retained. (TNI 2016 V1M2 5.8.7.4)

7.4.17 A complete chain of custody record, if utilized, shall be maintained. (TNI 2016 V1M2 5.8.7.5)

7.4.18 A legal chain of custody is used for evidentiary or legal purposes. If a customer specifies that a sample will be used for evidentiary purposes, then a laboratory shall have a procedure for how that laboratory will carry out legal chain of custody. (TNI 2016 V1M2 5.8.8)

7.4.18.a When a legal chain of custody is specified, the procedure for legal chain of custody shall be agreed upon by the laboratory and customer before samples are accepted. Records of the agreement shall be maintained.

7.4.18.b Legal chain of custody procedures shall follow any applicable state or federal program and establish an intact, continuous record of the physical possession, storage and disposal of used sample containers, collected samples, sample aliquots, and sample extracts or digestates.

7.4.18.c The legal chain of custody records shall identify all individuals who physically handled individual samples.

7.4.19 Samples shall be stored according to the conditions specified by preservation procedures. (TNI 2016 V1M2 5.8.9.a)

7.4.20 Samples that require thermal preservation shall be stored under refrigeration that is ± 2 °C of the specified preservation temperature unless regulatory or method specific acceptance criteria exist. For samples with a specified storage temperature

of 4 °C, storage at a temperature 0 °C to 6 °C shall be acceptable.

7.4.20.a Samples that are delivered to the laboratory on the same day they are collected shall be considered acceptable if the samples were received on ice or with evidence the cooling process has begun.

7.4.20.b If sample analysis is begun within 15 minutes of collection, thermal preservation is not required.

7.4.20.c If the laboratory receives and refrigerates the sample within 15 minutes of collection, thermal preservation is not required in transit unless required by method or regulation.

7.4.21 Samples shall be stored away from all standards, reagents, and food. Samples shall be stored in such a manner to prevent cross contamination. (TNI 2016 V1M2 5.8.9.a.ii)

7.4.22 Sample fractions, extracts, leachates, and other sample preparation products shall be stored according to above or according to specifications in the method. (TNI 2016 V1M2 5.8.9.b)

7.4.23 The laboratory shall have procedures for the disposal of samples, digestates, leachates and extracts or other sample preparation products. (TNI 2016 V1M2 5.8.9.c)

7.4.23.a Disposal of the physical sample shall occur only with the concurrence of the customer who submitted the sample if those samples are disposed of before any project-specified time limit. Samples that are completely consumed during analysis shall be recorded as such for their final disposition.

7.4.23.b All conditions of disposal and all records and correspondence concerning the final disposition of the physical sample shall be recorded and retained.

7.4.23.c **(DOE-Only Requirement)** Records shall indicate the date of disposal, the nature of disposal (such as sample depleted, sample disposed in hazardous waste facility, or sample returned to customer), and the name of the individual who performed the task.

7.5 Technical Records

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 7.5.

7.5.2.a The laboratory shall include the reason for the alteration in the amended technical record.

7.5.2.b Requirements for amendments to technical records also apply to changes to the original output of the automated software algorithms such as manual integrations and eliminating laboratory determined “false positives” (e.g., “Q delete”). These changes to the original output of the automated software algorithms shall be reviewed by a technically qualified supervisor or data review specialist. Records of this review shall be maintained.

7.5.3 The records considered critical to be maintained for historical reconstruction of laboratory activities include but are not limited to:

7.5.3.a all raw and processed data for calibrations, sample analyses and QC measures, including analysts' worksheets and instrument output;

7.5.3.b all sample receiving records;

7.5.3.c dates and times of sample collection if available, preparation, and analysis;

7.5.3.c.i For the purpose of preparation batch processing, the start and stop dates and times of the preparation batch preparation shall be recorded. The start time of sample preparation is the time when analytes from the first sample in a batch of samples begin to be removed from the matrix (e.g., extraction, digestion, distillation, etc.). The stop time of sample preparation is the time when the last sample extract or digestate is ready for additional clean up or analysis.

7.5.3.d all sample preparation records;

7.5.3.e dates of sample receipt by the laboratory;

7.5.3.f dates of sample result reporting;

7.5.3.g all instrumentation identification and operating conditions/parameters used for analysis;

7.5.3.h all information necessary to reconstruct calculations;

7.5.3.i final test reports;

7.5.3.j all standard and reagent origin, including associated Certificates of Analysis or purity and recommended storage conditions;

7.5.3.k all standard and reagent receipt, preparation, and use records, which shall include:

7.5.3.k.i lot numbers allowing traceability to purchased stocks or neat compounds;

7.5.3.k.ii constituents and quantities;

7.5.3.k.iii dates of preparation; and

7.5.3.k.iv expiration dates.

7.5.3.l all QC results and assessment;

7.5.3.m method performance/QC acceptance limits with supporting data if generated by the laboratory;

7.5.3.n proficiency test results;

7.5.3.o records of demonstrations of capability for each method, instrument, and analyst;

7.5.3.p names, initials, and signatures of all individuals who sign or initial laboratory records;

7.5.3.q obsolete versions of all procedures used to support laboratory activities; and

7.5.3.r all other records required under this standard.

7.5.4 All records shall be maintained for five years from last use. A record is considered in use when it supports current laboratory activities.

Note: For example, method validation records are considered to support current laboratory activities as long as the method is used for accredited testing; analyst training records are considered to support current laboratory activities as long as the analyst is employed at the laboratory; and traceability records are considered to support current laboratory activities as long as any materials or equipment requiring traceability are in use in the laboratory.

7.5.5 When laboratory notebooks (logbooks) are utilized, the notebooks shall have measures to prevent the removal or addition of pages. Electronic logbooks are acceptable. For laboratory notebooks, the following applies:

7.5.5.a Laboratory notebook pages shall be pre-numbered, all entries shall be signed or initialed and dated by the person responsible for performing the activity at the time the activity is performed, and all entries shall be recorded in chronological order.

7.5.5.b All laboratory notebook pages shall be closed when the activities recorded are completed or carried over to another page. The person responsible for performing the closure shall be the one who performed the last activity recorded. Closure shall occur at the end of the last activity recorded on a page, as soon as practicable, thereafter. Satisfactory records of closure include analyst initials and date.

7.5.5.c Each laboratory notebook shall have a unique serial number clearly displayed.

7.5.6 Records that are stored only on electronic media shall be supported by the hardware and software necessary for their retrieval. (TNI 2016 V1M2 4.13.3.d)

7.5.7 All generated data, except those that are generated electronically, shall be recorded legibly in permanent ink. (TNI 2016 V1M2 4.13.3.g)

7.5.8 The laboratory shall have a plan to ensure that records are maintained or transferred according to the customers' instructions in the event that a laboratory transfers ownership or ceases operations. In addition, appropriate regulatory and state legal requirements concerning laboratory records shall be followed. (TNI 2016 V1M2 4.13.3.h)

7.6 Evaluation of Measurement Uncertainty

All requirements in ISO/IEC 17025:2017 Section 7.6 are applicable.

7.7 Ensuring the Validity of Results

The following is a note to ISO/IEC 17025:2017 Clause 7.7.1.

Note: Items listed in 7.7.1 not specifically required by reference method, Modules 3-8, or Appendix B are not considered applicable for the purposes of the QSM.

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 7.7.

7.7.4 The laboratory shall have detailed procedures in place to monitor the following quality controls:

7.7.4.a positive and negative controls (e.g., as described in Modules 3-8); (TNI 2016 V1M2 5.9.3.a.i)

7.7.4.b tests to define the variability and/or repeatability of the laboratory results such as replicates; (TNI 2016 V1M2 5.9.3.a.ii)

7.7.4.c measures to assure the accuracy of the method including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures; (TNI 2016 V1M2 5.9.3.a.iii)

7.7.4.d measures to evaluate method capability, such as limit of detection and limit of quantitation or range of applicability such as linearity; (TNI 2016 V1M2 5.9.3.a.iv)

7.7.4.e selection of appropriate formulas to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses; (TNI 2016 V1M2 5.9.3.a.v)

7.7.4.f selection and use of reagents and standards of appropriate quality; (TNI 2016 V1M2 5.9.3.a.vi)

7.7.4.g measures to assure the selectivity of the test for its intended purpose; and (TNI 2016 V1M2 5.9.3.a.vii)

7.7.4.h measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the method such as temperature, humidity, light, or specific instrument conditions. (TNI 2016 V1M2 5.9.3.a.viii)

7.7.5 The laboratory shall have procedures for the development of QC acceptance criteria where no method or regulatory acceptance criteria exist. The QC acceptance criteria specified by the laboratory's procedures shall be followed. The laboratory shall incorporate the acceptance criteria outlined in Modules 3-8, Appendix B, reference method, or regulation, whichever are more stringent, into the test method procedures. When it is not apparent which is more stringent, the QC in the reference method or regulation shall be followed. (TNI 2016 V1M2 5.9.3.c)

7.7.6 Quality control samples shall be processed in the same manner as field samples and shall be analyzed and reported with their associated field samples.

7.7.7 The laboratory shall have procedures to ensure validity of reported results which

shall include the following:

7.7.7.a requirements that internal data reviews consist of a tiered or sequential system of verification with at least three tiers: 100% review by the analyst, 100% verification review by a technically qualified supervisor or data review specialist, and a final administrative review;

7.7.7.b specification of which records shall be reviewed;

7.7.7.c determination of whether the results meet the laboratory-specific QC acceptance criteria before results are reported;

7.7.7.d checks to determine consistency with customer-provided acceptance criteria, if available;

7.7.7.e checks to ensure that reported data are free from transcription errors;

7.7.7.f checks to ensure that the appropriate sample preparatory and analytical procedures and methods were followed, calculations were performed correctly, and that chain of custody and holding time requirements were met; and

7.7.7.g checks for complete and accurate explanations of anomalous results, corrections, and the use of data qualifiers in the case narrative.

7.7.8 Records of these activities shall be maintained.

7.7.9 For a test method with a maximum holding time measured in hours, the holding time shall be tracked by the hour. For a test method with a maximum holding time measured in days, the holding time shall be tracked by the day. For a test method with a maximum holding time measured in months, the holding time shall be converted to days and tracked by day. One month is defined as 30 days.

Note: For example, an exceedance of holding time for a sample with a 48-hour holding time will occur when the 49th hour is reached (e.g., a sample with a 48-hour holding time collected at 830 AM on April 4th must be analyzed or extracted by 9 AM on April 6th, or an exceedance will be considered to have occurred). An exceedance of holding time for a sample with a 14-day holding time will occur when the 15th day is reached (e.g., a sample with a 14-day holding time collected at 840 AM on April 4th must be analyzed or extracted by 12 AM on April 19th, or an exceedance will be considered to have occurred). An exceedance of holding time for a sample with a 6-month holding time will occur when 6 months have passed (e.g., a sample with a 6-month holding time collected at 830 AM on April 5th must be analyzed or extracted by 12 AM on October 2nd, or an exceedance will be considered to have occurred).

7.7.10 If time of sample collection is not provided by the customer, the laboratory shall contact the customer to obtain the time or use the most conservative time (i.e., 12:00 a.m. on the day of collection) for the purposes of calculating holding time.

7.8 Reporting of Results

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.8.2.1.

Reports shall include the following requirements unless specifically waived by the customer:

7.8.2.1.q All required information listed in ISO/IEC 17025:2017 Clause 7.8.2.1 a-p;

7.8.2.1.r an index or a table of contents;

7.8.2.1.s a table summarizing samples received, providing a correlation between field sample identification and laboratory sample identification;

7.8.2.1.t a case narrative that includes the following information, if applicable;

7.8.2.1.t.1 additions to, deviations, or exclusions from the method;

7.8.2.1.t.2 information on any non-standard conditions that may have affected the quality of the results;

7.8.2.1.t.3 Identification of sample results reported under a non-accredited method;

7.8.2.1.t.4 a description of extractions or analyses that were performed outside of holding times;

7.8.2.1.t.5 identification of deviations of any calibration standards or QC sample results from acceptance limits and a discussion of the associated actions taken by the laboratory to address the deviations;

7.8.2.1.t.6 a list of preparation batches for which no matrix spike and/or duplicate were performed due to lack of adequate sample material; and

7.8.2.1.t.7 occurrence of analytes for which manual integration occurred.

7.8.2.1.u LOQ and associated precision and bias at the LOQ, where the determination of precision & bias at the LOQ is required;

7.8.2.1.v before and after chromatographs of analytes for which manual integration occurred including the justification for the change;

7.8.2.1.w the LOD and LOQ verification data when the infrequent method option described in Module 4 is used;

7.8.2.1.x records of customer approval and technical justification for any waiver from QSM requirements shall be included in all affected reports;

7.8.2.1.y all QC required by the method and specified in the applicable Appendix B Table, including acceptance criteria used by the laboratory;

7.8.2.1.z chain of custody records;

7.8.2.1.aa records generated by the laboratory which detail the condition of the samples upon receipt at the laboratory (e.g., sample cooler receipt forms,

cooler temperature, and sample pH);

7.8.2.1.bb records of communication with the customer associated with actions taken or quality issues;

7.8.2.1.cc records of sample compositing done by the laboratory;

7.8.2.1.dd sample preparation records, including start time and date of the first and last sample;

7.8.2.1.ee standard traceability;

7.8.2.1.ff instrument output (raw data);

7.8.2.1.gg instrument run logs or sequence run logs; and

7.8.2.1.hh a sample result summary form for each field sample reported by the laboratory that includes the following information:

7.8.2.1.hh.1 field sample identification as written on custody form;

7.8.2.1.hh.2 laboratory sample identification;

7.8.2.1.hh.3 preparation batch unique identifier;

7.8.2.1.hh.4 matrix;

7.8.2.1.hh.5 date and time sample collected if the laboratory performs sampling or if provided by the customer;

7.8.2.1.hh.6 date and time sample prepared;

7.8.2.1.hh.7 date and time sample analyzed;

7.8.2.1.hh.8 data file name;

7.8.2.1.hh.9 method identification for all preparation, cleanup, and analytical methods including the version number;

7.8.2.1.hh.10 unique instrument identification;

7.8.2.1.hh.11 DL, LOD, and LOQ; if determined and applicable, adjusted for sample-specific factors which impact calculation of sample results;

7.8.2.1.hh.12 sample-specific factors (e.g., sample aliquot or weight of soil/sediment, final extraction volume, dilution factor, and percent moisture or percent solids);

7.8.2.1.hh.13 identification when reported results are converted from the as-sampled basis (e.g., dry weight);

7.8.2.1.hh.14 surrogate recovery with control limits;

7.8.2.1.hh.15 concentration units;

7.8.2.1.hh.16 the result for each target analyte from the lowest dilution that met all QC acceptance criteria; and

7.8.2.1.hh.17 analyte or parameter with the Chemical Abstracts Service (CAS) Registry Number, if available, for all requested analytes including non-detects.

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.8.3.1.

7.8.3.1.f When analytical nonconformances occur, and samples cannot be reanalyzed, the laboratory shall qualify associated sample results in the report. The laboratory shall unambiguously define the data qualifiers used within the report, and their use shall be consistent with any project-specific requirements (e.g., the contract, and project-planning documents). Within the report, data qualifiers shall be located immediately adjacent to the analyte results to which they apply.

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 7.8.7.

7.8.7.4 Opinions and interpretations expressed in reports shall be contained in the case narrative.

7.9 Complaints

All requirements in ISO/IEC 17025:2017 Section 7.9 are applicable.

7.10 Nonconforming Work

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 7.10.

7.10.4 The laboratory shall upon discovery of potential data quality issues resulting from nonconforming work, notify all affected customers within 15 business days. For data reported to affected customers more than 90 days prior to the discovery of the potential data quality issue, the AB shall also be notified. Notification shall be performed according to a procedure. Records of corrections taken or proposed corrective actions to resolve the nonconformance shall be submitted to the customer within 30 business days of discovery.

7.11 Control of Data and Information Management

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 7.11.

7.11.7 The laboratory shall have procedures that address all requirements of ISO/IEC 17025:2017 7.11.2

7.11.8 The laboratory shall maintain records of LIMS validation that include:

7.11.8.a LIMS description, version, and functional requirements;

7.11.8.b listing of algorithms and formulas; and

7.11.8.c installation, operation, and maintenance records.

7.11.9 The laboratory shall maintain records of LIMS versions, procedures, and changes so analytical data can be unequivocally associated with the LIMS version used to generate the data.

7.11.10 The laboratory shall have a procedure to ensure all LIMS users have unique login authentication credentials. The mechanism employed may be a unique username and password combination, or biometric authentication. Where passwords are used, the passwords shall be changed a minimum of once per year.

7.11.11 Spreadsheets used for calculations shall be verified before initial use and after any changes to equations or formulas, or software revision upgrades. Records of verification shall be maintained. Formula cells shall be write-protected to minimize inadvertent changes to the formulas. Printouts from any spreadsheets shall include all information used to calculate the data.

7.11.12 Electronic Data Security measures shall ensure:

7.11.12.a system events, such as log-on failures or break-in attempts, are monitored and recorded;

7.11.12.b the electronic data management system is protected from the introduction of computer viruses;

7.11.12.c system backups occur on a regular and published schedule and may be performed by more than one person within the laboratory;

7.11.12.d system backups are recorded and demonstrate that the backup systems contain all required data; and

7.11.12.e 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.

7.11.13 The laboratory shall have procedures that address how manual integrations are performed and how records are maintained.

8.0 Management System Requirements

All requirements in ISO/IEC 17025:2017 Section 8 are applicable, except 8.1.3.

8.1 Options

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 8.1.

8.1.4 A laboratory may only gain and maintain DoD ELAP and/or DOECAP-AP accreditation using Option A.

8.2 Management System Documentation

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 8.2.1.

8.2.1.a The laboratory shall have a quality manual and the quality manual shall address or make reference to:

8.2.1.a.i procedures and policies that support the management system;

8.2.1.a.ii test methods, however named, under which the laboratory performs testing;

8.2.1.a.iii impartiality requirements;

8.2.1.a.iv confidentiality requirements;

8.2.1.a.v organizational structural requirements, including its place in any parent organization, and relevant organizational charts;

8.2.1.a.vi personnel requirements;

8.2.1.a.vii facility and environmental condition requirements;

8.2.1.a.viii equipment requirements;

8.2.1.a.ix metrological traceability requirements;

8.2.1.a.x requirements for externally provided products and services;

8.2.1.a.xi requirements for review of requests, tenders, and contracts;

8.2.1.a.xii requirements for the selection and verification of methods;

8.2.1.a.xiii requirements for the validation of methods;

8.2.1.a.xiv sampling and subsampling requirements;

8.2.1.a.xv requirements for the handling of test or calibration items;

8.2.1.a.xvi requirements for technical records;

8.2.1.a.xvii evaluation of measurement uncertainty requirements;

8.2.1.a.xviii requirements for ensuring the validity of results;

8.2.1.a.xix reporting requirements;

8.2.1.a.xx requirements for handling complaints;

8.2.1.a.xxi nonconforming work requirements;

8.2.1.a.xxii control of data and information management requirements;

8.2.1.a.xxiii management system documentation requirements;

8.2.1.a.xxiv requirements for the control of management system documents;

8.2.1.a.xxv requirements for the control of records;

8.2.1.a.xxvi requirements for actions to address risks and opportunities;

8.2.1.a.xxvii requirements for improvement;

8.2.1.a.xxviii requirements for corrective actions;

8.2.1.a.xxix requirements for internal audits;

8.2.1.a.xxx requirements for management reviews;

8.2.1.a.xxxi procedures for permitting deviations from management system requirements or standard specifications, including which personnel may approve the deviation;

8.2.1.a.xxxii **(DOE-Only Requirement)** materials (waste) management; and

8.2.1.a.xxxiii **(DOE-Only Requirement)** health and safety (e.g., Chemical Hygiene Plan, Radiation Safety Plan).

8.2.1.b The quality manual shall contain a table of contents or equivalent guide to navigate the document.

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 8.2.3.

8.2.3.a **(DOE-Only Requirement)** The quality manual shall contain the signed and dated concurrence (with appropriate names and titles) of all responsible parties, including the Quality Manager, Technical Manager, and the agent in charge of all laboratory activities, such as the Laboratory Director or Laboratory Manager. (TNI 2016 V1M2 4.2.8.3.f)

8.3 Control of Management System Documents

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 8.3.1.

8.3.1.a The laboratory shall have procedures for how all internally generated and externally sourced documents relating to the laboratory's management system are controlled.

The following shall be implemented in addition to ISO/IEC 17025:2017 Clause 8.3.2.

The laboratory shall ensure that:

8.3.2.g affected personnel are notified of changes to management systems documents and supporting procedures, including technical documents;

8.3.2.h internal or external reviews of management system documentation are maintained and made available for assessment;

8.3.2.i any documents providing instructions to laboratory personnel (e.g., operator aids) are considered part of the management system and are subject to document control procedures;

8.3.2.j documents contain date of issue, pagination, and the identification of the authorized approver; and

8.3.2.k all document versions are retained, suitably marked, and archived for a minimum of five years after retirement or revision of the procedure, or longer if required by regulation or customer contract agreements.

8.4 Control of Records

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 8.4.

8.4.3 The laboratory shall have procedures for creation, maintenance, storage, and disposal of quality and technical records.

8.4.4 The records control system procedures shall address the requirements for access to and control of the files including accountability for any records removed from storage.

8.4.5 (DOE-Only Requirement) The records disposal procedures shall address the requirements for obtaining written approval from all affected customers before disposal of records relevant to testing performed for them.

8.5 Actions to Address Risks and Opportunities

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 8.5.

8.5.4 The laboratory shall consider and plan mitigation for the risks and opportunities associated with the following laboratory activities:

8.5.4.a determining any effects the matrix may have when validating modifications to the analytical portion of a method;

8.5.4.b determining the need for metrological traceability and the impact to data validity and uncertainty;

8.5.4.c use of externally provided products and services;

8.5.4.d identifying likely contaminants which may be encountered in the laboratory facilities or supplies;

8.5.4.e determining minimum qualifications for technical, supervisory, and quality management personnel;

8.5.4.f ensuring alternate personnel are designated, trained, and authorized for key roles and responsibilities;

8.5.4.g determining which methods LOD and LOQ verifications will be performed quarterly, and which will be performed with each batch analyzed;

8.5.4.h use of electronic signatures;

8.5.4.i determining which versions of methods best fit the needs of the laboratory's customers;

8.5.4.j determining frequency and content of periodic in-depth monitoring for improper, inappropriate, or prohibited actions;

8.5.4.k determining frequency and content of periodic quality record reviews to ensure data integrity;

8.5.4.l determining the need for procedures when not specifically required for accreditation;

8.5.4.m determining acceptance criteria for auxiliary equipment verification and calibration, where acceptance criteria are not specified;

8.5.4.n application of correction factors, including how differing correction factors resulting from verification across a range of values will be applied to auxiliary equipment; and

8.5.4.o selecting a subset of analytes for validation of method modifications.

8.5.5 Records of the identification and mitigation of risk shall be maintained.

8.5.6 When a risk is identified, the laboratory shall act in a timely fashion to address the risk.

8.5.7 Identified risks and any mitigation plans shall be reviewed annually and updated as applicable.

8.5.8 Records of the annual review of risks and mitigation plans shall be maintained.

8.6 Improvement

All requirements in ISO/IEC 17025:2017 Section 8.6 are applicable.

8.7 Corrective Action

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 8.7.

8.7.4 The laboratory shall have procedures for performing corrective actions when nonconforming work or departures from management system or technical operation procedures have been identified. These procedures shall:

8.7.4.a address the requirements in ISO/IEC 17025:2017 Clauses 8.7.1.a – f;

8.7.4.b identify individuals or positions responsible for each of the requirements;

8.7.4.c define the records to be maintained; and

8.7.4.d include a system for tracking corrective actions to completion.

8.8 Internal Audits

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 8.8.

8.8.3 The laboratory shall have procedures that ensure any activity that has the potential to affect the validity of results or is required for compliance to this standard is audited, including technical and quality management systems.

8.8.4 The internal audit schedule shall ensure that all areas of the laboratory are reviewed over the course of one year, with no area exceeding a period of 18 months between audit events.

8.8.5 Internal audits shall be conducted by personnel independent of the activity being 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.

8.8.6 When an internal audit casts doubt on the validity of results, the laboratory shall notify affected customers within 15 business days of discovery.

8.8.7 **(DOE-Only Requirement)** The laboratory QA program shall identify the required internal distribution of internal audit reports and all related records.

8.9 Management Reviews

The following shall be implemented in addition to ISO/IEC 17025:2017 Section 8.9.

8.9.4 The laboratory shall have procedures that address the requirements in ISO/IEC 17025:2017 Clauses 8.9.1 – 8.9.3.

8.9.5 Management reviews shall be completed on an annual basis. (TNI 2016 V1M2 4.15.3).

8.9.6 **(DOE-Only Requirement)** Management reviews shall also include laboratory radiation health and safety, radioactive hazardous waste, and radioactive materials management functions, where applicable (i.e., when radioactive samples are analyzed).

8.9.7 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. (TNI 2016 V1M2 4.15.2)

8.9.8 (DOE-Only Requirement) The laboratory QA program shall identify the required internal distribution of management review reports and all related documentation.

9.0 (DOE-Only Requirement) Hazardous and Radioactive Materials Management and Health and Safety Practices

DOE is committed to ensuring that environmental laboratories handling derived waste resulting from samples and analysis conduct 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. Therefore, DOE chooses to use only those analytical laboratories that can demonstrate effective management controls and reasonably diligent health and safety practices.

DOE sites submitting samples to laboratories shall disclose known or suspected hazards associated with the samples. Sample information is based on a good faith effort, available process knowledge, or other hazard information (radiological, toxicity, or biological) and shall be provided to the receiving laboratories before shipment of the samples unless prior arrangements have been made regarding sample receipt.

The laboratory shall determine its ability to safely receive and process the samples. The laboratory shall have the appropriate capabilities, procedures, and licenses to receive samples from a DOE site. Upon sample receipt, the laboratory shall assume the responsibility and liability for the safe and compliant management, storage, and disposal of the samples, and any associated analysis-derived wastes. Some DOE sites permit returning sample residues by pre-arrangement.

The laboratory shall comply with all applicable federal, state, and local regulations governing laboratory operations by developing, training, and implementing procedures. Plans shall be developed as they apply specifically to the laboratory's facility, staff, and DOE contractual requirements, deliverables, and obligations. The laboratory shall be assessed to these plans and their implementation on site.

Failure to include in these plans/procedures specific federal, state, licensure, or permit requirements shall be identified as a finding requiring implementation of the nonconforming work procedure. The following sections discuss requirements for plans and procedures to support and achieve DOE's objectives:

9.1 Radioactive Materials Management Plan

9.2 Waste Management Plan

9.3 Chemical Hygiene Plan

9.4 Sample Receiving and Control

9.5 Records

9.6 Training

9.1 (DOE-Only Requirement) Radioactive Materials Management Plan

This plan shall include, but not be limited to the following subject requirements detailed in the sections listed below:

9.1.1 Radioactive Materials Management

9.1.2 Radioactive Materials License (RML) Requirements

9.1.3 Identification of Radiation Safety Personnel

9.1.4 Radiation Safety Training

9.1.5 Radiation Survey Plan and Equipment

9.1.6 Radioactive Material Receipt and Control

9.1.1 (DOE-Only Requirement) Radioactive Materials Management

9.1.1.a (DOE-Only Requirement) For laboratories accepting, receiving, or handling radioactive samples, or potential radioactive samples, the laboratory shall develop and implement a radioactive materials management plan or radiation safety plan. This plan, however named, shall comply with, identify, and address all applicable site-specific related federal and state regulations governing radioactive materials control and radiological protection.

9.1.1.b (DOE-Only Requirement) The laboratory shall review, at least annually, the radiation protection program content and implementation.

9.1.1.c (DOE-Only Requirement) The laboratory shall develop and implement an effective radiological controls program and procedures for radioactive material handling, emergency actions, and use of instrumentation.

9.1.1.d (DOE-Only Requirement) Airborne releases of radioactivity to the environment shall be monitored, evaluated, and controlled.

9.1.2 (DOE-Only Requirement) Radioactive Materials License (RML) Requirements

9.1.2.a (DOE-Only Requirement) The laboratory shall describe how they address, implement, and manage the requirements of its site-specific radioactive materials license. The 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.

9.1.3 (DOE-Only Requirement) Identification of Radiation Safety Personnel

9.1.3.a (DOE-Only Requirement) 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 with the necessary training and experience to perform the duties of the RSO if the RSO is not available. A procedure shall document how and when an alternate RSO will be necessary and available.

9.1.3.b (DOE-Only Requirement) Initial and refresher training required of the RSO and the alternate RSO shall be identified and completed on an established frequency no less than once every 3 years.

9.1.4 (DOE-Only Requirement) Radiation Safety Training

9.1.4.a (DOE-Only Requirement) Training may consist of General Employee Orientation, Radiation Safety Training, Contractor Training and/or special briefings as established for the exposure potential as determined by the RSO.

9.1.4.b (DOE-Only Requirement) All individuals entering any portion of a restricted area shall be instructed in the potential health effects of exposure to radioactive materials or radiation, precautions/procedures to minimize exposure, and the purpose and functions of protective devices employed.

9.1.5 (DOE-Only Requirement) Radiation Survey Plan and Equipment

9.1.5.a (DOE-Only Requirement) A survey and monitoring program shall be developed and implemented to assess the magnitude and extent of radiation levels, concentrations or quantities of radioactive material, and the extent of potential radiological hazards.

9.1.5.b (DOE-Only Requirement) Radiological survey equipment shall be calibrated according to the manufacturer's recommendation or more frequent procedures as documented by the laboratory. Before use, an operational performance check shall be conducted on radiological survey equipment, including a battery check and measurements of a radiological source and the nominal background. All performance checks shall be recorded.

9.1.6 (DOE-Only Requirement) Radioactive Material Receipt and Control

The laboratory shall ensure:

9.1.6.a (DOE-Only Requirement) active use of a radioactive materials inventory program capable of tracking standards, tracers, and all radiological samples and radioactive waste. The radioactive material inventory shall be updated according to the schedule established by the laboratory's Radioactive Material License. If no schedule is established by the license, then the laboratory shall update the inventory within seven days of receipt of radioactive materials;

9.1.6.b (DOE-Only Requirement) low-level and high-level samples shall be identified, segregated, and processed in order to prevent sample cross-contamination;

9.1.6.c (DOE-Only Requirement) at sample receiving, samples from potentially radioactive sites shall be screened to ensure that;

9.1.6.c.i customer identification of radioactivity (or lack of radioactivity) is correct;

9.1.6.c.ii the sample is properly categorized (per the laboratory's definition of radioactivity) for sample handling in the laboratory;

9.1.6.c.iii data input is obtained for the radioactive materials license tracking system in the absence of customer-supplied information;

9.1.6.c.iv the shipping container does not exhibit loose contamination or unacceptable external radiation readings; and

9.1.6.c.v that licensed material is secure from unauthorized access or removal.

9.2 (DOE-Only Requirement) Waste Management Plan

This plan shall include, but not be limited to the following subject requirements detailed in the sections listed below:

9.2.1 Waste Management Plan Requirements

9.2.2 Waste Disposal

9.2.3 Waste Storage Areas

9.2.4 Toxic Substances Control Act (TSCA) Material

9.2.1 **(DOE-Only Requirement)** Waste Management Plan Requirements

The laboratory shall develop and implement a waste management plan identifying how it complies with all federal, state, and local regulations governing waste management and disposal. The plan shall:

9.2.1.a **(DOE-Only Requirement)** identify all waste streams generated by the laboratory, including universal wastes such as batteries, thermostats, etc.;

9.2.1.b **(DOE-Only Requirement)** identify the process for management and disposal of the various waste streams;

9.2.1.c **(DOE-Only Requirement)** track the disposition of waste samples by Sample Delivery Group;

9.2.1.d **(DOE-Only Requirement)** demonstrate compliance through administrative programs to manage effluent discharges as required by regulatory agencies and applicable DOE Orders;

9.2.1.e **(DOE-Only Requirement)** provide training procedures, required frequency, and management of training records in waste management, shipping, waste handling, and radioactive materials control;

9.2.1.f **(DOE-Only Requirement)** communicate radioactive volumetric and surface release policies;

9.2.1.g **(DOE-Only Requirement)** detail permits and licenses to handle hazardous and radioactive waste;

9.2.1.h **(DOE-Only Requirement)** give policy or direction on how to conduct waste brokering and treatment, storage, and disposal facility (TSDF) evaluation to ensure proper disposition of DOE waste. This includes waste packaging, control and tracking, labeling, classification identification, and preparing/forwarding manifests;

9.2.1.i **(DOE-Only Requirement)** provide tracking of individual sample containers from receipt to final disposition;

9.2.1.j **(DOE-Only Requirement)** address waste minimization and pollution prevention program requirements or plans which include substitution (when permitted), segregation, and recycling; and

9.2.1.k **(DOE-Only Requirement)** identify how radioactive and mixed wastes shall be segregated from non-radioactive waste.

9.2.2 (DOE-Only Requirement) Waste Disposal

Laboratories shall develop and implement procedures to address waste disposition resulting from the receipt, analysis, and shipping of DOE samples, which shall address the following requirements:

9.2.2.a **(DOE-Only Requirement)** Waste shipments shall only be transferred to a qualified facility/person specifically licensed to receive the waste.

9.2.2.b **(DOE-Only Requirement)** The laboratory shall develop criteria for evaluating waste brokers and TSDFs based upon a site visit to the waste facility or a desktop review that includes information from audits conducted by state or federal agencies. The evaluation shall include liability coverage, financial stability, any Notice of Violation from the last three years, applicable permits and licenses to accept the waste, and other relevant information. DOECAP TSDF audits can be used in place of onsite visit requirements, provided other requirements not included in these audits are addressed (e.g., financial stability, liability insurance, etc.).

9.2.2.c **(DOE-Only Requirement)** The laboratory shall remove or deface all sample container labels before container disposal such that they are rendered illegible.

9.2.2.d **(DOE-Only Requirement)** Analytical process waste shall be segregated and removed to a designated storage area to minimize the potential for cross-contamination.

9.2.2.e **(DOE-Only Requirement)** Laboratory analysis for derived waste characterization shall be repeated at a frequency adequate to account for all known variations in the waste streams.

9.2.2.f **(DOE-Only Requirement)** Samples that are consumed during analysis shall be included in the sample accountability tracking.

9.2.2.g (DOE-Only Requirement) Management of excess samples whether they are bulked, special samples, or drain disposed.

9.2.2.h (DOE-Only Requirement) The laboratory shall address how it manages the requirements for the pre-treatment requirements if disposal includes a Publicly Owned Treatment Works or wastewater treatment system. The program shall address how the laboratory demonstrates compliance with these requirements.

9.2.2.i (DOE-Only Requirement) Satellite Accumulation Area-Laboratories shall not accumulate more than 55 gallons of hazardous and mixed waste or no more than one quart of acutely hazardous waste at, or near, any point of generation. The labelling of these waste containers are to be properly marked with the words ‘‘Hazardous Waste”. The container label shall also indicate the applicable hazard (accepted labels include completed Department of Transportation (DOT) shipping label, National Fire Protection Association (NFPA) label, or Resource Conservation and Recovery Act (RCRA) waste characterization code). When the container is full, it shall be marked with the accumulation start date and moved to the central accumulation area (CAA) within 3 days.

9.2.2.j (DOE-Only Requirement) Radioactive and mixed wastes generated during laboratory sample processing shall be labeled as radioactive.

9.2.3 (DOE-Only Requirement) Waste Storage Areas

9.2.3.a (DOE-Only Requirement) The laboratory shall identify the waste storage area’s, or CAA’s affiliation and requirements for its RCRA status as a very small, small, or large quantity generator and shall identify the locations, storage limitations, and container sizes for each accumulation and storage area identified. The laboratory shall record the area's specific site affiliation and generator size, and define central and satellite accumulation, and other requirements in accordance with the applicable federal, state, and local regulations.

9.2.3.b (DOE-Only Requirement) The laboratory shall select and label hazardous waste containers according to the use to include, at a minimum:

9.2.3.b.i labeled with the words “Hazardous Waste”; and

9.2.3.b.ii labeled with an indication of the hazards of the contents.

9.2.3.c (DOE-Only Requirement) The laboratory shall maintain records of weekly inspections of CAAs. The procedure shall require inspections be performed by trained personnel and in accordance with federal, state, and local regulations and containers shall be labeled with the accumulation start date.

9.2.3.d (DOE-Only Requirement) Ignitable and reactive waste shall be stored at least 50 feet from the property line.

9.2.3.e (DOE-Only Requirement) Incompatible wastes shall not be stored together. Containers incompatible with any waste or other materials accumulated nearby shall be separated or protected from them by any practical means such as by dike, berm, wall, or other device. Refer to the Safety Data Sheet for proper

storage requirements and precautions.

9.2.3.f (DOE-Only Requirement) The waste storage area shall provide secondary containment of sufficient capacity for the waste expected to be stored in the areas.

9.2.3.g (DOE-Only Requirement) Accumulation containers shall be:

9.2.3.g.i in good condition;

9.2.3.g.ii compatible with the waste; and

9.2.3.g.iii kept closed at all times when not in immediate use.

9.2.4 (DOE-Only Requirement) Toxic Substances Control Act (TSCA) Material

9.2.4.a (DOE-Only Requirement) The laboratory shall develop and implement a plan or program stating how laboratory operations comply with all federal regulations governing TSCA materials control and protection.

9.2.4.b (DOE-Only Requirement) The laboratory shall segregate all radioactive TSCA materials from all other analytical samples and associated derived wastes.

9.2.4.c (DOE-Only Requirement) The laboratory shall have a procedure for return to the customer of radioactive TSCA materials for which there are no commercial treatment or disposal options.

9.2.4.d (DOE-Only Requirement) The TSCA polychlorinated biphenyl (PCB) waste shall be stored for less than one year from the date the material was first placed in storage.

9.2.4.e (DOE-Only Requirement) The TSCA PCB waste containers shall be labeled with the accumulation start date.

9.2.4.f (DOE-Only Requirement) The TSCA one-year waste storage area shall meet the storage facility requirements for PCB waste (floor curbing, above the 100-year flood plain, no floor drains, etc.).

9.2.4.g (DOE-Only Requirement) Wastes from samples containing PCBs at greater than 50 ppm shall be segregated from other laboratory wastes as TSCA regulated waste.

Note: This does not apply to the extracted sample residual, BUT it does apply to the extract and other laboratory process wastes.

9.2.4.h (DOE-Only Requirement) Laboratory-generated TSCA PCB wastes shall not be stored in a Temporary Storage Area for more than 30 days from the time of generation unless the area meets one-year storage facility requirements.

9.2.4.i (DOE-Only Requirement) TSCA PCB waste containers and sample storage areas shall be marked with the required TSCA PCB labeling.

9.3 (DOE-Only Requirement) Chemical Hygiene Plan (CHP)

9.3.1 (DOE-Only Requirement) A CHP shall be developed and implemented in the laboratory and available to all employees. Procedures relating to safety and health considerations shall be developed and implemented. The contingency plan shall address temporary closures by identifying steps to prepare the laboratory before a closure, ramping down operations, and planning for bringing the laboratory back up to operational status. The plan shall include handling of samples, radiation protection, chemical hazards, and waste.

9.3.2 (DOE-Only Requirement) The laboratory shall have a written contingency plan and ensure a copy is available at the facility.

9.3.3 (DOE-Only Requirement) The following information shall be included in the plan and posted next to the phone in the vicinity of the CAA:

9.3.3.a name and number of the emergency coordinator;

9.3.3.b location of fire extinguishers and spill control material; and

9.3.3.c fire department number or a direct alarm.

9.3.4 (DOE-Only Requirement) Required equipment shall be available at the assembly area. Equipment includes, but is not limited to:

9.3.4.a internal communication or alarm system;

9.3.4.b hand-held two-way radio or cell phone;

9.3.4.c portable fire extinguishers/fire control equipment; and

9.3.4.d spill control equipment and water at adequate volume and pressure (i.e., > 15 minutes of continuous pressure).

9.3.5 (DOE-Only Requirement) An emergency eyewash shall be located within the immediate work area, unobstructed, and shall be available to all personnel. Location requirements and ease of access, frequency for testing, refilling, or restocking as needed, and an emergency shower shall be addressed in the plan. All tests and inspections shall be clearly marked by a tag on each device.

9.3.6 (DOE-Only Requirement) The laboratory shall provide mounted, located, identified, and inspected portable fire extinguishers to be available to all employees without subjecting the employees to possible injury. The location requirements and the frequency for inspection shall be established in the Chemical Hygiene Plan, or equivalent plan. All tests and inspections shall be clearly marked by a tag on each device.

9.3.7 (DOE-Only Requirement) The laboratory shall have a spill control plan developed and implemented to include a record of spillage of customer samples or significant spillage of chemicals.

9.3.8 (DOE-Only Requirement) The facility shall be equipped with an alarm system capable of being detected and recognized by the employee in case of an emergency.

9.3.9 (DOE-Only Requirement) Initial and periodic exposure monitoring for hazardous chemicals shall be conducted. Exposure limits and actions to be taken should an exceedance occur shall be identified or referenced.

9.3.10 (DOE-Only Requirement) Safety Data Sheets shall be on file for all hazardous chemical substances maintained by the laboratory and readily accessible to all employees.

9.3.11 (DOE-Only Requirement) The laboratory shall have a procedure for ongoing verification and maintenance of ventilation hoods.

9.3.11.a The procedure shall include verification of flow rates on a semi-annual basis at a minimum, using a smoke test or flow meter measurement. The laboratory shall maintain records of the verification which include the tested flow rate for each hood.

9.3.11.b The procedure shall include monitoring ventilation hoods for radioactive contamination at a prescribed frequency. Records shall be maintained of the monitoring.

9.3.12 (DOE-Only Requirement) If respirators are used during sample or waste handling/processing, the laboratory shall have an appropriate written respiratory protection program, including:

9.3.12.a procedures governing the fit-testing of personnel using respirators;

9.3.12.b selection and use of respirators; and

9.3.12.c an annual evaluation to ensure effectiveness.

9.3.13 (DOE-Only Requirement) Chemical hazard labeling on chemical containers shall be in accordance with the laboratory's approved CHP.

9.3.14 (DOE-Only Requirement) A laboratory safety inspection program shall be developed and implemented that includes routine inspections of laboratory areas for health and safety-related concerns.

9.3.15 (DOE-Only Requirement) Safety orientation briefings shall be required of all visitors, vendors, contractors, maintenance personnel, and auditors before entering the laboratory.

9.3.16 (DOE-Only Requirement) The laboratory shall have a designated and alternate Hazardous Waste Operations and Emergency Response (HAZWOPER) trained person on staff.

9.3.17 (DOE-Only Requirement) The laboratory shall develop an emergency response plan to include re-entry procedures once the laboratory is safe to return.

9.3.18 (DOE-Only Requirement) The laboratory shall require clear posting of signs on doors, workstations, and/or safety devices to indicate use of:

9.3.18.a safety glasses required;

9.3.18.b laboratory coats or protective clothing;

9.3.18.c appropriate footwear;

9.3.18.d safety showers;

9.3.18.e eyewash stations;

9.3.18.f other safety and first aid equipment;

9.3.18.g exits; and

9.3.18.h areas where food and beverage consumption and storage are not permitted.

9.3.19 (DOE-Only Requirement) Areas containing biological hazards shall be appropriately posted.

9.3.20 (DOE-Only Requirement) The laboratory shall establish and implement a procedure for identifying hazardous and toxic chemicals located within the laboratory, locations stored, and training of personnel. The procedure shall address precautions for handling and storing all hazardous and toxic chemicals used to include proper identification of storage areas.

9.3.21 (DOE-Only Requirement) All hazardous or toxic chemical cabinets shall be appropriately labeled with the following:

9.3.21.a identity of the hazardous chemical; and

9.3.21.b appropriate hazard warnings.

9.3.22 (DOE-Only Requirement) All exits shall be properly identified and unobstructed.

9.3.23 (DOE-Only Requirement) Locations and procedures for personal protective equipment (PPE), (to include laboratory coats, safety glasses, shoes, etc.) shall be established. These procedures shall identify when, what, and where PPE is required and allowed.

9.4 (DOE-Only Requirement) Sample Receiving and Control

9.4.1 (DOE-Only Requirement) The laboratory shall have a documented system for uniquely identifying the items (samples) to be tested.

9.4.2 (DOE-Only Requirement) The laboratory shall have procedures in place to address the following:

9.4.2.a containers are opened in a manner to prevent worker exposure;

9.4.2.b checking sample preservation (pH);

9.4.2.c proper containers;

9.4.2.d preserving samples when required;

9.4.2.e recording and notifying customers of shipping or sample anomalies;

9.4.2.f checking holding times and notifying laboratory personnel of short holding times;

9.4.2.g use of fume hoods for opening samples and shipping containers;

9.4.2.h how chain of custody is maintained during times when laboratory personnel are not present;

9.4.2.i access to all samples and subsamples is controlled and recorded;

9.4.2.j chain of custody forms remain with the samples during transport or shipment; and

9.4.2.k recording the chronology of sample entry into the laboratory, including, but not limited to, time, date, customer, sample identification numbers, signature, or initials of person making the entry.

9.4.3 (DOE-Only Requirement) Materials submitted to the laboratory for industrial hygiene or asbestos analyses shall be opened in an established manner to prevent worker exposure. Sample receiving practices shall be developed and implemented for the receipt of beryllium, beryllium oxide, and asbestos materials.

9.4.4 (DOE-Only Requirement) The sample receipt personnel shall record anomalies encountered in the sample receiving process.

9.4.5 (DOE-Only Requirement) A sample receiving logbook or equivalent system shall be used to record the chronology of sample entry into the laboratory, including, but not limited to, time, date, customer, sample identification numbers, signature, or initials of the person making the entry.

9.4.6 (DOE-Only Requirement) When the laboratory receives samples, there shall be an internal chain of custody procedure in place. Internal custody shall be maintained until final disposition or return of the sample to the customer.

9.5 (DOE-Only Requirement) Records

9.5.1 (DOE-Only Requirement) The following records shall be maintained for a minimum of five years:

9.5.1.a radioactive material management audit, review, and inspection reports;

9.5.1.b records of airborne release of hazardous materials;

9.5.1.c daily operational checks of radiological survey equipment;

9.5.1.d TSDF waste brokering evaluation or review reports and a list of approved facilities;

9.5.1.e waste disposal certificates of disposal or destruction;

9.5.1.f waste characterization information, including analytical test results and process knowledge determinations; and

9.5.1.g semi-annual ventilation hood and protective equipment contamination control verifications.

9.6 (DOE-Only Requirement) Training

9.6.1 (DOE-Only Requirement) The following training shall be provided to all appropriate laboratory employees and records of training maintained:

9.6.1.a RSO training for both the designated RSO and backup RSO;

9.6.1.b radiation general awareness, security, and safety for those laboratory personnel and contractors dealing with radioactive waste management;

9.6.1.c Hazardous Waste Satellite Accumulation Area management;

9.6.1.d spill detection, cleanup procedures, and spill kit location;

9.6.1.e safety training (annual); and

9.6.1.f HAZWOPER.

Module 3

Quality Systems for Asbestos Testing

1.0 Introduction

This module provides specific requirements for laboratories seeking accreditation for asbestos testing. Laboratories seeking accreditation for asbestos testing shall also meet the requirements presented in Module 1 and 2 of this standard. This module is organized by analytical technique including transmission electron microscopy (TEM) for the analysis of water, wastewater, air, and bulk samples; phase contrast microscopy (PCM) for analysis of workplace air; and polarized light microscopy (PLM) for analysis of bulk samples. These procedures for asbestos analysis involve sample preparation followed by detection of asbestos.

2.0 Scope

This module provides quality assurance (QA) and quality control (QC) requirements for laboratories seeking to obtain and maintain accreditation for asbestos testing methods. This module is not intended to relieve additional quality control requirements that are specified by method, regulation, or customer.

3.0 Terms and Definitions

Definitions are considered requirements. Definitions are included in Module 2, Section 3.0.

4.0 Method Selection

The requirements in the Module 2 section on “Selection, Verification and Validation of Methods” apply.

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

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

5.0 Method Validation

Before acceptance and institution of any method for which data will be reported, all methods

shall be validated. For all methods except reference methods, validation shall meet the requirements in the Module 2 section “Selection, Verification and Validation of Methods” as well as all criteria in this Module.

6.0 Demonstration of Capability (DOC)

6.1 General

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

6.1.1.a The Initial DOC shall be a blind sample (i.e., one whose true type and value is unknown to the analyst).

6.1.2 Thereafter, ongoing DOCs in accordance with this manual are required.

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

6.1.4 All demonstrations shall be recorded. All data applicable to the demonstration shall be retained and readily available at the laboratory.

6.2 Initial DOC

An individual shall successfully perform an initial DOC before using any method (see 1.6.1.a) above), and at any time there is a change in instrument type or method, or any time that a method has not been performed by the analyst in a 12-month period.

6.2.1 The laboratory shall maintain records of each initial DOC in a manner such that the following information is readily available for each affected employee:

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

6.2.1.b matrix;

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

6.2.1.d identification of method(s) performed;

6.2.1.e identification of laboratory-specific procedures used for analysis, including revision number;

6.2.1.f date(s) of analysis; and

6.2.1.g summary of analyses, including information outlined in Section 6.2.2.c.

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

6.2.2.a The analyte(s) shall be diluted in a volume of clean quality system matrix (a sample in which no target analytes or interferences are present at concentrations that will impact the results of a specific method) sufficient to prepare four aliquots.

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

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

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

6.2.2.e When one or more of the tested analytes fail at least one of the acceptance criteria, the analyst shall proceed by beginning with c) above, repeat the test for all analytes that failed to meet criteria.

6.2.2.f Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate, and correct the source of the problem and repeat the test for all compounds of interest beginning with b).

6.3 Ongoing DOC

6.3.1 The laboratory shall have a procedure describing ongoing DOC that includes procedures for how the laboratory will identify data associated with ongoing DOCs. The analyst(s) shall demonstrate on-going capability by routinely meeting the QC requirements of the method, laboratory procedures, customer specifications, and/or this standard. If the method has not been performed by the analyst in a 12-month period, an initial DOC shall be performed. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

6.3.2 For asbestos, this ongoing DOC may be one of the following:

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

6.3.2.b another initial DOC;

6.3.2.c at least four consecutive laboratory control samples (LCS) with acceptable levels

of precision and accuracy. The laboratory shall determine the acceptable limits for precision and accuracy before analysis. The laboratory shall tabulate or be able to readily retrieve four (4) consecutive passing LCS or reference sample(s) for each method for each analyst each year;

6.3.2.d following a procedure for reviewing records of QC samples meeting the QC requirements of the method, laboratory procedure, customer requirements, and/or this standard. A review of these records may be used to identify patterns and determine if implementation of the nonconforming work process and/or retraining is necessary; or

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

7.0 Technical Requirements

7.1 Calibration

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

7.1.1 Transmission Electron Microscopy

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

7.1.1.1 Water and Wastewater

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

7.1.1.1.a Magnification Calibration. Magnification calibration shall be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000 and 20,000x. Records of calibration shall be maintained. Calibrations shall be performed monthly to establish the stability of magnification. Calibration data shall be recorded such that trends are detectable.

7.1.1.1.b Camera Constant. The camera length of the TEM in the Selected Area Electron Diffraction (SAED) mode shall be calibrated before SAED patterns of unknown samples are observed. The diffraction specimen shall be at the eucentric position for this calibration. This calibration shall allow accurate (less than 10% variation) measurement of layer-line spacings on the medium used

for routine measurement, i.e., the phosphor screen or camera film. This shall also allow accurate (less than 5% variation) measurement of zone axis SAED patterns on permanent media (e.g., film). Calibrations shall be performed monthly to establish the stability of the camera constant. Where non-asbestiform minerals may be expected (e.g., winchite, richterite, industrial talc, vermiculite, etc.), an internal camera constant standard such as gold, shall be deposited and measured on each sample to facilitate accurate indexing of zone axis SAED patterns. In such cases, layer line analysis alone shall not be used. Calibration data shall be recorded such that trends are detectable.

7.1.1.1.b.i A gold standard grid shall be used to obtain the characteristic diffraction rings from which the camera constant can be calculated.

7.1.1.1.c Spot Size. The diameter of the smallest beam spot at crossover shall be less than 250 nm as calibrated quarterly. Calibration data shall be recorded such that trends are detectable.

7.1.1.1.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 greater than 1 µm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 15 seconds.

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

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

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

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

7.1.1.1.e.iv The detector resolution shall be checked quarterly to ensure a full-width half maximum resolution of less than 175 eV at Mn Kα (5.90 keV). Calibration data shall be recorded such that trends are detectable.

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

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

7.1.1.1.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 shall be recorded such that trends are detectable.

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

7.1.1.2 Air

All calibrations shall be performed in accordance with Section 7.1.1.1, except for magnification. Magnification calibration shall be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 15,000 to 20,000x. Records of calibration shall be maintained. Calibrations shall be performed monthly to establish the stability of magnification.

7.1.1.3 Bulk Samples

All calibrations shall be performed in accordance with Section 7.1.1.1.

7.1.2 Phase Contrast Microscopy

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

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

7.1.2.3 Before ordering the Walton-Beckett graticule, calibration, in accordance with National Institute for Occupational Safety and Health (NIOSH) 7400, shall be performed to obtain a counting area 100 µm in diameter at the image plane. The diameter, dc (mm), of the circular counting area and the disc diameter shall be specified when ordering the graticule. The field diameter (D) shall be verified (or checked), to a tolerance of 100 µm ± 2 µm, with a stage micrometer upon receipt of the graticule from the manufacturer. When changes (zoom adjustment, disassembly, replacement, etc.) occur in the eyepiece-objective-reticle combination, field diameter shall be re-measured (or recalibrated) to determine

field area (mm^2). Recalibration of field diameter shall also be required when there is a change in interpupillary distance (i.e., change in analyst). Acceptable range for field area shall be 0.00754 mm^2 to 0.00817 mm^2. The actual field area shall be recorded and used.

7.1.3 Polarized Light Microscopy

7.1.3.1 Microscope Alignment

7.1.3.1.a Both stereoscope and polarized light microscope shall be aligned and checked for function and optimized for correct operation before every use by every analyst.

7.1.3.1.b Records of all alignments and function checks shall be maintained.

7.1.3.2 Refractive Index Liquids. Series of nD = 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.

7.2 Quality Control

7.2.1 Negative Controls

7.2.1.1 Transmission Electron Microscopy

7.2.1.1.a Water and Wastewater

7.2.1.1.a.i Blank determinations shall be made before 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 less than 0.01 million fibers per liter (MFL) greater than 10 µm.

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

7.2.1.1.b Air

7.2.1.1.b.i A blank filter shall be prepared with each set of samples. A blank filter shall be left uncovered during preparation of the sample set and a wedge from that blank filter shall be prepared alongside wedges from the sample filters. At minimum, the blank filter shall be analyzed at a frequency of one per 20 samples analyzed.

7.2.1.1.b.ii 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.

7.2.1.1.c Bulk Samples

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

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

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

7.2.1.2 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 before analysis. Results from field blank samples shall be used in the calculation to determine final airborne fiber concentration. The identity of blank filters shall be unknown to the counter until all counts have been completed. If a field blank yields greater than seven fibers per 100 graticule fields, report possible contamination of the samples.

7.2.1.3 Polarized Light Microscopy

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

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

7.3 Test Variability/Reproducibility

7.3.1 Transmission Electron Microscopy. Quality assurance (QA) analyses shall be performed regularly covering all time periods, instruments, tasks, and personnel. The selection of samples shall be random, and samples of special interest may be included in the selection of samples for QA analyses. When possible, the checks on personnel performance shall be executed without their prior knowledge. A disproportionate number of analyses shall not be performed before internal or external audits. It is recommended that a laboratory initially be at 100% QC (all samples re-analyzed). The proportion of QC samples may later be lowered gradually, as control indicates, to a minimum of 10%.

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

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

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

7.3.1.1.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 in accordance with NISTIR 5351, *Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0* (S. Turner and E.B. Steel, 1994). This comparison shall be performed at a frequency of one per 20 samples. Qualified analysts shall maintain an average of greater than or equal to 80% true positives, less than or equal to 20% false negatives, and less than or equal to 10% false positives.

7.3.1.2 Air

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

7.3.1.2.b The laboratory and TEM analysts shall obtain mean analytical results on NIST SRM 1876b so that trimmed mean values fall within 80% of the lower limit and 110% of the upper limit of the 95% confidence limits as published on the certificate. These limits are derived from the allowable false positives and false negatives given in Section 7.3.1.1.c, Verified Analysis. SRM 1876b shall be analyzed a minimum of once per year by each TEM analyst.

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

7.3.1.2.d Inter-laboratory analyses shall be performed to detect laboratory bias. The frequency of inter-laboratory verified analysis shall correspond to a minimum of one per 200 grid square analyses for customers.

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

7.3.1.2.e.i Replicate. A second, independent analysis shall be performed in accordance with Section 7.3.1.1.a.

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

7.3.1.2.e.iii Verified Analyses. A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section 7.3.1.1.c.

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

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

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

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

7.3.2 Phase Contrast Microscopy

7.3.2.a Inter-Laboratory Precision. Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory quality assurance program that includes participation of at least two 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 quality assurance group. Each laboratory shall submit slides typical of its own workload for use in this program. The round robin shall be designed, and results analyzed using appropriate statistical methodology. Results of this QA program shall be posted in each laboratory to keep the microscopists informed.

7.3.2.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.

7.3.3 Polarized Light Microscopy

Refer to Section 7.3.1.3.

7.4 Other Quality Control Measures

7.4.1 Transmission Electron Microscopy

7.4.1.a Water and Wastewater

7.4.1.a.i 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 greater than 10 µm 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 NISTIR 5351.

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

7.4.1.b Air

7.4.1.b.i Filter preparations shall be made from all six asbestos types in accordance with Section 7.4.1.a.i.

7.4.1.b.ii NIST SRM 1876b shall be analyzed annually.

7.4.1.c Bulk Samples. All analysts shall 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).

7.4.2 Phase Contrast Microscopy

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

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

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

7.4.3 Polarized Light Microscopy

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

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

7.5 Analytical Sensitivity

7.5.1 Transmission Electron Microscopy

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

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

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

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

7.5.3 Polarized Light Microscopy. The laboratory shall utilize a method that provides a limit of detection that is appropriate and relevant for the intended use of the data. Limit of detection shall be determined by the procedure in the method or applicable regulation.

7.6 Quality of Standards and Reagents

7.6.1 Transmission Electron Microscopy

7.6.1.a The quality control program shall establish and maintain provisions for asbestos standards.

7.6.1.b Reference standards that are used in an asbestos laboratory shall be obtained from NIST, EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. Any reference standards purchased outside the United States shall be traceable back to each country's National Metrology Institute.

7.6.1.c All reagents used shall be analytical reagent grade or better.

7.6.1.d 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.

7.6.2 Phase Contrast Microscopy. Standards of known concentration have not been developed for this testing method. Routine workload samples that have been statistically validated and national proficiency testing samples such as Proficiency Analytical Testing (PAT) and Asbestos Analysts Registry (AAR) samples available from the American Industrial Hygiene Association (AIHA) may be utilized as reference samples 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 NIOSH 7400.

7.6.3 Polarized Light Microscopy

Refer to Section 7.6.1.

7.7 Data Acceptance/Rejection Criteria

7.7.1 Transmission Electron Microscopy

7.7.1.1 Water and Wastewater

7.7.1.1.a The concentration of asbestos in a given sample shall be calculated in accordance with EPA/600/R-94/134, Method 100.2, Section 12.1, *Determination of Asbestos Structures Over 10 µM in Length in Drinking Water*.

7.7.1.1.b Measurement Uncertainties. The laboratory shall calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

7.7.1.2 Air

7.7.1.2.a The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized.

7.7.1.2.b Measurement Uncertainties. The laboratory shall calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

7.7.1.3 Bulk Samples

7.7.1.3.a The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, *Method for the Determination of Asbestos in Bulk Building Materials*).

7.7.1.3.b Measurement Uncertainties. Proficiency testing for floor tiles analyzed by TEM following careful gravimetric reduction has revealed an inter-laboratory standard deviation of approximately 20% for residues containing 70% or more asbestos. Standard deviations range from 20% to 60% for residues with lower asbestos content.

7.7.2 Phase Contrast Microscopy

7.7.2.1 Airborne fiber concentration in a given sample shall be calculated in accordance with NIOSH 7400.

7.7.2.2 Measurement Uncertainties. The laboratory shall calculate and report the intra-laboratory and inter-laboratory relative standard deviation with each set of results in accordance with NIOSH 7400.

7.7.2.3 Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with greater than 50% of the filter area covered with particulate shall be reported as “uncountable” or “probably biased”. Other fiber counts outside the 100-1300 fibers/mm^2 range shall be reported as having “greater than optimal variability” and as being “probably biased”.

7.7.3 Polarized Light Microscopy

7.7.3.1 The concentration of asbestos in a given sample shall be calculated in accordance with the method utilized (e.g., EPA 600/M4-82-020, *Interim Method for the Determination of Asbestos in Bulk Insulation Samples*).

7.7.3.2 Method Uncertainties. Precision and accuracy shall be determined by the

individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonable expanded errors shall be generated for different concentrations of asbestos.

7.8 Constant and Consistent Test Conditions (Sample and Sampling Requirements)

7.8.1 Samples shall be transported to the laboratory as soon as possible after collection. Date and time of sampling shall be noted on submittal forms. The names of the collectors with their signatures and the site shall be included on the chain-of-custody forms. No preservatives are required during sampling.

7.8.2 The laboratory shall establish and adhere to written procedures to minimize the possibility of cross contamination between samples.

7.8.3 Refer to the specific method of analysis for additional requirements.

Module 4

Quality Systems for Chemical Testing

1.0 Introduction

This module provides specific requirements for laboratories seeking accreditation for chemical testing. Laboratories seeking accreditation for chemical testing shall also meet the requirements presented in Module 1 and 2 of this standard and the applicable requirements found in Appendix B.

2.0 Scope

This module provides quality assurance (QA) and quality control (QC) requirements for laboratories seeking to obtain and maintain accreditation for chemical testing methods. This module is not intended to relieve additional quality control requirements that are specified by method, regulation, or customer.

3.0 Terms and Definitions

Definitions are considered requirements. Definitions are included in Module 2, Section 3.0.

4.0 Method Selection

The requirements in the Module 2 section on “Selection, Verification and Validation of Methods” apply.

When adding a new analyte to a reference method, the inclusion of the analyte in the method shall meet all required calibration requirements and the QC requirements of the method to which the analyte is being added. If no QC requirements exist in the method, the laboratory shall adhere to the requirements outlined in a reference method of the same technology (when available). When adding an analyte, the laboratory shall follow the requirements of the relevant regulations to determine whether the addition of an analyte represents a method modification.

5.0 Method Validation

5.1 Validation of Methods

Before acceptance and institution of any method for which data will be reported, all methods shall be validated.

5.1.1 Method validation shall meet the requirements in the Module 2 section on “Selection, Verification, and Validation of Methods” as well as all criteria in this module.

5.1.2 The laboratory shall validate reference methods through the initial determinations of a detection limit (DL) if required, a limit of detection (LOD) if required, and a limit of

quantitation (LOQ) as well as an Initial Demonstration of Capability (DOC). Requirements for DL, LOD and LOQ are contained in this module's section on "Detection Limits, Limits of Detection, and Limits of Quantitation." If the reference method has additional validation requirements, these requirements shall also be met.

5.1.3 In addition to the QC procedures for reference methods, the laboratory shall validate modified reference methods and non-reference methods (including laboratory-developed methods) using QC procedures and acceptance criteria that are consistent with those of similar reference methods or technologies, and the validation shall include the following:

5.1.3.a scope;

5.1.3.b calibration verification;

5.1.3.c interferences and cross-contamination;

5.1.3.d analyte identification;

5.1.3.e analyte quantitation;

5.1.3.f selectivity;

5.1.3.g sensitivity; and

5.1.3.h precision and bias.

5.1.4 The use of any modified or non-reference method shall be approved by the customer before use.

5.1.5 Methods shall be validated when substantive modifications are made to reference methods (e.g., stoichiometry, technology, mass tuning acceptance criteria, quantitation ions, compressing digestion or extraction timeframes, reducing reagent or solvent volumes, or changing solvents).

5.1.6 When a modification of a method includes changes to sample preparation steps, the validation process shall include analysis of field samples in the matrices of concern. The field samples shall represent a range of characteristics encountered or expected in customer samples. Examples of characteristics to consider include organic matter content, clay content, moisture content, pH, and dissolved/suspended solids. Validation shall include parallel studies to compare performance of the reference method to the modified method, where possible.

5.1.7 The field samples shall contain target analytes either found natively in the samples or spiked into the sample. Validation shall include multiple levels of target analyte concentrations. Where the method includes more than 45 target analytes, a representative subset of at least 45 analytes may be used. If a subset of analytes is used for validation, the subset shall include all chemistries and target analytes that show the most bias when analyzed using the reference method.

5.1.8 Where modifications to only the analytical portion of the method are planned, the

laboratory shall take into consideration any effects the matrix may have on the analysis as part of its risk assessment.

5.2 Detection Limit, Limit of Detection, and Limit of Quantitation

For each combination of analyte-matrix-method, the laboratory shall have procedures for determining and verifying DL, LOD, and LOQ, as defined by the QSM, that reflect current operating conditions. DL, LOD, and LOQ determinations are not required for methods/analytes for which a DL, LOD and LOQ is not applicable such as pH, color, odor, temperature, residues, or leaching procedures. DL determinations, LOD determinations, and LOD and LOQ verifications are not required for analytes for which no low-level spiking solutions are available. DL and LOD determinations are not required if results are not reported below the LOQ, unless required by regulation or method.

For each preparation method listed on the scope of accreditation, the laboratory shall determine a DL, LOD, and LOQ, unless it falls within one of the stated exceptions. Although the laboratory is not required to determine a separate DL, LOD and LOQ for all possible combinations of preparation and cleanup techniques in use, the laboratory shall determine the DL, LOD, and LOQ using the combination of processes most likely to interfere with sensitivity (i.e., preparation method with all applicable cleanup steps including drying, grinding, and incremental sampling, where applicable).

The DL, LOD and LOQ shall be reported for all analyte-matrix-method combinations unless it is not applicable to the test or specifically excluded by customer requirements.

Records of all supporting data for DL, LOD, and LOQ determinations and verifications shall be maintained.

5.2.1 Determination of the Detection Limit

The laboratory shall determine a DL using a procedure that is compliant with the requirements of the 40 CFR Part 136 Appendix B, Revision 2 for each combination of analyte-matrix-method.

5.2.2 Initial Determination of the Limit of Detection

The LOD determination procedure shall address the following requirements:

5.2.2.a After each DL determination, the laboratory shall establish the LOD by spiking a quality system matrix at a concentration greater than or equal to the DL. The LOD is equal to the concentration of this spike.

5.2.2.b The apparent signal to noise (S/N) ratio at the LOD shall be at least three and the results shall meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition).

5.2.2.c For data systems that do not provide a measure of noise, the signal produced by the LOD spike shall produce a response that is at least three standard deviations

greater than the mean Method Blank concentration. The mean Method Blank concentration and standard deviation are initially estimated based on a minimum of four Method Blank results and later established with a minimum of 20 Method Blank results.

5.2.2.c.i Alternatively, the signal produced by the LOD spike shall generate a result above the MDLb derived in accordance with EPA 821-R-16-006.

5.2.2.d If the LOD spike response does not meet the requirements, the laboratory shall repeat the DL and/or LOD determination at a higher level, or implement its nonconforming work procedure, until the requirements are met.

5.2.3 Ongoing Verification of the Limit of Detection

The LOD verification procedure shall address the following requirements:

5.2.3.a The laboratory shall verify the LOD on a quarterly basis, at a minimum. The verification is made by repeating the LOD spike process at a concentration that is greater than or equal to half the current LOD and less than or equal to double the current LOD (i.e., 1/2x LOD ≤ Ongoing LOD Spike ≤ 2x LOD) provided the ongoing spike concentration is greater than or equal to the DL. The Ongoing LOD verification shall meet the same acceptance criteria as the Initial LOD verification for signal-to-noise and analyte identification. If the acceptance criteria are met, the Initial LOD is verified and may continue to be used.

5.2.3.b In the event the verification fails, the laboratory shall redetermine the LOD and, if necessary, the DL, or implement its nonconforming work procedure, until the requirements are met.

5.2.3.c If the method is altered in a way other than routine maintenance, and the change can be expected to elevate the detection limit, the LOD shall be reverified using the Ongoing LOD verification procedure.

5.2.3.d If there are multiple instruments that will be assigned the same LOD, then the LOD verification spike shall meet the requirements on each instrument.

5.2.3.e In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform ongoing LOD verifications on a one-per-batch basis, before sample analysis, in lieu of quarterly verification. The verification data shall meet the requirements of this section and be reported to the customer. Whichever verification frequency is chosen shall be continued for a minimum of 12 months.

5.2.4 Initial and Ongoing Verification of the Limit of Quantitation

The LOQ verification procedure shall address the following requirements:

5.2.4.a For methods using multi-level calibration, the laboratory shall select an LOQ for each analyte that is greater than or equal to the LOD and greater than or equal to the lowest non-zero calibration standard. For methods using a single-point calibration, the LOQ shall be greater than or equal to the LOD and greater than or

equal to the low-level calibration check standard.

5.2.4.b The LOQ shall be verified through analysis of verification samples. The LOQ verification sample consists of a spiked quality system matrix greater than or equal to the LOD or one-half the LOQ, whichever is less, and less than or equal to double the LOQ (i.e., LOD or 1/2 LOQ ≤ LOQ spike ≤ 2x LOQ). The LOQ verification shall meet the same criteria as the initial LOD verification for signal-to-noise and analyte identification and shall be within the laboratory's stated acceptance criteria. The laboratory acceptance criteria shall be determined based on a maximum of three standard deviations from the mean of historical data, but no wider than the LCS acceptance criteria with an additional 20% allowance above and below. Additionally, the lower limit shall be greater than or equal to 10% recovery.

5.2.4.c In the event the verification fails, the laboratory shall redetermine the LOQ and, if necessary, the DL and/or LOD; or implement its nonconforming work procedure, until the requirements are met.

5.2.4.d The laboratory procedure for establishing the LOQ shall include how precision and bias are determined at the LOQ for each combination of analyte-matrix-method and shall meet the requirements of this section. The LOQ and associated precision and bias shall be within the laboratory established acceptance criteria and meet customer-provided requirements, when available, and shall be reported in each data package. If the method is modified, precision and bias at the new LOQ shall be demonstrated.

5.2.4.e If there are multiple instruments that will be assigned the same LOQ, then these LOQ verification spikes shall be distributed across all the instruments and the results shall be included in the precision and bias determination.

5.2.4.f The laboratory shall verify the LOQ quarterly, at a minimum. The precision and bias shall be updated annually, at a minimum, using any additional LOQ verification sample results.

5.2.4.g In situations where methods are set up and used on an infrequent basis, the laboratory may choose to perform Ongoing LOQ verifications on a one-per-batch basis, before sample analysis, in lieu of quarterly verification. The verification data shall meet requirements and be reported to the customer. Whichever verification frequency is chosen shall be continued for a minimum of 12 months.

Note: Precision and bias at the LOQ may be demonstrated by analyzing multiple LOQ-verification samples over multiple days. A minimum of four samples is recommended to be analyzed to establish the initial precision and bias. If specific methods recommend a greater number of LOQ-verification replicates, then follow the method (e.g., SW846 6010D and 6020B recommend seven replicates).

Precision may be determined by calculating the standard deviation for each set of analyte-matrix-method results. If precision is determined using standard deviation, then it may be reported as a percent of the spiked value. Bias may be determined by calculating the difference of the average

result from the spiked value. Bias may be reported either as average percent recovery or average percent difference at the LOQ. Bias will be negative, positive, or zero.

When reporting precision and bias at the LOQ within a data package, a table for each analyte-matrix-method should present the LOQ concentration and the associated precision and bias at that concentration. The table should also identify how precision and bias were calculated, the spike concentration if different from the LOQ, and how many data points (results) were used in the calculation.

5.2.5 The laboratory shall provide the following when DL, LOD, and LOQ summary information is requested:

5.2.5.a indication of which analyte/matrix/prep method/analytical method and instrument used;

5.2.5.b DL;

5.2.5.c claimed LOD;

5.2.5.d concentration of initial LOD spike and verification spike, if different;

5.2.5.e statement of compliance with analyte identification requirements;

5.2.5.f signal-to-noise value or statement of compliance with requirements;

5.2.5.g claimed LOQ;

5.2.5.h concentration of LOQ spike;

5.2.5.i recovery or result of LOQ spike;

5.2.5.j calculated precision and bias at the LOQ that incorporates historic and current data;

5.2.5.k accuracy acceptance criteria at the LOQ;

5.2.5.l description of precision and bias calculations; and

5.2.5.m if specifically requested, raw data to support parameters reported.

5.3 Evaluation of Method Precision and Bias

5.3.1 The laboratory shall determine the precision and bias of a method for each analyte of concern for each quality system matrix or follow a documented alternate procedure when the analyte cannot be spiked into the sample matrix and QC samples are not commercially available.

5.3.2 The laboratory shall have a procedure for determining precision and bias.

5.3.3 The laboratory shall process the samples through the entire measurement system for each analyte of interest.

5.3.4 Precision and bias measurements shall evaluate the method across the analytical calibration range of the method.

5.3.5 The laboratory shall compare results of the precision and bias measurements with criteria established by the customer, criteria given in the reference method, and/or criteria established by the laboratory.

5.4 Evaluation of Selectivity

The laboratory shall evaluate selectivity by following the checks established within the method, which may include mass spectral tuning, second column confirmation, inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, and electrode response factors.

6.0 Demonstration of Capability (DOC)

6.1 General

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

6.1.2 Thereafter, the individual shall perform ongoing DOC.

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

6.1.4 All data applicable to the DOC shall be retained and readily available at the laboratory.

6.2 Initial DOC

Each individual shall successfully perform an initial DOC before using any method, any time there is a change in instrument type or method that could potentially affect the precision and bias, sensitivity, or selectivity of the output, or any time that a method has not been performed by the individual in a 12-month period.

Note: Examples of method changes that could potentially affect the precision and bias, sensitivity, or selectivity of the output include a change in the detector, column type, matrix, method revision, or other components of the sample measurement system.

6.2.1 The laboratory shall have a procedure for performing an initial DOC.

6.2.2 The laboratory shall maintain records of each initial DOC in a manner such that the following information is readily available for each individual:

6.2.2.a individual(s) involved in preparation and/or analysis;

6.2.2.b matrix;

6.2.2.c analyte(s), class of analyte(s);

6.2.2.d identification of method(s) performed;

6.2.2.e identification of laboratory-specific procedure used for analysis, including revision number;

6.2.2.f date(s) of analysis; and

6.2.2.g summary of analyses, including information outlined in Section 6.2.3.c.

6.2.3 If the reference method or regulation does not specify how to perform an initial DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to initial DOC are adequate. For methods where spiking is not a viable option (e.g., leaching procedures), the laboratory approach shall include observation and evaluation of negative controls.

6.2.3.a The analyte(s) shall be spiked in a volume of clean quality system matrix (i.e., a matrix in which no target analytes or interferences are present at concentrations that will impact the results of a specific method) sufficient to prepare four aliquots at the concentration specified in the reference method, or if unspecified, at a concentration of one to four times the LOQ. The quality system matrix shall be similar to the associated samples. For analysis of metals in solids, materials such as washed sand or non-reactive beads are acceptable as a matrix.

6.2.3.b At least four aliquots shall be prepared and analyzed according to the method.

6.2.3.c Using all the results, calculate the mean recovery in the appropriate reporting units and the standard deviations of the sample (in the same units) for each analyte of interest. Where it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory shall assess performance against established and documented criteria.

6.2.3.d Compare the information from 6.2.3.c above to the corresponding acceptance criteria for precision and accuracy in the method, if applicable, or in laboratory-generated acceptance criteria if there are not acceptance criteria in the method. If all analytes meet the acceptance criteria, the analysis of actual samples may begin. If any one of the analytes does not meet the acceptance criteria, the performance is unacceptable for that analyte.

6.2.3.e When one or more of the tested analytes fail at least one of the acceptance criteria, the analyst shall proceed according to i) or ii) below:

6.2.3.e.i Locate and correct the source of the failure and repeat the DOC procedure for

all analytes of interest.

6.2.3.e.ii Repeat the DOC procedure for all analytes that failed to meet criteria.

6.2.3.f Repeated failure, however, confirms a general problem with the measurement system. If repeated failure occurs, locate and correct the source of the failure, and repeat the DOC procedure for all analytes.

6.2.3.g When an analyte not currently found on the laboratory's list of accredited analytes is added to an existing accredited method, an initial demonstration shall be performed for that analyte.

6.3 Ongoing DOC

6.3.1 The laboratory shall have a procedure for ongoing DOC that includes how the laboratory will identify data associated with ongoing DOCs. The individual shall demonstrate on-going capability by routinely meeting the QC requirements of the reference method, laboratory procedure, customer requirements, and/or this standard. If the method has not been performed by the individual in a 12-month period, an initial DOC shall be performed. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate. For methods where spiking is not a viable option (e.g., leaching procedures), the laboratory approach shall include observation and evaluation of negative controls.

6.3.2 This on-going demonstration may be one of the following:

6.3.2.a acceptable performance of a blind sample (single blind to the individual) or a blind proficiency testing sample on a similar method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624.1, or 5030/8260);

Note: Acceptable results for both detected and non-detected analytes are considered acceptable performance.

6.3.2.b another initial DOC;

6.3.2.c at least four consecutive laboratory control samples (LCS) with acceptable levels of precision and accuracy. The laboratory shall determine the acceptable limits for precision and accuracy, and those limits shall be within laboratory-developed LCS acceptance criteria. The laboratory shall tabulate or be able to readily retrieve four consecutive passing LCSs or reference samples for each method for each individual performed within the last 12-month period;

6.3.2.d following a procedure for reviewing records of QC samples meeting the QC requirements of the method, laboratory procedure, customer requirements, and/or this standard. A review of these records may be used to identify patterns and determine if implementation of the nonconforming work procedure and/or retraining is necessary; or

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

7.0 Technical Requirements

7.1 Calibration

This section specifies the essential elements that shall define the procedures and required records for initial calibration and continuing calibration verification for methods that use calibration models including, but not limited to, average response factor or linear or quadratic regression, to ensure that the data shall be of known quality for the intended use.

Calibration requirements for auxiliary equipment are specified in Module 2. This section does not specify detailed procedural steps for calibration but does establish the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which requirements are more stringent, then the requirements of the regulation or mandated method shall be followed.

7.1.1 Initial Calibration

Each reported analyte shall be associated with an acceptable initial calibration. If the initial calibration is not acceptable, the laboratory shall implement its nonconforming work procedure, and all associated samples shall be reanalyzed. The following items are required elements of initial calibration:

7.1.1.a The details of the initial calibration including calculations, integrations, acceptance criteria, and associated statistics shall be included or referenced in the procedure. When initial calibration procedures are referenced then the referenced procedures shall be retained by the laboratory.

7.1.1.b Sufficient raw data records shall be retained to permit reconstruction of the initial calibration (e.g., calibration date, method, unique instrument identification, analysis date, each analyte name, individual initials or signature, concentrations, calibration curve, response factors, or unique equation or coefficient used to reduce instrument responses to concentration).

7.1.1.c The laboratory shall use the most recent initial calibration analyzed prior to the analytical batch, unless otherwise specified by the method.

7.1.1.d The laboratory shall perform calibrations for reported analytes with Certified Reference Materials (CRM) specifically identified as such in an accompanying Certificate of Analysis from a Reference Material Producer (RMP) accredited to ISO 17034 or Standard Reference Materials (SRM) from a National Metrology Institute (NMI), when available.

7.1.1.e The laboratory shall have a written procedure addressing removal and replacement of calibration standards. The procedure shall comply with the following requirements:

7.1.1.e.i The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of individual analytes in interior levels is not permitted.

7.1.1.e.ii The laboratory may remove one entire calibration standard from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. A laboratory that chooses to remove a calibration level from the interior of the calibration shall remove that calibration level for all analytes. Removal of calibration level from the interior of the curve shall not to be used to compensate for lack of maintenance or repair to the instrument.

7.1.1.e.iii The laboratory shall adjust the LOQ and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.

7.1.1.e.iv The laboratory shall ensure that the remaining initial calibration levels are sufficient to meet the minimum requirements for the number of initial calibration levels as mandated by this standard, the method, and/or regulatory requirements.

7.1.1.e.v The laboratory may replace a calibration level provided that:

7.1.1.e.v.a the laboratory analyzes the replacement standard within 24 hours of the original calibration standard analysis for that particular calibration level;

7.1.1.e.v.b the laboratory replaces all analytes of the replacement calibration standard if a level within the interior of the calibration is replaced; and

7.1.1.e.v.c the laboratory shall limit the replacement to one calibration level.

7.1.1.e.vi The laboratory shall record a technically valid reason for either removal or replacement of any interior calibration level.

7.1.1.f For regression or average response/calibration factor calibrations, the minimum number of non-zero calibration standards shall be as specified in the table below:

Type of Calibration Curve	Minimum Number of Calibration Standards[b,c]
Threshold Testing[a]	1
Average Response	5
Linear Fit	5
Quadratic Fit	6

[a]The initial one-point calibration shall be at the threshold level provided by the customer and results shall be reported qualitatively with uncertainty, and in compliance with a decision rule.

[b]Fewer calibration standards may be used only if equipment firmware or software cannot accommodate the specified

number of standards. Records detailing that limitation shall be maintained by the laboratory.

[c]Ion-selective electrode analyses (e.g., pH, ammonia) are not covered by this table. The laboratory shall use the minimum number of standards as recommended or required in the reference method, or manufacturer's instructions.

7.1.1.g The lowest non-zero calibration standard shall be at or below the lowest concentration for which quantitative data are to be reported without qualification.

7.1.1.h The highest calibration standard shall be at or above the highest concentration for which quantitative data from undiluted samples are reported without qualification. When the undiluted results exceed the calibration range, the laboratory shall dilute and reanalyze the sample to bring results uncorrected for dilution within the calibration range, if sufficient sample material is available. If results are outside the calibration range and reanalysis is not possible, report results with appropriate data qualifiers.

7.1.1.h.i For methods utilizing inductively coupled plasma analysis, the laboratory may report a sample result with a response above the calibration range if the laboratory analyzes and passes (within 10% of the true value) a high-level check standard that exceeds the sample concentration but is within the linear dynamic range (provided the high-level check standard is analyzed in the same manner as the sample and evaluated with the same calibration).

7.1.1.i Sample results shall be quantitated from the initial calibration and may not be quantitated from any continuing calibration verification unless otherwise required by regulation, method, or program.

7.1.1.j Criteria for the acceptance of an initial calibration shall be documented (e.g., correlation coefficient or relative standard deviation).

7.1.1.k The laboratory shall use and document a measure of relative error in the calibration.

7.1.1.k.i For calibrations evaluated using an average response factor, the determination of the relative standard deviation (%RSD) is the measure of the relative error.

7.1.1.k.ii For calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either:

7.1.1.k.ii.a Measurement of the Relative Error (%RE)

Relative error is calculated using the following equation:

$$\%RE = \frac{x' - x}{x} \times 100$$

$x =$ True value for the calibration standard

x' = Measured concentration of calibration standard

This calculation shall be performed for two calibration standards: the standard at or near the mid-range of the initial calibration and a standard less than or equal to the LOQ.

The Relative Error at both levels shall meet the criteria specified in the method. If no criterion for the lowest calibration level is specified in the method, the criterion and the procedure for deriving the criterion shall be specified in the laboratory procedure; or

7.1.1.k.ii.b Measurement of the Relative Standard Error (%RSE)

Relative Standard Error is calculated using the following equation:

$$\%RSE = 100 \times \sqrt{\sum_{i=1}^{n}\left[\frac{x_i' - x_i}{x_i}\right]^2 \div (n-p)}$$

x_i = True value of the calibration standard

x_i' = Measured concentration of calibration standard

p = Number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3)

n = Number of calibration points

The %RSE shall meet the criterion specified in the method. If no criterion is specified in the method, the maximum allowable %RSE shall be numerically identical to the requirement for %RSD in the method. If there is no specification for %RSE or %RSD in the method, then the %RSE shall be specified in the laboratory procedure.

7.1.1.l When procedures are employed that specify calibration with a single calibration standard and a zero point (blank or zero, however specified by the procedure), the following shall occur:

7.1.1.l.i The zero point and single calibration standard within the linear range shall be analyzed at least daily and used to establish the slope of the calibration.

7.1.1.l.ii To verify adequate sensitivity a standard shall be analyzed at or below the lowest concentration for which quantitative data are to be reported without qualification. This standard shall be analyzed before sample analysis with each calibration and shall meet the recovery acceptance criteria at the LOQ established by the method or in Appendix B, whichever is more stringent.

7.1.1.m For analysis of Aroclors which use a linear through origin model (or average response factor) the laboratory shall perform an initial multi-level calibration for a

subset of Aroclors (e.g., a mixture of 1016/1260) and use a one-point initial calibration for pattern recognition for the remaining Aroclors. If one of the remaining Aroclors is identified, a multi-level calibration shall be performed for the specific Aroclor detected, and the sample extract reanalyzed using the multi-level calibration for quantitation.

7.1.1.n If calibrations for reported analytes are not performed with CRM specifically identified as such in an accompanying Certificate of Analysis from a RMP accredited to ISO 17034 or SRM from a NMI, then the laboratory shall use standards from an authoritative source and verify all initial calibrations with a standard from an authoritative, independent, second source. The use of a calibration material from a second lot obtained from the same manufacturer, independently prepared from different source materials, is acceptable for use as a second-source standard. The concentration of the second-source standard calibration verification standard shall be at the mid-range or lower. The calibration verification shall meet the acceptance criteria of the reference method, or if not specified in the reference method, the acceptance criteria for continuing calibration verification shall be used.

7.1.1.o The laboratory shall explain in the case narrative when calibrations are not performed with a CRM or SRM and an initial calibration verification using a second-source standard is required.

7.1.1.p For those methods where reporting non-detected analytes based on successful completion of a sensitivity check is allowed (like threshold testing but only for non-detects), the requirements of this standard do not prohibit the practice.

7.1.1.q Surrogates shall be associated with an acceptable calibration. The laboratory may quantitate surrogates using a single-level calibration. The calibration of the surrogates shall meet the requirements of the analytes, or for a single-level calibration the %RSD for each surrogate shall be less than or equal to 20%.

7.1.2 Continuing Calibration Verification

The validity of the initial calibration shall be verified before sample analyses by a calibration verification with each analytical batch. The following items are essential elements of continuing calibration verification.

7.1.2.a The details of the continuing calibration verification procedure, calculations, and associated statistics shall be included or referenced in the procedure.

7.1.2.b Calibration shall be verified for each compound, element, or other discrete chemical species, except for multi-component analytes such as Aroclors, chlordane, total petroleum hydrocarbons, or toxaphene, where a representative chemical, related substance or mixture may be used.

7.1.2.c The concentration of the continuing calibration verification sample (CCV) shall be greater than or equal to the low calibration standard and less than or equal to the mid-range.

7.1.2.d Instrument calibration verification shall be performed at the beginning and end of

each analytical batch, and at the frequency defined in the method except:

7.1.2.d.i a second source initial calibration verification that passes the continuing calibration verification criteria may be used in place of CCV;

7.1.2.d.ii a LCS may be used in place of a CCV (but not as a replacement for a failing CCV) for methods where the calibration goes through the same process as the LCS (using the continuing calibration verification acceptance criteria).

7.1.2.e Sufficient raw data records shall be retained to permit reconstruction of the calibration verification (e.g., method, unique instrument identification, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations). Continuing calibration verification records shall explicitly connect the continuing calibration verification data to the initial calibration.

7.1.2.f Criteria for the acceptance of a continuing calibration verification shall be established. If the continuing calibration verification results obtained are outside the established acceptance criteria, the following steps shall be taken:

7.1.2.f.i If a cause for the calibration verification failure is identified that impacts only the CCV (e.g., a missed autosampler injection), then analysis may proceed if a second CCV is analyzed immediately (within one hour and no samples analyzed) and the result is within acceptance criteria (i.e., passing). Samples analyzed previously shall be considered valid if bracketed by a passing CCV. The laboratory shall maintain records of the cause for the failure of the first calibration verification result.

7.1.2.f.ii If a cause for the calibration verification failure is not isolated to the CCV or not identified, then the laboratory shall implement its nonconforming work procedure and repeat the CCV and all associated samples since the last successful CCV.

7.1.2.f.iii Qualifying data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory shall notify the customer before reporting data associated with a failed CCV.

7.1.2.f.iv Data associated with an unacceptable CCV shall be qualified if reported but shall not be reported if prohibited by the customer, a regulatory program or regulation.

7.1.2.f.iv.a When the CCV acceptance criteria are exceeded high (i.e., high bias) and there are associated samples with analytes that are non-detects, then those non-detect results may be reported with a data qualifier and sample reanalysis is not required.

7.2 Quality Control

The laboratory shall have QC procedures for monitoring the validity of environmental

tests undertaken as specified in this Section.

7.2.1 Negative Control – Method Performance: Method Blank

7.2.1.a The method blank is used to assess the samples in the preparation batch for possible contamination during the preparation and processing steps.

7.2.1.b The method blank shall be processed along with and under the same conditions as the associated samples to include all steps.

7.2.1.c Procedures shall be in place to determine if a method blank is contaminated.

7.2.1.d Any affected samples associated with a contaminated method blank shall be reprocessed for analysis or the results reported with an appropriate data qualifier.

7.2.1.e The method blank shall be analyzed at a minimum of one per preparation batch.

7.2.1.f When no separate preparation method is used (e.g., volatiles in water), the preparation 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, not including method blanks, LCS, matrix spikes and matrix duplicates.

7.2.1.g The method blank shall consist of a quality system matrix similar to the associated samples and known to be free of analytes of interest. For analysis of metals in solids, materials such as washed sand or non-reactive beads are acceptable as a matrix.

7.2.1.h Method blanks are not applicable for certain analyses (e.g., pH, conductivity, flash point, and temperature).

7.2.1.i For chromatographic analyses, when samples that are extracted together are analyzed on separate instruments or in separate analytical shifts, the method blank associated with those samples (i.e., extracted with the samples) shall be analyzed on at least one of those instruments. A method blank, solvent blank, or instrument blank shall be analyzed on all other instruments on which the set of samples was analyzed to demonstrate the instrument is not contributing contaminants to the samples.

7.2.2 Positive Control – Method Performance: Laboratory Control Sample

7.2.2.a The LCS is used to evaluate the performance of the total measurement system. The LCS shall be processed along with and under the same conditions as the associated samples and include all steps.

7.2.2.b The LCS shall be analyzed at a minimum of one per preparation batch.

7.2.2.c Exceptions are allowed for those analytes for which no spiking solutions are available (e.g., pH, color, odor, temperature, dissolved oxygen, or turbidity).

7.2.2.d In instances for which no separate preparation method is used (e.g., volatiles in

water), the preparation 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, not including method blanks, LCS, matrix spikes, and matrix duplicates.

7.2.2.e The LCS shall be a quality system matrix similar to the associated samples, known to be free of analytes of interest, spiked with known concentrations of analytes. For analysis of metals in solids, materials such as washed sand or non-reactive beads are acceptable as a matrix.

7.2.2.f Alternatively, the LCS may consist of a media containing known and verified concentrations of analytes or a Certified Reference Material. All analyte concentrations shall be within the calibration range of the methods.

7.2.2.g The components to be spiked shall be as specified by the reference method or regulation, or as requested by the customer. In the absence of specified spiking components, the laboratory shall spike as follows:

7.2.2.g.i All reported analytes shall be spiked in the LCS (with the exception of Aroclor analysis, which is spiked per the method). This may require the preparation of multiple LCSs to avoid interferences.

7.2.2.g.ii The concentration of the spiked compounds shall be at or below the mid-range of the calibration if customer-provided concentrations are not specified.

Note: The matrix spike may be used in place of the LCS if the acceptance criteria are as stringent as the LCS acceptance criteria.

7.2.3 Sample-Specific Controls

The laboratory shall document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of quality system matrix specific QC samples and are designed as data quality indicators for a specific sample using the designated method. These controls alone are not used to judge laboratory performance.

Examples of matrix-specific QC include Matrix Spike (MS), Matrix Spike Duplicate (MSD), matrix duplicate (MD), and surrogate spike. The laboratory shall have procedures in place for tracking, managing, and handling matrix-specific QC criteria, including spiking appropriate components at appropriate concentrations, calculating percent recovery (%R), relative percent difference (RPD), and other appropriate statistical measures; and evaluating and reporting results based on performance of the QC samples.

7.2.3.a Matrix Spikes and Matrix Spike Duplicates

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 preparation batch.

7.2.3.a.i Each preparation batch of samples shall contain an associated MS and MSD using the same matrix collected for the specific project if recommended or required by the method or the applicable B-Table. The requirements for MS/MSD are not applicable to all methods. If adequate sample material is not available, then the lack of MS/MSDs shall be noted in the case narrative, and a LCS Duplicate (LCSD) shall be used to determine precision. Additional MS/MSDs may be required by a customer.

7.2.3.a.ii The MS and MSD shall be spiked with all reported analytes (except for Aroclor analysis, which is spiked per the method).

7.2.3.b Matrix Duplicates

7.2.3.b.i Each preparation batch of samples shall contain a MD when precision is not monitored through the analysis of a MS/MSD pair. MDs 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 MD may provide a usable measure of sample homogeneity. It may also provide a measure of precision when target analytes are present. If adequate sample material is not available, then the lack of MD shall be noted in the case narrative, and a LCSD shall be used to determine precision.

7.2.3.b.ii The frequency of the analysis of a MD is as specified by the customer or method.

7.2.3.b.iii Matrix duplicates shall be performed on replicate aliquots of actual samples. The composition is usually not known.

7.2.3.c Surrogate Spikes

7.2.3.c.i Except where the matrix precludes its use or when not commercially available, surrogate compounds shall be added to all samples, standards, and blanks for all appropriate methods.

7.2.3.c.ii Surrogates shall be chosen to reflect the chemistries of the targeted components of the method. Often this is accomplished by using deuterated analogs of select compounds.

7.2.3.c.iii Surrogates shall be added before sample preparation/extraction.

7.2.4 Data Reduction

The laboratory shall have a procedure for data reduction (e.g., linear regression), and records shall be maintained.

7.2.5 Reagent Quality, Water Quality, and Checks

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

7.2.5.b The quality of water sources shall meet recommendations or requirements of the reference method, if applicable. The laboratory shall monitor that the water sources meet the requirements and shall maintain records.

7.2.5.c The laboratory shall verify the concentration of titrants in accordance with written laboratory procedures and shall maintain records.

7.2.5.d The quality (e.g., purity) specifications for all standards and reagents (including water) shall be included or referenced in procedures.

7.2.6 Selectivity

The laboratory shall validate selectivity by following the checks established within the method and/or Appendix B.

7.2.6.a For chromatography methods where confirmation is recommended or required in the reference method or Appendix B, all results greater than the DL shall be confirmed. Confirmation techniques include further analysis using a second column with dissimilar stationary phase, using a second detector type, or by other recognized confirmation techniques. HPLC UV-Diode Array detectors are not considered confirmation for a UV detector.

7.2.6.a.i Confirmation techniques using the same detector type (e.g., second-column confirmation) shall meet the same calibration and QC criteria as the initial or primary analysis.

7.2.6.a.ii The RPD of results from the primary and confirmation technique using the same detector type shall be less than or equal to 40%.

7.2.6.a.iii If using a second column for confirmation, the laboratory shall identify the primary column for each target analyte. If results are reported from the second column due to interference, QC failure, or customer requirements, the laboratory shall discuss in the case narrative.

7.2.6.a.iv If using a mass spectrometer for confirmation, the laboratory shall have a procedure that includes acceptance criteria for selectivity and sensitivity.

7.2.6.a.v When reporting data for methods that require analyte confirmation, customer reporting requirements shall be followed. If customer requirements are not available, the laboratory shall follow the reporting requirements in the method. If the method does not include reporting requirements, the laboratory shall report the results from the primary column or detector, unless there is a scientifically valid and documented reason for not doing so, and concurrence is obtained from the customer.

7.2.6.a.vi The customer shall be notified of any results that are unconfirmed (e.g., confirmation was not performed, or confirmation was obscured by interference), and the results shall be identified in the test report using data qualifiers and described in the case narrative. Analyte presence shall only be reported if both original and confirmation signals are positive or if confirmation signal cannot be discerned from interference.

7.3 Data Acceptance/Rejection Criteria

7.3.1 Negative Control – Method Performance: Method Blank

7.3.1.a While the goal is to have no detectable contaminants, each method blank exhibiting potential contamination shall be evaluated as to the nature of the interference and the effect on the analysis of each sample within the preparation batch. A method blank shall be considered contaminated if:

7.3.1.a.i the concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ or 1/10th the amount measured in any associated sample, whichever is greater; or

7.3.1.a.ii the concentration of any target analyte identified as a common laboratory contaminant in the blank exceeds the LOQ or 1/10th the amount measured in any associated sample, whichever is greater.

7.3.1.b When a method blank is contaminated, the laboratory shall reprepare and analyze the MB and all affected QC and field samples in the associated preparation batch if sufficient sample material is available. Samples are affected if the sample result is greater than the DL and less than 10X the amount detected in the MB. If the affected samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparation batch and explain in the case narrative.

7.3.2 Positive Control – Method Performance: Laboratory Control Sample

7.3.2.a The laboratory shall calculate results of the individual preparation batch LCS in %R or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall include the calculation in the procedure.

7.3.2.b The laboratory shall evaluate the LCS against acceptance criteria provided by the customer.

7.3.2.c In the absence of customer-provided LCS acceptance criteria, the laboratory shall evaluate the LCS against its laboratory-developed acceptance criteria. The laboratory shall develop processes or procedures to incorporate these limits.

7.3.2.d When LCS results are outside of acceptance criteria, the laboratory shall reprepare and analyze the LCS and all affected QC and field samples in the associated preparation batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparation batch and explain in the case narrative.

7.3.2.d.i When the LCS acceptance criteria are exceeded high (i.e., high bias) and there are associated samples with analytes that are non-detects, then those non-detect results may be reported with a data qualifier and sample repreparation and analysis is not required.

7.3.2.e Regardless of which limits are used for LCS evaluation, the laboratory shall develop acceptance criteria for all analytes on its scope of accreditation that:

7.3.2.e.i are statistically derived based on the laboratory's historical data, using scientifically valid and documented procedures;

7.3.2.e.ii meet the limits within the reference method, if available;

7.3.2.e.iii are updated on at least an annual basis or as recommended or required in the reference method, whichever is more frequent, and re-established after major changes in the measurement system (e.g., new instrumentation);

7.3.2.e.iv are based on at least 30 data points generated under the same measurement system;

7.3.2.e.v do not exclude failed LCS recovery data and statistical outliers from the calculation, unless there is a scientifically valid and documented reason (e.g., incorrectly made standard, instrument malfunction);

7.3.2.e.vi are not outside ± 3 times the standard deviation of the mean LCS recovery; and

7.3.2.e.vii are used for trend analysis, and preparation batch control if applicable.

7.3.2.f Control charts or data analysis software shall be maintained and used to detect trends and prevent out-of-control conditions. Control limits shall be monitored at least quarterly for shifts in mean recovery, changes in standard deviation, and development of trends. The laboratory may choose representative compounds for control charts for the purpose of trend analysis. The basis for selecting representative compounds shall be documented and shall be scientifically valid.

7.3.2.g The laboratory shall review control charts at a specified frequency for out-of-control conditions and initiate corrective actions when appropriate. Data analysis software may also be used for the statistical evaluation of data for trends.

7.3.2.h The laboratory shall use its laboratory-developed LCS control limits for the purpose of trend analysis and may use those control limits as a component in estimating measurement uncertainty.

7.3.2.i Marginal Exceedances: If many analytes are in the LCS, it becomes statistically likely that a few will be outside acceptance criteria. This may not indicate that the system is out of control, therefore corrective action may not be necessary. This ME approach is relevant for methods with long lists of analytes. It shall not apply to target analyte lists with fewer than 11 analytes.

7.3.2.i.i The laboratory shall have a procedure for monitoring the application of ME allowances to the LCS.

7.3.2.i.ii Sporadic marginal exceedances are allowed for those analytes outside the 3 standard deviation control limits but still within 4 standard deviations.

7.3.2.i.iii The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS acceptance criteria than is allowed, or if any one analyte exceeds four standard deviations, the LCS fails, and corrective action is necessary.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

7.3.2.i.iv The same analyte exceeding the LCS acceptance criteria in two out of three consecutive preparation batches is indicative of non-random behavior. If this occurs, the laboratory shall implement its nonconforming work procedure and the LCS and associated samples shall be reprepared and analyzed.

7.3.2.i.v Sporadic MEs are not allowed for target analytes (i.e., chemicals of concern identified by the customer) without customer approval.

7.3.2.j Laboratory Control Sample Duplicate

7.3.2.j.i If sufficient sample material was not available for a MS/MSD or MD and a LCSD was used to measure precision of analytical results, the laboratory shall evaluate the LCSD recovery and RPD using acceptance criteria provided by the customer, or if customer requirements are not provided, using the appropriate Appendix B Table. If these acceptance criteria are not available, the laboratory shall evaluate the LCSD recovery and RPD using the reference method, or if not specified in the reference method, using laboratory-developed limits.

7.3.2.j.ii If LCSD results are outside of acceptance criteria, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparation batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparation batch and explain in the case narrative.

7.3.3 Sample-Specific Controls

7.3.3.a Matrix Spike; Matrix Spike Duplicates

The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), RPD, or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall include in a procedure the calculation for %R, RPD or other statistical treatment used.

7.3.3.a.i The laboratory shall evaluate MS/MSD recoveries using the same acceptance criteria used for the LCS.

7.3.3.a.ii The laboratory shall evaluate the MS/MSD RPD against customer requirements, or if not specified, with the appropriate Appendix B Table for the technology. If these acceptance criteria are not available, the laboratory shall evaluate the MS/MSD RPD to the reference method, or if not specified, with its laboratory-developed limits.

7.3.3.a.iii If MS/MSD results or MS/MSD RPD are outside the acceptance criteria, and an assignable cause isolated to only the MS or MSD is identified, reanalyze the MS or MSD or reprepare and analyze the MS or MSD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

7.3.3.b Matrix Duplicates

The results from matrix duplicates are primarily designed to assess the homogeneity of the particular sample chosen. If that sample is homogenous, it may also describe the precision of analytical results in a given matrix. These may be expressed as RPD or another statistical treatment (e.g., absolute differences).

7.3.3.b.i The laboratory shall include in a procedure the calculation for RPD or other statistical treatments.

7.3.3.b.ii The laboratory shall evaluate the MD RPD against customer requirements, or if not specified, with the appropriate Appendix B Table. If these acceptance criteria are not available, the laboratory shall evaluate the MD RPD to the reference method, or if not specified, with its laboratory-developed limits.

7.3.3.b.iii If the MD RPD is outside of acceptance criteria, and an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

7.3.3.c Surrogate Spikes

7.3.3.c.i The laboratory shall evaluate the surrogate spike recoveries using acceptance criteria provided by the customer, or if customer requirements are not provided, using the appropriate Appendix C limits. If these acceptance criteria

are not available, the laboratory shall evaluate surrogate spike recoveries using acceptance criteria developed from LCS recovery data.

7.3.3.c.ii If surrogate results are outside the acceptance criteria, and an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. Otherwise, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

7.4 Sample Handling

7.4.1 The laboratory shall implement procedures for checking sample preservation using readily available techniques, such as pH or chlorine, before, or during sample preparation or analysis. For volatile organic analysis, chemical preservation shall be checked after analysis.

7.4.2 A storage blank shall be stored with all volatile organic samples, regardless of suspected concentration levels. Storage blanks shall be used to determine if cross-contamination may have occurred. If cross-contamination greater than ½ LOQ (Methylene chloride, Acetone, 2-Butanone, greater than LOQ) is found in the storage blank, the laboratory shall implement the nonconforming work procedure. The laboratory shall have procedures and acceptance criteria for evaluating storage blanks appropriate to the types of samples being stored. The storage blanks shall be stored in the same manner as the customer samples. The storage blanks shall be analyzed every 14 days at a minimum.

Module 5

Quality Systems for Microbiological Testing

1.0 Introduction

This module provides specific requirements for laboratories seeking accreditation for microbiological testing. Laboratories seeking accreditation for microbiological testing shall also meet the requirements presented in Module 1 and 2 of this standard. Microbiological testing refers to and includes the detection, isolation, enumeration, or identification of microorganisms (and/or their metabolites), or determination of the presence or absence of growth in materials and media. The evaluation of laboratories for this discipline is in conjunction with a quality system as specified in the general requirements module. Adherence to those quality system requirements and all quality control (QC) procedures specified in this module will ensure that microbiological test results are fit for the intended use.

2.0 Scope

This module provides quality assurance (QA) and quality control (QC) requirements for laboratories seeking to obtain and maintain accreditation for microbiological testing methods. This module is not intended to relieve additional quality control requirements that are specified by method, regulation, or customer.

3.0 Terms and Definitions

Definitions are considered requirements. Definitions are included in Module 2, Section 3.0. Additional definitions related to this module are listed below.

Source Water: When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.

4.0 Method Selection

The requirements in the Module 2 Section on “Selection, Verification and Validation of Methods” apply.

5.0 Method Validation

Prior to acceptance and institution of any method for which data will be reported, all methods shall be validated. For all methods except reference methods, validation shall meet the requirements in the Module 2 section on “Selection, Verification and Validation of Methods” as well as all criteria in this module.

5.1 Accuracy

Use at least one known pure positive reference culture at the anticipated environmental conditions and compare the method results to that of a reference method.

5.2 Precision

Perform at least 10 replicate analyses with both the proposed and reference method, using a sample containing the target microorganisms of choice. The results shall show that the precision of the proposed method is statistically equivalent or better than that of the reference method.

5.3 Selectivity (sensitivity)

Verify all responses in at least 10 samples using mixed cultures that include the target organism(s) and at varying concentrations (microbial identification testing or equivalent processes may be used). Calculate the number of false positive and false negative results.

6.0 Demonstration of Capability (DOC)

6.1 General

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

6.1.2 Thereafter, ongoing DOC, shall be performed and recorded at least every 12 months.

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

6.2 Initial DOC

Each individual shall successfully perform an initial DOC prior to using any method, any time there is a change in instrument type or method that could potentially affect the precision and bias, sensitivity, or selectivity of the output, or any time that a method has not been performed by the individual in a 12-month period.

The laboratory shall have a procedure for performing an initial DOC.

6.2.1 The laboratory shall maintain records of each initial DOC in a manner such that the following information is readily available for each individual:

6.2.1.a individual(s) involved in preparation and/or analysis;

6.2.1.b matrix;

6.2.1.c organism(s);

6.2.1.d identification of method(s) performed;

6.2.1.e identification of laboratory-specific procedures used for analysis, including revision number;

6.2.1.f date(s) of analysis; and

6.2.1.g summary of analyses, including information outlined in Section 6.2.2.c.

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

6.2.2.a The target organism(s) shall be diluted in a volume of sterile, quality system matrix (a sample in which no target organisms or interferences are present at concentrations that will impact the results of a specific method). When required by method, the diluent shall be sterile buffered water and/or sterile peptone water unless specified by the manufacturer. Prepare at least four aliquots at the concentration specified, or if unspecified, to the countable range for plate methods or working range for most probable number (MPN) type methods.

6.2.2.b At least four aliquots shall be prepared and analyzed concurrently according to the method.

6.2.2.c Using all the results, convert these results to logarithmic values, then calculate the mean recovery and standard deviation of the log converted results in the appropriate reporting units for each organism of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence, the laboratory shall assess performance against established and documented criteria.

6.2.2.d For qualitative tests, acceptable performance in a blind study, either internally or externally generated, may be used to meet this standard, provided that the study consists of a minimum of a blank, a negative culture, and a positive culture for each target organism.

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

6.2.2.f When one or more of the tested parameters fail at least one of the acceptance criteria, the individual shall proceed according to i) or ii) below.

6.2.2.f.i Locate and correct the source of the problem and repeat the initial DOC for all parameters of interest beginning with b) above.

6.2.2.f.ii Repeat the initial DOC for all parameters that failed to meet criteria.

6.2.2.g Repeat failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all organisms of interest beginning with b) above.

6.3 Ongoing DOC

6.3.1 The laboratory shall have a documented procedure describing ongoing DOC that includes how the laboratory will identify data associated with ongoing DOCs. The individual shall demonstrate ongoing capability by routinely meeting the QC requirements of the method, laboratory procedure, customer specifications, and/or this standard. If the method has not been performed by the individual in a 12-month period, an initial DOC shall be performed prior to performing analysis. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

6.3.2 This ongoing demonstration may include one of the following:

6.3.2.a another initial DOC;

6.3.2.b analysis of one sample of clean matrix that is fortified with a known quantity of the target organism, with results meeting the laboratory acceptance criteria for accuracy and, where applicable to the testing technique, also meeting the observational details expected for the presumptive, confirmed and completed phases defined in the method;

6.3.2.c analysis of one positive sample in duplicate for each target organism and test, with results meeting the laboratory acceptance criterion for precision;

6.3.2.d acceptable results for a blind proficiency test sample or sample set, as required by the program, for target organisms in each field of accreditation;

6.3.2.e performance of an alternate adequate procedure for the field of accreditation, the procedure and acceptance criteria being documented in the laboratory's quality system;

6.3.2.f following a procedure for reviewing records of QC samples meeting the QC requirements of the method, laboratory procedure, customer requirements, and/or this standard. A review of these records may be used to identify patterns and determine if implementation of the nonconforming work process and/or retraining is necessary; or

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

7.0 Technical Requirements

7.1 Calibration

7.1.1 The laboratory shall have documented procedures for calibration, verification, and

QC of auxiliary equipment including conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments. These procedures shall refer to applicable reference methods.

7.1.2 For instruments that are continuous monitors, such as in-line specific conductance meters:

7.1.2.a the laboratory shall verify calibration at least once a month and retain records; and

7.1.2.b an initial calibration shall be performed if a calibration verification is unacceptable, or when the instrument is being returned to service after having been taken off-line.

7.2 Continuous Calibration

Reserved for specific procedures.

7.3 Quality Control

7.3.1 Quality and Sterility of Standards, Reagents, Materials, and Media

The laboratory shall demonstrate that the quality of the reagents and media used is appropriate for the test concerned including, but not limited to, test conditions and incubation times. Records shall be maintained.

7.3.1.a Sterility checks – All materials and supplies that are needed to process the sample and are required to be sterile prior to use (whether sterilized in the laboratory or purchased as sterilized) shall be checked by the laboratory once per purchased or prepared lot using non-selective growth media as appropriate. Certificates of analysis provided by vendors shall be verified by the laboratory and retained. These checks shall include, but are not limited to:

7.3.1.a.i The laboratory shall perform a sterility check for each lot of prepared, ready-to-use, media and on each batch of media prepared in the laboratory.

7.3.1.a.i.a For chromo/fluorogenic media: add media to sterile deionized water and incubate at the appropriate temperature and time.

7.3.1.a.i.b For all other media, incubate uninoculated at the appropriate temperature and time. Where media are made as concentrates (e.g., double strength), then the medium shall be diluted to working strength with sterile deionized water before testing.

7.3.1.a.ii The laboratory shall perform a sterility check on one funnel per lot of pre-sterilized single use funnels using non-selective growth media. The laboratory shall perform a sterility check on one funnel per batch of laboratory-sterilized funnels, using non- selective growth media perform a sterility check for each lot of prepared, ready-to-use, media and on each batch of media prepared in the laboratory.

7.3.1.a.iii The laboratory shall perform a sterility check on at least one container for each lot of purchased, pre-sterilized sample containers with non-selective growth media. The laboratory shall perform a sterility check on one container/object per sterilization batch sterilized in the laboratory with nonselective growth media.

7.3.1.a.iv The laboratory shall perform a sterility check on each batch of dilution water prepared in the laboratory and on each lot of pre-prepared, ready-to-use dilution water with non-selective growth media. The concentration of the non-selective growth media shall be single strength after the addition of dilution water.

7.3.1.a.v The laboratory shall perform a sterility check on at least one filter from each new lot of membrane filters with nonselective growth media.

7.3.1.b Media – Culture media may be prepared from commercial dehydrated powders or may be purchased ready-to-use.

7.3.1.b.i All media shall be tested for performance (e.g., for selectivity, sensitivity, sterility, growth promotion, and growth inhibition). These tests shall be performed at a minimum with first use.

7.3.1.b.ii The laboratory shall use all media within the expiration date or shelf-life provided by the manufacturer.

7.3.1.b.iii The laboratory shall use all laboratory-prepared media within the holding time limits specified in the accredited method.

7.3.1.b.iv The laboratory shall have detailed testing criteria information defined in the laboratory's methods, procedures, or similar documentation.

7.3.1.c The laboratory shall use reagents, media, and commercial dehydrated powders within the shelf-life of the product and shall maintain records.

7.3.1.d Reagent Water

7.3.1.d.i The laboratory shall monitor the quality of the reagent water used in the laboratory, which will come into contact with test organisms and is used in preparation of media, solutions, and buffers, for bactericidal and inhibitory substances. This water shall be distilled water, deionized water, or reverse-osmosis-produced water.

7.3.1.d.ii The laboratory shall monitor the quality of the water for disinfectant residual, specific conductance, total organic carbon, and heterotrophic bacteria plate count monthly (when in use), when maintenance is performed on the water treatment system, or at startup after a period of disuse longer than one month. Analysis may be performed by another certified laboratory.

7.3.1.d.iii The laboratory shall monitor the quality of the water for metals (Cd, Cr, Cu, Ni, Pb, and Zn) and the Bacteriological Water Quality Test (to determine presence of toxic agents or growth promoting substances) annually. An

exception to performing the Bacteriological Water Quality Test shall be given to laboratories that can supply records to show that its water source meets the criteria, as specified by the method, for High Quality (Type I) or Medium Quality (Type II) reagent water. Analysis may be performed by another certified laboratory.

7.3.1.d.iv Results of the above analyses shall meet the specifications of the required method.

7.3.1.d.v Reagent water purchased from an outside source and used for the preparations of media, solutions and buffers shall meet the criteria specified in items ii) and iii) above. The laboratory shall maintain records of this information.

7.3.1.d.vi Reagent water that has been opened for longer than the testing intervals specified in items i) through iv), or in the accredited method, shall either be re-tested or discarded.

7.3.1.e Dilution water, however used, includes buffer water and/or peptone water. The laboratory shall monitor the quality of the dilution water for sterility, pH and volume once per lot or batch whether purchased or prepared by the laboratory.

7.3.1.f Records for media and reagents prepared in the laboratory shall include date of preparation, preparer's initials, type, manufacturer, lot number, final pH, expiration date, and the amount of reagents used. Records for media purchased pre-prepared, ready-to-use (including reagent water purchased from outside sources) shall include manufacturer, lot number, type of media received, date of receipt, expiration date of the media, and pH of the media. Records shall be maintained.

7.3.2 Method Blanks

The laboratory shall demonstrate that the filtration equipment and filters, sample containers, media, and reagents have not been contaminated through improper handling or preparation, or environmental exposure.

7.3.2.a For filtration technique, the laboratory shall conduct method blanks per the analytical method. At a minimum, the filtration series shall include a beginning and ending blank. The filtration series may include single or multiple filtration units, which have been sterilized prior to beginning the series.

7.3.2.b The filtration series is considered ended when more than 30 minutes elapses between successive filtrations. During a filtration series, filter funnels shall be rinsed with three 20-30 ml portions of sterile rinse water after each sample filtration. In addition, laboratories shall insert a method blank after every 10 samples or sanitize filtration units by UV light (254 nm) after sample filtration.

7.3.2.c For pour plate technique, method 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.

7.3.3 Test Variability/Reproducibility

For methods that specify counts (i.e., cfu/100 mL or MPN/100 mL), such as membrane filter, plated media or other methods which specify a quantitative result, 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 results on the same sample. Counts shall be within ten percent (10%) difference to be acceptable. In a laboratory with only one microbiology analyst, the same sample shall be counted twice by the analyst, with no more than a five percent (5%) difference between the counts.

7.3.4 Sample-Specific Controls (where applicable)

7.3.4.a The laboratory shall perform matrix spikes per method requirements.

7.3.4.b The laboratory shall perform sample matrix duplicates per method requirements.

7.3.5 Data Reduction

The calculations, data reduction and statistical interpretations specified by each method shall be identified and followed.

7.3.6 Selectivity

7.3.6.a All growth and recovery media shall be checked to assure that the target organism(s) respond in an acceptable and predictable manner once per lot or batch.

7.3.6.b To ensure that analysis results are accurate, target organism identity shall be verified as specified in the method (e.g., by use of the completed test or by use of secondary verification tests such as a catalase test; or by the use of a selective medium such as Brilliant Green Lactose Bile Broth, Escherichia coli (EC) broth, or EC + 4-methylumbelliferyl-β-D-glucuronide broth).

7.3.6.c To ensure identity and traceability, reference cultures used for positive and negative controls shall be obtained from a recognized national collection, organization, or manufacturer recognized by the accreditation body. Microorganisms may be single-use preparations or cultures maintained for their intended use by documented procedures that demonstrate the continued purity and viability of the organism.

7.3.6.c.i Reference cultures may be revived (if freeze-dried) or transferred from slants and sub-cultured once to provide reference stocks. The reference stocks shall be preserved by a technique that maintains the characteristics of the strains. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they shall not be refrozen and re-used.

7.3.6.c.ii Working stocks shall not be sequentially cultured more than five times and shall not be sub-cultured to replace reference stocks.

7.3.6.d Culture Controls (i.e., working cultures)

7.3.6.d.i Negative Culture Controls

7.3.6.d.i.a Negative culture controls demonstrate that the medium does not support the growth of non-target organisms or does not exhibit the typical positive reaction of the target organism(s).

7.3.6.d.i.b Each pre-prepared, ready-to-use lot of selective medium (including chromo/fluorogenic 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.

7.3.6.d.ii Positive Culture Controls

7.3.6.d.ii.a Positive culture controls demonstrate that the medium can support the growth of the target organism(s), and that the medium produces the specified or expected reaction to the target organism(s).

7.3.6.d.ii.b Each pre-prepared, ready-to-use lot of medium (including chromo/fluorogenic reagent) and each batch of medium prepared in the laboratory shall be tested with at least one or more known pure positive culture controls (i.e., target organism) as appropriate to the method and that produce typical results based on the method. This shall be done prior to first use of the medium.

7.3.7 Selectivity

7.3.7.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.

7.3.7.b Laboratory Equipment

7.3.7.b.i Temperature Measuring Devices

The laboratory shall use temperature measuring devices such as liquid-in-glass thermometers, thermocouples, or platinum-resistance thermometers to assess equipment temperatures. Records shall be maintained. The temperature measuring devices shall be appropriate quality to meet specification(s) in the method.

The graduation and range of the temperature measuring devices shall be appropriate for the required accuracy of the measurement. Temperature measuring devices shall be verified to national or international standards for temperature. Verification shall be performed at least annually. This verification may be accomplished by a single point provided that it represents the method mandated temperature and use conditions.

7.3.7.b.ii Sterilization Equipment

7.3.7.b.ii.a Autoclaves

7.3.7.b.ii.a.1 The laboratory shall evaluate the performance of each autoclave initially by establishing its functional properties and performance, for example, heat distribution characteristics with respect to typical uses. Autoclaves shall meet specified temperature tolerances. Pressure cookers shall not be used for sterilization of growth media.

7.3.7.b.ii.a.2 The laboratory shall demonstrate proper sterilization temperature by use of a continuous temperature recording device or by use of a maximum registering thermometer with every cycle. The laboratory shall, at least once during each month that the autoclave is used, demonstrate the effective sterilization through the use of appropriate biological indicators. The selected biological indicator shall be effective at the sterilization temperature and time needed to sterilize lactose-based media. The laboratory shall use temperature-sensitive tape with the contents of each autoclave run to indicate that the autoclave contents have been processed.

7.3.7.b.ii.a.3 The laboratory shall maintain records of autoclave operations for every cycle. Records shall include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out), and analyst's initials.

7.3.7.b.ii.a.4 Autoclave maintenance, internally or by service contract, shall be performed annually, and shall include a pressure check and verification of temperature device. Records of the maintenance shall be maintained. When it has been determined that the autoclave has no leaks, pressure checks can be made using the formula PV = nRT.

7.3.7.b.ii.a.5 The laboratory shall check the autoclave mechanical timing device quarterly against a stopwatch and record the actual time elapsed.

7.3.7.b.ii.b Ovens

The laboratory shall check ovens used for sterilization for sterilization effectiveness monthly with appropriate biological indicators. The laboratory shall maintain records for each cycle that include date, cycle time, temperature, contents, and analyst's initials. The laboratory shall use temperature sensitive tape with the contents of each run to indicate that the contents have been processed.

7.3.7.b.iii Volumetric Equipment

The laboratory shall verify equipment used for measuring volume as follows:

7.3.7.b.iii.a Equipment with movable parts, such as automatic dispensers, dispensers/ diluters, and mechanical hand pipettes, shall be verified for accuracy quarterly.

7.3.7.b.iii.b Equipment, such as filter funnels, bottles, non-Class A glassware, and other containers with volumetric markings (including sample analysis vessels), shall be verified once per lot prior to first use.

7.3.7.b.iii.c The volume of the disposable volumetric equipment, such as sample bottles and disposable pipettes, shall be checked once per lot.

7.3.7.b.iii.d Verification of volume shall be considered acceptable if the accuracy is within 2.5% of expected volume. This verification may be volumetric as compared to Class A or gravimetric.

7.3.7.b.iv UV Instruments

The laboratory shall evaluate UV instruments used for sanitization quarterly for effectiveness with an appropriate UV light meter, by plate count, agar spread plates, or other methods providing equivalent results, such as UV-cide strips. Replace bulbs if output is less than 70% of original for light tests or if count reduction is less than 99% for a plate containing 200 to 300 organisms.

7.3.7.b.v Incubators, Water Baths

7.3.7.b.v.a The laboratory shall establish the uniformity of temperature distribution and equilibrium conditions in incubators and water baths prior to first use after installation or service. The equilibrium check shall include time required after test sample addition to re-establish equilibrium conditions under full capacity load appropriate for the intended use.

7.3.7.b.v.b During periods when samples are under test, the laboratory shall have a system in place to monitor and record the temperature of incubators and water baths twice daily, at least four hours apart. “Under test” is defined as the time period that the sample is in the incubation phase of the method. Data loggers, continuous temperature monitoring devices, or other temperature monitoring equipment may be used as long as they can be calibrated. Records shall be maintained.

Note: There is no intent to take the temperature of incubation units during periods when there are no samples under test.

7.3.7.b.vi Labware (Glass and Plasticware)

7.3.7.b.vi.a The laboratory shall have a documented procedure for washing labware, if applicable. Detergents designed for laboratory use shall be used.

7.3.7.b.vi.b Glassware shall be made of borosilicate or other non-corrosive material, free of chips and cracks, and shall have readable measurement marks.

7.3.7.b.vi.c Labware that is washed and reused shall be tested for possible presence of residues that may inhibit or promote growth of microorganisms by performing the Inhibitory Residue Test initially and each time the laboratory changes the detergent formulation or washing procedures.

7.3.7.b.vi.d Washed labware shall be tested at least once daily, each day of washing, for possible acid or alkaline residue by testing at least one piece of labware with a suitable pH indicator such as bromothymol blue. Records of tests shall be maintained.

7.4 Data Acceptance/Rejection Criteria

Methods criteria and evaluation methods shall be used.

7.5 Sampling Handling

Receipt of samples shall comply with Module 2, as well as:

7.5.1 Microbiological samples from known chlorinated sources (such as wastewater effluent), unknown sources where disinfectant (e.g., chlorine) usage is suspected (such as a new customer or a new source), and all potable water supplies (including source water) shall be checked for absence of disinfectant residual in the laboratory unless all of the following conditions are met:

7.5.1.a The laboratory can show that the received sample containers are from its laboratory or have been appropriately tested and recorded;

7.5.1.b Sufficient sodium thiosulfate was in each container before sample collection to neutralize at minimum 5 mg/L of chlorine for drinking water and 15 mg/L of chlorine for wastewater samples;

7.5.1.c 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 5 mg/L chlorine or 15 mg/L chlorine as appropriate and the check is recorded; and

7.5.1.d Disinfectant residual is checked in the field and actual concentration is provided with sample submission.

Module 6

Quality Systems for Radiochemical Testing

1.0 Introduction

This module provides specific requirements for laboratories seeking accreditation for radiochemical testing. Laboratories seeking accreditation for radiochemical testing shall also meet the requirements presented in Module 1 and 2 of this standard. These requirements apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. Radiochemical determinations involve chemical separation and subsequent detection of the radioactive emissions of the analyte (or indicative decay progeny) and, if employed, tracer isotopes, often following the chemical separation from the sample matrix.

2.0 Scope

This module provides quality assurance (QA) and quality control (QC) requirements for laboratories seeking to obtain and maintain accreditation for radiochemical testing methods. This module is not intended to relieve additional quality control requirements that are specified by method, regulation, or customer.

This module employs terms, definitions, and requirements from other documents, such as the Safe Drinking Water Act (SDWA), Clean Water Act, or the Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual.

3.0 Terms and Definitions

Definitions are considered requirements. Definitions are included in Module 2, Section 3.0. Additional definitions related to this module are listed below.

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

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

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

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

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

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

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

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

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

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

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

Decision Level: 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 decision level is sometimes called the critical level 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.

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

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

Minimum Detectable Activity: Estimate of the smallest true activity that ensures a specified high confidence, (1–β), of detection above the Decision Level, and a low probability β of false negatives below the Decision Level. For radiometric methods, β is often set at 0.05.

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

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

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

Tracers and Carriers: Tracers and Carriers chemically mimic but do not interfere with the target analyte through radiochemical separations. Isotopic tracers are typically radioactive materials (e.g., Plutonium-242, Strontium-85). They are added to samples to determine the overall chemical yield for the analytical preparation steps.

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

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

Note: Radiochemical results are generally reported in association with the Total Uncertainty or the Counting Uncertainty. Either of these estimates of uncertainty may be reported as the Standard Uncertainty (1 standard deviation) or an Expanded Uncertainty (k-standard deviation, where $k > 1$).

Uncertainty, Measurement: Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand (Guide to the expression of uncertainty in measurement).

Uncertainty, Random: Component of the reported uncertainty shall include, but is not necessarily limited to, the total random counting uncertainty associated with each sample and shall be appropriately propagated when more than one variable is used to determine the result.

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

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

Uncertainty).

3.1 Exclusions and Exceptions

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

4.0 Method Selection

The requirements in Module 2 Section on “Selection, Verification and Validation of Methods” apply.

5.0 Method Validation

5.1 Validation of Methods

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

5.1.2 The laboratory shall validate the method in each quality system matrix for which it is applicable by demonstrating the method’s detection capability, precision, bias, Measurement Uncertainty, and selectivity using the procedures specified in the Detection Capability, Evaluation of Precision and Bias, Measurement Uncertainty, and Evaluation of Selectivity Sections of Module 6.

5.1.3 The laboratory shall perform validation for each method for which documented data are not available to demonstrate that the above requirements are met. For reference methods, published data, if available, may be used to satisfy these requirements.

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

5.1.5 For all methods, the validation shall meet the requirements in the Module 2 Section on “Selection, Verification, and Validation of Methods” as well as all criteria in this module.

5.1.6 The laboratory shall maintain records of the results obtained, the procedure used for

the validation, and a statement as to whether the method is suitable for the intended use.

5.1.7 Validation procedures shall include, whenever available, externally-produced quality control samples obtained from a Reference Material Producer accredited to ISO 17034 or a National Metrology Institute (NMI). When such reference materials cannot be obtained, that laboratory may use materials from a Proficiency Testing Provider accredited to ISO/IEC 17043 or from another authoritative source. The laboratory shall evaluate the results of these analyses to determine its ability to produce acceptable data.

5.2 Detection Capability

5.2.1 The laboratory shall establish the detection capability for each method/matrix/instrumentation combination. Detection Capability may refer to the Decision Level, MDA, or SDWA detection level.

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

5.2.3 The procedure a laboratory uses to determine the detection capability of a method shall comply with the specific requirements of the Minimal Detectable Activity, Decision Level, and Required Detection Limit for Drinking Water Compliance Sections of Module 6.

5.2.4 Method validation records shall include identification of software used for detection capability calculations and the software shall conform to the requirements in Module 2.

5.2.5 Minimal Detectable Activity

The laboratory shall utilize a method that is capable of providing an MDA that is appropriate and relevant for the intended use of the data. The laboratory shall determine MDAs using the procedure specified in mandated methods. If no procedure is specified, the laboratory shall select a procedure that reflects instrument limitations and the intended application of the method.

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

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

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

5.2.5.d Equations to calculate the decision level and the minimum detectable concentration (or activity) shall be included in the analytical procedures or

management system procedures.

5.2.5.e MDA Factors and Conditions

MDAs are determined based on factors and conditions such as instrument settings and matrix type, which influence the measurement. The MDA is used to evaluate the capability of a method relative to the required Decision Level. 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 Decision Levels. If Decision Levels are not achieved, then the cause shall be discussed comprehensively in the case narrative.

5.2.5.f MDA Calculation

The basic MDA calculation shall be based on the concepts developed by L. A. Currie from his paper "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry*, March 1968, Vol. 40, or from Chapter 20 of the MARLAP Manual, Volume III (EPA 402-B-04-001C).

5.2.5.f.i MDA Calculation with a Blank Population

The following general equations derived from the work of L. A. Currie may be used to calculate the MDA: Equations may need to be modified depending on the measurement technique in use.

$$MDA = \frac{3.29 * s_b}{KT_S} + \frac{3}{KT_S}$$

Where:

K = efficiency * $e^{-\lambda t}$ * aliquot fraction * tracer recovery*Yield

T_S = count time of the sample in minutes

s_b = standard deviation of the blank population where the blank population is in net blank counts in count time T_S

5.2.5.f.i.a Use of blank populations for calculation of MDAs requires the selection of an implementation method (e.g., identification of blanks to be used in the population, number of blanks to use in the population, changes in the blank population and limitations on the deletion of blanks).

5.2.5.f.i.b The method of implementation shall not introduce any statistical bias.

5.2.5.f.i.c The appropriate blank subtraction shall be the mean blank value of the blank population.

5.2.5.f.i.d The implementation of blank populations for calculation of MDAs shall be described in detail in a procedure.

5.2.5.f.i.e In the original Currie derivation, a constant factor of 2.71 was used. Since that time, it has been shown and generally accepted that a constant factor of three is more appropriate (Multi Agency Radiation Survey and Site Investigation Manual, Aug. 2000). However, it is acceptable to use a constant of 2.71 in situations where that factor is built into instrument software without an option to use 3. In that case, obtain permission from the customer and discuss the use of 2.71 in the case narrative or document it in procedures available to the customer.

5.2.5.f.ii MDA Calculation without a Blank Population

The following general equations derived from the work of Currie or MARLAP calculations may be used to calculate the MDA.

$$MDA = \frac{3.29 * \sqrt{\frac{b}{T_S} + \frac{b}{T_B}}}{K} + \frac{3}{K * T_S}$$

Where:

K = efficiency * e $^{-\lambda t}$ * aliquot fraction * tracer Recovery*Yield

T_S = count time of the sample in minutes

T_B = count time of the background in minutes

b = background count rate in cpm

Note: The above equation is used when sample and background count times are different. Other equations, where sample and background count times are the same may also be used.

Note: The above equation for MDA has the units of dpm/sample. Any other units will require appropriate conversion.

5.2.5.g MDA Requirements for Elevated Samples:

Samples with elevated activities shall be handled according to the following requirements:

5.2.5.g.i The appropriate sample size shall be determined based on the activity level in the sample. It shall be large enough to generate data, which meet the following criteria:

5.2.5.g.i.a measurement uncertainty shall not be greater than 10% (1 standard deviation) of the sample activity; and

5.2.5.g.i.b the MDA for the analysis shall be a maximum of 10% of the sample activity.

5.2.5.h Sample-specific MDAs shall be calculated and reported. If MDAs are reported as a nominal detection capability of the measurement process, that shall be clearly stated in the data package.

5.2.5.i The definition of the MDA presupposes that an appropriate detection threshold (i.e., the decision level) has already been defined. In the most commonly used equation for the MDA, the alpha probability of 5% used for the decision level is also used for beta probability. The laboratory shall confirm the MDA calculations meet the customer's expectations for alpha and beta probability factors.

5.2.6 Decision Level

5.2.6.a Decision Level Factors and Conditions

Decision levels shall be determined based on sample-specific sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency.

5.2.6.b Decision Level Calculation

The basic decision level calculation shall be based on concepts developed by L. A. Currie, "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry*, March, 1968, Vol. 40, or MARLAP Chapter 20. Decision levels are used as the default detection threshold. The following general equation below may be used to calculate the decision level.

The decision level may either be based on the Combined Standard Uncertainty (CSU) of the blank (preparation/or method/instrument type) or the standard deviation determined from a set of appropriate blanks.

5.2.6.b.i Decision Level Calculation with a Blank Population

When determined from the standard deviation of a set of appropriate blanks, the decision level evaluates the level at which 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 = the decision level in disintegrations per minute per unit volume or weight (dpm/unit);

S_B = the standard deviation of a set of appropriate blanks net count rate after background subtraction for blanks counted for the same length of time as the sample;

R_B = the average blank count rate in counts per minute (cpm);

t = the student t factor for appropriate degrees of freedom and confidence level;

E = the fractional detector efficiency (c/d) for the sample;

R = the fractional chemical yield for the sample;

IDF = the ingrowth or decay factor for the sample; and

W = the weight or volume of the sample.

5.2.6.b.ii Decision Level Calculation without a Blank Population.

Decision levels for samples without a blank population may be determined if based on appropriate L. A. Currie or MARLAP calculations using combined standard uncertainty (CSU).

5.2.7 Required Detection Limit for Drinking Water Compliance

Laboratories performing radiochemical testing of drinking water samples for SDWA compliance monitoring shall meet the requirements of 40 CFR Part 141. These laboratories shall use only approved methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR Part 141. The detection capability shall be expressed in terms of the Decision Level instead of Method Detection Limit.

5.3 Evaluation of Precision and Bias

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

5.3.1 The laboratory shall utilize a method that provides precision and bias data for each of the analytes of interest that is appropriate and relevant for the intended use of the data. Precision and bias shall be characterized across the range of activities that brackets those applicable in samples, including zero activity.

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

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

5.3.4 Where there are no established criteria, the laboratory shall develop acceptance

criteria for precision and bias based on one or more of the following:

5.3.4.a intended use of the data;

5.3.4.b applicable regulations; or

5.3.4.c guidelines in publications such as MARLAP, Validation and Peer Review of U.S. Environmental Protection Agency Radiochemical Methods of Analysis (FEM Document Number 2006-01), and/or The Fitness for Purpose of Analytical Methods, A Laboratory Guide to Method Validation and Related Topics (Second Edition, 2014).

5.4 Measurement Uncertainty

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

5.4.1.a Total Uncertainty shall be documented by the laboratory's quality system consistent with the Guide to the Expression of Uncertainty in Measurement, the recommendations in Chapter 19 of MARLAP Volume III (EPA 402-B-04-001C), or other equivalent approaches.

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

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

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

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

5.4.2.c indicate whether the uncertainty is the Standard Uncertainty (e.g., "1 standard deviation") or an Expanded Uncertainty (e.g., "k standard deviation"); and

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

5.4.3 The results of the precision evaluation shall be evaluated against the uncertainty estimates as a check on the validity of the uncertainty evaluation procedures.

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

should be re-evaluated.

5.4.3.b A comparison of the experimentally-observed precision evaluation need not be performed for measurements that are required to be reported only with Counting Uncertainty.

5.4.4 Combined Standard Uncertainty

5.4.4.a All measurement uncertainties shall be propagated and reported with each result.

5.4.4.b The formula for calculating the CSU of a result shall be documented in the appropriate procedure. The CSU shall include both systematic and random uncertainty.

5.4.4.c CSU shall be 1 standard deviation.

5.4.4.d Results shall be reported at the 95% confidence level, which is 1.96 standard deviations (often abbreviated as 2 standard deviations).

5.4.4.e The uncertainty of a count may be estimated as the square root of counts except when there are zero counts.

5.4.4.f In the case of zero counts, the uncertainty of the count is assumed to be the square root of one count. The uncertainty of a net count would have to propagate the uncertainty of the sample and background. Thus, the uncertainty for a zero count background and zero count sample is assumed to be 1.4 (square root of 2).

5.4.4.g For counting methodologies where very low counts are possible, MARLAP (equation 19.57) may be used if accepted by the customer. The laboratory shall maintain records of the customer acceptance.

5.4.5 The Systematic Uncertainty component of the reported uncertainty shall include, but is not necessarily limited to:

5.4.5.a the uncertainty from all measurement devices, including pipettes, balances, etc.; and

5.4.5.b the uncertainty of known values of tracer solutions, calibration uncertainties, etc.

5.4.6 The Random Uncertainty component of the reported uncertainty shall include, but is not necessarily limited to, the total random counting uncertainty associated with each sample and shall be appropriately propagated when more than one variable is used to determine the result.

5.5 Evaluation of Selectivity

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

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

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

5.5.1.c spectral and instrumental interferences.

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

6.0 Demonstration of Capability (DOC)

6.1 General

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

6.1.2 Thereafter, the individual shall perform ongoing DOC.

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

6.1.4 All DOCs shall be documented. All data applicable to the DOC shall be retained and available for review at the laboratory.

6.2 Initial DOC

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

6.2.1 The laboratory shall have a procedure for performing an initial DOC.

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

6.2.2.a individual(s) involved in preparation and/or analysis;

6.2.2.b matrix;

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

6.2.2.d identification of method(s) performed;

6.2.2.e identification of laboratory-specific procedure used for analysis, including revision number;

6.2.2.f date(s) of analysis; and

6.2.2.g summary of analyses.

6.2.3 If the method, regulation or contract does not specify an initial DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to initial DOC are adequate. For methods where spiking is not a viable option the laboratory approach shall include observation and evaluation of negative controls.

6.2.3.a Prepare four Test Samples consistent with Section 7.2.3. The individual shall also prepare four blank samples of clean quality system matrix in which no target analytes or interferences are present at activities that will impact the results of a specific method.

6.2.3.b Where gamma-ray spectrometry is used to identify and quantify more than one analyte, the Test Sample shall contain gamma-emitting radionuclides that represent the low (e.g., Americium-241), medium (e.g., Caesium-137), and high (e.g., Cobalt-60) energy range of the analyzed gamma-ray spectra. As indicated by these examples, the nuclides need not exactly bracket the calibrated energy range or the range over which nuclides are identified and quantified.

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

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

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

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

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

6.3 Ongoing DOC

6.3.1 The laboratory shall have a procedure for ongoing DOC that includes how the

laboratory will identify data associated with ongoing DOCs. The individual shall demonstrate on-going capability by routinely meeting the QC requirements of the reference method, laboratory procedure, customer specifications, and/or this standard. If the method has not been performed by the individual in a 12-month period, an initial DOC shall be performed. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate. For methods where spiking is not a viable option the laboratory approach shall include observation and evaluation of negative controls.

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

6.3.2.a acceptable performance of blank(s) and sample(s) that have known, accepted values, single blind to the individual;

6.3.2.b another initial DOC;

6.3.2.c at least four consecutive spiked samples (e.g., batch laboratory control samples) each with levels of precision and accuracy consistent with those specified in the method scope; and four consecutive blank samples, each with activity consistent method performance specified in the method scope (e.g., generally activity less than Decision Level). The laboratory shall tabulate or be able to readily retrieve four consecutive passing Laboratory Control Samples (LCS) and four consecutive blank samples for each method for each individual performed within the last 12-month period. The laboratory shall specify acceptable limits for precision and accuracy prior to analysis;

6.3.2.d A procedure of reviewing ongoing QC samples by an individual or a predefined group of analysts relative to the QC requirements of the reference method, laboratory procedure, customer specifications, and/or this standard. This review should be used to identify patterns for individuals or groups of analysts and identify the need for corrective action or retraining as necessary; or

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

7.0 Technical Requirements

7.1 Instrument Set-Up, Calibration, Performance Checks, and Background Measurements

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

These requirements ensure that the measurements will be of known and appropriate quality for meeting regulatory and contractual requirements and for supporting decision making. This section does not specify detailed procedural steps for these operations but establishes essential elements for selection of the appropriate technique(s). This allows

flexibility and permits employment of a wide variety of analytical procedures and statistical approaches.

At a minimum, the instrument QC program shall incorporate requirements imposed by the reference method, laboratory procedure, customer specifications, and/or this standard. Where imposed regulations are more stringent than this standard, the imposed regulations take precedence. If it is not apparent which standard is more stringent, the laboratory shall follow the requirements of the regulation or the method in that order. Where there are no established mandatory requirements, the laboratory shall incorporate guidelines consistent with MARLAP or other consensus standard organizations.

7.1.1 Initial Set-Up of Instrumentation

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

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

7.1.1.c The laboratory shall periodically verify user-maintainable values for operational parameters to ensure consistency with values recorded at the time of initial calibration to ensure the continued integrity of system configuration. If system configuration or operating parameters have changed, the laboratory shall implement its nonconforming work process to determine and ameliorate any potential impact.

7.1.2 Initial Calibration

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

7.1.2.a The laboratory shall have procedures describing when calibration of instrumentation is required. Radiation measurement systems shall be calibrated prior to initial use and any time the following conditions occur:

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

7.1.2.a.ii after a repair when subsequent performance checks indicate a change in

performance;

7.1.2.a.iii after modification of system parameters that affect instrument response;

7.1.2.a.iv when instrument performance checks exceed predetermined acceptance criteria (i.e., limit of a statistical or tolerance control chart or other QC parameters) indicating a change in instrument response since the initial calibration;

7.1.2.a.v when indicated by corrective actions; or

7.1.2.a.vi when calibration is due according to a predetermined frequency.

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

7.1.2.b.i channel-energy calibration of alpha or gamma spectrometers;

7.1.2.b.ii energy-efficiency calibration of gamma spectrometers;

7.1.2.b.iii mass-efficiency (mass-attenuation) calibration of gas-flow proportional or x-ray detectors;

7.1.2.b.iv quench-efficiency calibration of liquid scintillation detectors;

7.1.2.b.v mass-crosstalk calibration of gas-flow proportional; and

7.1.2.b.vi quench-crosstalk calibration of liquid scintillation detectors.

7.1.2.c Detected efficiency shall be determined with sources obtained from a reference material producer accredited to ISO 17034 or from a NMI, when available. When such materials are not available or not feasible for use, the laboratory may use reference materials obtained from external or internal sources. The laboratory shall have procedures and criteria for ensuring these sources have one or more properties sufficiently well established to be used for calibration or assessment of a measurement method. Records shall be maintained.

7.1.2.d The laboratory shall base instrument calibrations on physical measurement of reference standards. These standards shall have general physical characteristics (e.g., geometry, density, composition, nuclear decay properties, etc.) that match as closely as possible those of the samples to which the calibration will be applied.

7.1.2.e In some cases, calibration standard characteristics do not exactly match sample characteristics. The laboratory may use empirical techniques (e.g., gamma transmission) and/or computational techniques (e.g., Monte Carlo or efficiency modeling techniques) to generate corrections that are applied to calibrations

performed with reference standards to account for minor differences between the physical characteristics of the calibration standard (i.e., geometry, density, coincidence-summing, etc.) and the samples to which the correction is to be applied, if:

7.1.2.e.i the laboratory has performed a validation of the correction method or model by physical measurement of reference standards. Validations shall be performed according to a procedure and records shall be maintained. The validation shall span the entire range of physical characteristics observed in samples to which the correction shall be applied (e.g., geometry, density, etc.);

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

7.1.2.e.iii the laboratory has estimated and validated the uncertainty associated with the correction and included it in the uncertainty reported with each associated sample result.

7.1.2.f The following items are essential elements of initial instrument calibration:

7.1.2.f.i The laboratory shall have procedures for performing initial instrument calibration. The procedures shall include, at a minimum:

7.1.2.f.i.a the type of calibrations to be performed;

7.1.2.f.i.b the number of calibration points required;

7.1.2.f.i.c a description of the calibration standards required;

7.1.2.f.i.d the preparation of the calibration standards;

7.1.2.f.i.e the counting of the calibration standards;

7.1.2.f.i.f the maximum permissible uncertainty for calibration measurements (e.g., a maximum relative combined uncertainty of the calibration parameter or a minimum number of counts collected); and

7.1.2.f.i.g all calculations.

7.1.2.f.ii The laboratory procedures shall contain acceptance criteria appropriate to the calibration technique.

7.1.2.f.iii If the initial instrument calibration results are outside established acceptance criteria, the laboratory shall implement its nonconforming work process.

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

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

7.1.3 Calibration Verification

7.1.3.a Prior to use of an initial calibration for analysis of samples, the laboratory shall verify the initial instrument calibration with a reference standard. The laboratory shall obtain the standard from a source or a lot independent of the reference standard used in the initial calibration, if available. The calibration verification may take two forms:

7.1.3.a.i performing a second set of calibration measurements to be evaluated against the initial calibration; or

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

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

7.1.3.c The laboratory shall specify calibration verification acceptance criteria in its procedures (e.g., for the relative combined uncertainty of the prepared standard recovery). If the acceptance criteria for the calibration verification are not met, the laboratory shall implement its nonconforming work process.

7.1.4 Instrument Performance Checks

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

7.1.4.a The following are essential elements of instrument performance checks:

7.1.4.a.i The check source used for instrument performance checks need not be a reference standard;

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

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

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

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

7.1.4.a.vi The laboratory shall monitor the results of instrument performance checks

using control or tolerance charts to ensure that instrument performance does not change significantly relative to the point of the initial calibration;

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

7.1.4.a.viii When results for instrument performance checks are outside acceptance criteria (i.e., limit of a statistical or tolerance chart or other QC parameters), the laboratory shall implement its nonconforming work process.

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

7.1.4.b.i Gamma-ray spectrometry systems

Detection efficiency, energy calibration, and peak resolution.

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

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

7.1.4.b.ii Alpha-particle spectrometry systems

7.1.4.b.ii.a Energy calibration: Weekly.

7.1.4.b.ii.b Detection efficiency: Monthly.

7.1.4.b.iii Gas-proportional and semiconductor alpha/beta detectors

Alpha and beta efficiency: Day of use.

7.1.4.b.iv Liquid scintillation detectors

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

7.1.4.b.iv.b Efficiency with unquenched Hydrogen-3 and Carbon-14 standards: Day of use.

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

Efficiency: Day of use.

7.1.4.b.vi For radon scintillation detectors, efficiency shall be verified at least annually, when the system is in use. Laboratories may rotate batch LCS samples through Cell/Detector pairs to provide evidence of continuing calibration verification.

7.1.4.c Exceptions to minimum frequencies for performance checks:

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

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

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

7.1.5 Background Subtraction Measurements

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

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

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

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

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

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

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

measurements at the following minimum frequencies:

7.1.5.c.ii.a Gamma-ray spectrometry systems: Monthly.

7.1.5.c.ii.b Alpha-particle spectrometry systems: Monthly.

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

7.1.5.c.ii.d Liquid scintillation detectors.

7.1.5.c.ii.d.1 Individual quenched background: Once per Preparation Batch.

7.1.5.c.ii.d.2 Quenched background curve: According to frequency specified in laboratory procedures.

7.1.5.c.ii.e Solid-state scintillation detectors (e.g., zinc sulfide) used for non-spectrometric measurements: Day of use.

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

7.1.5.c.iii Composite measurements, in which the background subtraction is determined by combining background measurements collected in a manner that results in a representative determination of the background with a combined counting time at least as long as the longest associated Test Source count time.

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

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

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

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

7.1.5.e When the background subtraction has changed since the previous determination such that significant bias is imparted to intervening Test Source measurements, the laboratory shall initiate its nonconforming work process. If the bias cannot be resolved, the laboratory shall qualify all affected results.

7.1.5.f Background Subtraction Count (BSC) measurements shall be conducted after calibration and thereafter at the minimum frequencies identified in this module.

7.1.5.g Measurements shall be monitored for trends to ensure that a laboratory maintains

its capability to meet required project objectives.

7.1.5.h A background subtraction shall also be collected before and after any counting chamber changes are made (i.e., cleaning, liner replacement, or instrument modification).

7.1.6 Short Term Background Checks

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

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

7.1.6.a.i indicate the frequency and length of checks;

7.1.6.a.ii establish control or tolerance charts and acceptance criteria of short-term background checks; and

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

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

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

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

7.1.6.c When short-term background has changed since the previous determination, such that significant background bias is imparted to intervening Test Source measurements, the laboratory shall initiate its nonconforming work process. If the bias cannot be resolved, the laboratory shall qualify all affected results.

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

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

7.1.6.f 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.

7.1.6.g A background check shall also be collected before and after any counting chamber changes are made (i.e., cleaning, liner replacement, or instrument modification).

7.1.7 Contamination Monitoring

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

7.2 Quality Control for Radiochemistry

7.2.1 General

7.2.1.a The laboratory shall follow a documented QC program that monitors and assesses the performance of the laboratory's measurement systems. At a minimum, the QC program shall incorporate requirements imposed by the reference method, laboratory procedure, customer specifications, and/or this standard. Where imposed regulations are more stringent than this Standard, the imposed regulations take precedence. If it is not apparent which requirement is more stringent, the laboratory shall follow the requirements of the regulation or the mandated method. Where there are no established requirements, the laboratory may reference guidelines consistent with MARLAP or other consensus standard organizations in its quality system.

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

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

7.2.1.b.ii Where testing is performed that does not involve physical or chemical processing which affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas

proportional detectors), an RMB may be initiated in lieu of a Preparation Batch. The samples and associated QC in the RMB shall share similar physical and chemical parameters, and analytical configurations (e.g., analytes, geometry, calibration, and background correction).

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

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

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

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

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

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

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

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

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

7.2.1.i When results do not meet acceptance criteria, the laboratory shall implement its

nonconforming work process. The laboratory shall consider samples associated with a Method Blanks and Laboratory Control Samples that are outside acceptance as suspect and shall, wherever possible, reprepare and analyze such samples. Where samples cannot be reprepared and analyzed, the laboratory shall report results with appropriate data qualifiers. Where sample specific quality controls are outside acceptance limits, the data shall be evaluated to determine if the source of the failure is analytical error. If so, reprepare and analyze the affected quality control and field samples if sufficient sample material is available. Otherwise, qualify specific analytes in the parent sample. The laboratory shall note the occurrence of a QC sample falling outside acceptance criteria and any associated actions in the laboratory report.

7.2.1.j Method specific QC requirements are located in Appendix B of this standard. All method QC samples shall follow customer requirements or Appendix B requirements.

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

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

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

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

7.2.2.b.i The laboratory shall prepare the MB using materials that are free of analytes of interest at levels that will interfere with the evaluation of the results. If an analyte-free matrix is not available, the laboratory shall use a surrogate matrix to simulate the quality system matrix.

7.2.2.b.ii The sample aliquot used for the MB shall be similar to that of routine samples. If the sample aliquot in a Preparation Batch varies (e.g., due to differences in sample density or restrictions on the activity or mass residue that may be processed), the laboratory shall use acceptance criteria that compensate for differing aliquot sizes (e.g., z-score per Section 18.4.1 of MARLAP Vol. III (EPA 402-B-04-001C)).

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

7.2.2.c.i the MB exceeds the pre-established upper or lower bounds for the measurement, where the upper and lower bounds are plus x times the Standard Uncertainty and negative y times the Standard Uncertainty (x and y are the coverage factors for the confidence interval as established by the

laboratory's quality system). The upper and lower bounds are not necessarily symmetrical; and

7.2.2.c.ii when applicable, the sample-specific MDA for the MB is greater than the required MDA.

7.2.2.d The laboratory shall implement its nonconforming work process if it is determined that a MB result is significantly different from zero and associated sample results are less than five times the MB activity, or if a MB result may impact the analytical results.

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

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

7.2.2.g 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.

7.2.2.h The following batch blank matrices shall be used for all radiochemistry analyses:

7.2.2.h.i For aqueous samples: Distilled or deionized water, analyte free, as demonstrated in Method Blanks;

7.2.2.h.ii For solid samples: Characterized solid material representative of the sample matrix; and

7.2.2.h.iii For filter samples: Preferably use a customer supplied filter blank from the same production lot number as the collected samples. Alternately, use filters physically and chemically identical to that collected by the customer (analyte free).

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

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

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

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

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

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

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

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

7.2.3.e When available, the standard used to prepare the LCS shall meet the requirements for reference standards. The final prepared LCS need not be traceable to a NMI. The LCS shall include all of the radionuclide(s) being determined with the following exceptions:

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

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

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

7.2.3.e.iii.a a radionuclide with similar gamma energies as those of the analyte(s) of interest may be used (e.g., Barium-133 may be used in place of Iodine-131); or

7.2.3.e.iii.b the LCS shall contain gamma-emitting radionuclides that, at a minimum,

represent the low (e.g., Americium-241) and high (e.g., Cobalt-60) energy range of the analyzed gamma-ray spectra. Commonly a medium energy radionuclide is also included in the LCS (e.g., Caesium-137). As indicated by these examples, the nuclides need not exactly bracket the calibration energy range or the range over which radionuclides are identified and quantified.

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

7.2.3.g Where more than one analyte is spiked at a level that meets the LCS requirements, each shall be assessed against the specified acceptance criteria.

7.2.3.h The LCS shall be counted for a sufficient time to quantify the activity level of the LCS.

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

7.2.4 Sample-Specific QC Measures

The laboratory shall document procedures for determining the effect of the sample matrix on the analytical results. These procedures relate to the analyses of specific QC samples and are designed as data quality indicators for a specific sample using the designated method. Examples of sample-specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD), Matrix Duplicate (MD), Tracers, and Carriers. The laboratory shall have procedures in place for tracking, managing, and handling sample-specific QC criteria including spiking radionuclides at appropriate activities, calculating recoveries, determining variability (e.g., relative percent difference and/or z-score), and evaluating and reporting results based on the performance of the QC samples.

7.2.4.a Matrix Spike

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

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

7.2.4.a.iii The radionuclides spiked shall be as specified by the mandated method, regulation or as determined as part of the contract review process. At minimum, they will be consistent with those specified for the LCS.

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

7.2.4.a.v When an MS is required, the lack of sufficient sample aliquot to perform an MS shall be explained in case narrative.

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

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

7.2.4.a.viii When available, the standard used to prepare the MS shall meet the requirements for reference standards. The final prepared MS need not be traceable to a NMI.

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

7.2.4.a.x Matrix spikes shall be run on a separate sample aliquot using the same analyte as that being analyzed.

7.2.4.a.xi Matrix Spike Selection and Level:

7.2.4.a.xi.a The matrix spike shall be added at a concentration of at least five times, but not greater than 20 times the Decision Level.

7.2.4.a.xi.b For samples having known significant activity of the targeted radionuclides, more than 20 times the Decision Level may be added to minimize the effect of the sample activity on determination of spike recoveries.

7.2.4.a.xi.c Other customer specifications may be required. In these cases, customer direction shall be recorded.

7.2.4.a.xii Counting:

7.2.4.a.xii.a The matrix spike shall be counted for a sufficient time to quantify the activity level of the spiking.

7.2.4.b Matrix Duplicates/Matrix Spike Duplicates

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

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

7.2.4.b.iii At a minimum, the laboratory shall analyze one MD per Preparation Batch or RMB. For RMBs, the MD shall consist of a second measurement of one sample. If the batch is counted on more than one detector, the MD shall be performed on a second detector.

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

7.2.4.c Chemical Yield Tracers and Carriers

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

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

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

7.2.4.c.iv The chemical yield shall be assessed against acceptance criteria specified in the method, regulation, contract or laboratory procedure. The laboratory shall develop its criteria for data acceptance based on guidelines established in the MARLAP or other criteria such as control charting developed by the laboratory. This assessment shall meet established project or program MQOs.

7.2.4.c.v When the established chemical yield acceptance criteria are not met, the

laboratory shall implement its nonconforming work process. The occurrence of a chemical yield outside acceptance and the actions taken shall be explained in the case narrative.

7.2.4.c.vi When tracers or carriers are used, each sample (including any batch associated QC samples) shall also be spiked with the same materials and individual sample yields shall be determined.

7.2.4.c.vii Requirements for indirect yield measurements:

7.2.4.c.vii.a When used, radiometric results shall be corrected for chemical yield using 'indirect' yield measurement techniques such as a second radiometric measurement of added tracer or gravimetric measurement of added carriers.

7.2.4.c.vii.b The acceptance criteria for the chemical yield for each sample determined using an indirect yield measurement method shall fall between 30% - 110% or as specified by the customer.

7.2.4.c.vii.c The technique used for the indirect yield measurement shall be sufficient to maintain relative uncertainties associated with the yield correction below 10% at 2 standard deviations.

7.2.4.c.vii.d When tracer impurities or peak tailing from the tracer effectively raise the detection limit of an intended analyte, smaller tracer activities may be used, with customer approval, to achieve an acceptable detection limit. Records of customer approval shall be maintained.

7.2.4.c.viii Sample results with yields below 30% are considered quantitative and acceptable if:

7.2.4.c.viii.a The relative uncertainty associated with the yield correction is less than 10% (2 standard deviations);

7.2.4.c.viii.b Spectral resolution requirements are met and there are no indications of spectral interferences (alpha spectroscopy); and

7.2.4.c.viii.c Detection limit requirements are met.

7.2.4.c.ix Reporting yield measurement uncertainties:

7.2.4.c.ix.a The uncertainty associated with chemical yield corrections shall be incorporated into the CSU of the associated sample results.

7.2.4.c.x Tracer yield requirements for isotope direct yield methods: (usually alpha spectroscopy).

7.2.4.c.x.a The acceptance criteria for chemical yield for isotope direct yield methods shall be 30% - 110% or as specified by the customer.

7.2.4.c.x.b Tracer activity and sample count duration shall be adequate to achieve

relative uncertainties for the tracer measurement of less than 10% at 2 standard deviations.

7.2.5 Data Reduction

7.2.5.a The procedures for data reduction shall be documented.

7.2.5.b Detection capability (e.g., MDA or Critical Level) shall be calculated as described in Section 5.2.

7.2.5.c Measurement uncertainties shall be calculated and reported.

7.2.5.d Negative Numbers:

7.2.5.d.i All negative activities shall be reported as such.

7.2.5.d.ii If the sum of the activity and the measurement uncertainty at ± 3 standard deviations is a negative number, the cause shall be investigated and evaluated to determine if it is systematic or random uncertainty.

7.2.5.d.ii.a If the cause is systematic, it shall be corrected.

7.2.5.d.ii.b If the cause is random, it shall be discussed in the case narrative.

7.2.5.d.iii Recurrent problems with significant negative results suggest that the background subtraction and/or blank subtraction, if applicable, are in question or that the estimate of uncertainty is low.

7.2.5.d.iv The laboratory shall investigate the cause of recurrent negative results. Records of these investigations shall be maintained. All instances of negative results shall be discussed in the case narrative.

7.2.6 Reagent Quality, Water Quality, and Checks

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

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

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

7.2.6.c.i Reference standards shall be obtained from a NMI or from suppliers of NMI reference standards. Alternatively, reference standards may be obtained from an ISO 17034 accredited reference material provider, or an ANSI N42.22 reference material manufacturer.

7.2.6.c.ii Reference standards shall be accompanied with a certificate of calibration that meets the requirements of either ISO Guide 31, or ANSI N42.22,

Section 8, Certificates, and shall include at least the following information: manufacturer, radionuclides calibrated, identification number, calibration method, activities or emission rates with associated uncertainties and the confidence limits, standard quantity, activity reference time (date or time as appropriate to the half-life of the radionuclide), physical and/or chemical description of the source, and radionuclide impurities.

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

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

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

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

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

7.2.6.d Standards shall be verified prior to initial use.

7.2.6.d.i Preparations of standards solutions used for a period exceeding one year shall be verified annually, at a minimum, and the laboratory shall maintain records of the verification.

7.2.6.d.ii At least three verification measurements of a standard shall be used to determine the mean value and standard deviation of the verification results.

7.2.6.d.iii The mean value shall be within 5% of the decay corrected certified value.

7.2.6.d.iv The 2 standard deviations used for the 95% confidence interval of the mean shall not exceed 10% of the mean value of the three verification measurements.

7.2.6.d.v If the above criteria are met, the certified value shall be used.

7.2.7 Constant and Consistent Test Conditions

7.2.7.a The laboratory shall assure that test instruments consistently operate within the specifications required of the application for which the equipment is used.

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

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

7.2.7.d Background contamination monitoring samples shall be analyzed at a sufficiently low level of detection to confirm that no impacts to customer samples have occurred due to cross-contamination.

7.2.7.e Samples shall be segregated by activity levels in sample receipt, processing areas, and storage areas.

7.3 Data Evaluation and Reporting

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

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

7.3.1.b If acceptance limits are not met, laboratory shall implement its nonconforming work process to investigate the source of contamination or other bias. If sample activity levels are greater than five times the activity found in the MB, lacking other requirements, it is acceptable to report qualified results for the samples associated with the MB. Otherwise, reprepare and analyze the associated samples.

7.3.1.c When sample results associated with a MB falling outside acceptance are reported, the laboratory shall implement its nonconforming work process and this shall be explained in the case narrative.

7.3.1.d The blank acceptance criteria shall be: $|Z_{Blank}| \leq 3$ (MARLAP 18.4.1) or a laboratory shall use MB laboratory-developed acceptance criteria of ± 3 standard deviations of the mean.

7.3.1.e When utilizing MARLAP, the following equation is defined in Section 18 (Equation 18.1) for Method Blank evaluations.

$$Z\text{Blank} = \frac{x}{uc(x)}$$

Where: x denotes the measured blank activity and $u_c(x)$ denotes its combined standard uncertainty.

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

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

7.3.2.b An LCS that is determined to be within established acceptance limits effectively demonstrates that the measurement system is in control and validates system performance for the samples in the associated batch. Samples associated with an LCS that falls outside acceptance limits are considered suspect and the samples shall be reprepared and analyzed. If samples cannot be reprepared and analyzed, the laboratory shall implement its nonconforming work process and this shall be explained in the case narrative.

7.3.2.c Derive limits using $|Z_{LCS}| \leq 3$ or use laboratory-developed acceptance criteria of LCS ± 3 standard deviations of the mean.

7.3.2.d Laboratory-developed acceptance criteria shall not fall more than 25% from the known LCS value.

7.3.2.e When utilizing MARLAP, the following equation defined in Section 18 of MARLAP (Equation 18.3) shall be used for LCS evaluations:

$$Z_{\text{LCS}} = \frac{x - d}{\sqrt{u_c^2(x) + u_c^2(d)}}$$

Where: *x* is the measured value of the spiked sample and *d is* the spike concentration added, and $u_c^2(x)$ and $u_c^2(d)$ are the squares of the respective standard uncertainties.

7.3.3 Sample-Specific Controls

7.3.3.a Matrix Spike, Matrix Duplicates, and Matrix Spike Duplicates

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

7.3.3.a.ii For results outside established criteria, the laboratory shall implement its nonconforming work process and the data reported with appropriate data qualifiers. QC results outside acceptance limits shall be explained in the case narrative.

7.3.3.a.iii Matrix spike recoveries shall be evaluated using the following criteria:

7.3.3.a.iv If the activity of the sample is less than five times the spiking level, matrix spike recoveries shall be within the acceptance criteria of 60 - 140%, or as specified by the customer.

7.3.3.a.v If the activity of the sample is greater than five times the spiking level, $|Z_{MS}| \leq 3$ shall be used.

7.3.3.a.vi When utilizing MARLAP, the following equation defined in Section 18 (Equation 18.4) shall be used for MS evaluations:

$$Z_{\mathrm{MS}} = \frac{x - x_0 - d}{\sqrt{u_c^2(x) + u_c^2(x_0) + u_c^2(d)}}$$

Where: x is the measured value of the spiked sample, d is the spike concentration added, x_0 is the measured concentration of the unspiked sample, and $u_c^2(x)$, $u_c^2(d)$, and $u_c^2(x_0)$ are the squares of the respective standard uncertainties.

7.3.3.a.vii The Matrix Duplicate activity shall not be averaged with the corresponding sample activity when reporting results.

7.3.3.a.viii Samples identified as FB shall not be used for Matrix Duplicate sample analysis.

7.3.3.a.ix The Matrix Duplicate shall be counted for the same duration as the corresponding original sample and to meet the required detection limit.

7.3.3.a.x Matrix Duplicates are evaluated using three possible criteria. The laboratory shall document in its management system which option shall be used for each method. When the MARLAP, DER or the RPD meet the acceptance criteria, then the Matrix Duplicate is acceptable.

7.3.3.a.x.a $|Z_{Dup}| \leq 3$ if using MARLAP (equation 18.2);

$$Z_{\mathrm{Dup}} = \frac{|x_1 - x_2|}{\sqrt{u_c^2(x_1) + u_c^2(x_2)}}$$

Where: x_1 and x_2 denote the two measured activity concentrations and $u_c^2(x_1)$ and $u_c^2(x_2)$ are the squares of the respective standard uncertainties;

7.3.3.a.x.b the Duplicate Error Ratio (DER) between the sample and the Matrix Duplicate is < 3; or

7.3.3.a.x.c the relative percent difference (RPD) is < 25%.

7.3.3.b Tracers and Carriers

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

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

7.3.3.b.iii Samples associated with Tracers or Carriers that fall outside acceptance limits are considered suspect, and the samples shall be reprocessed and/or reanalyzed. If samples cannot be reprocessed and/or reanalyzed, the laboratory shall implement its nonconforming work process and this shall be explained in the case narrative.

7.3.4 Evaluation of Sample Results

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

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

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

7.3.4.d If these objectives have not been met, then samples shall be reprepared and analyzed. If samples cannot be reprepared and analyzed, the laboratory shall implement its nonconforming work process and this shall be explained in the case narrative.

7.3.5 Reporting Results

7.3.5.a Reports delivered to the laboratory's customer shall be consistent with the requirements of this Standard.

7.3.5.b Results shall be reported directly as obtained, with appropriate units, even if the results are negative.

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

7.3.5.d All radiochemical results shall be reported with an estimate of uncertainty.

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

7.3.5.f Project- or customer-specified reporting requirements can take precedence over the requirements of this Standard.

7.4 Sample Handling

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

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

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

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

7.4.3 If the results of the preservation verification do not satisfy established criteria, the laboratory shall initiate its nonconforming work process (i.e., notification of the customer, preservation of the sample at the time of discovery) and qualify all impacted test results in the report to the customer.

8.0 Method Specific Directions

8.1 Isotopic Determinations by Alpha Spectrometry

In the absence of customer specified criteria, the criteria outlined in Table B-16, Alpha Spectrometry shall be used.

8.1.1 Calibration (Initial, Initial Verification, and Continuing Verification):

8.1.1.a Calibration of each alpha spectrometry detector used to produce data shall include channel vs. energy calibration, detector response, efficiency determination and background determination for each Region of Interest (ROI).

8.1.1.b An 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 recorded.

8.1.1.c Alpha spectrum regions of interest shall be selected with consistency from analyte to analyte.

8.1.1.d ROIs shall be clearly indicated either graphically or in tabular form on alpha printouts. Records including spectra with ROIs shall be maintained and made available for review upon request.

8.1.1.e The estimated uncertainty in preparing the source shall be propagated into the uncertainty of the efficiency determination.

8.1.1.f Detector Response:

8.1.1.f.i The response (efficiency) counts for the ROI shall be background corrected using the same ROI for the background. If the background is less than 0.5% of the total counts in the ROI, no background correction is necessary.

8.1.1.f.ii Records shall be maintained of the detector response and detector response uncertainty.

8.1.1.f.iii Records shall be maintained of the detector response check as determined by the check source and/or pulser count and the associated uncertainty and limits of acceptability for the check source result.

8.1.2 Background Correction:

8.1.2.a The gross counts in each target analyte and tracer ROI shall be corrected for the particular detector's background contribution in those same ROIs.

8.1.2.b The background total counts (or counts per unit time) for each target analyte and tracer isotope ROI shall be determines on each detector at least monthly, prior to initial use or after initial calibration. Records shall be maintained.

8.1.2.c The background for each ROI shall be sufficiently low to ensure that required detection limits are met.

8.1.2.d The limits of acceptability for each background ROI shall be defined. These shall be set such that Decision Levels can be obtained for backgrounds at the limit of acceptability.

8.1.3 Batch Quality Control (Method Blank, Laboratory Control Sample, Matrix Spike, Matrix Duplicate):

8.1.3.a In the absence of customer specified criteria, the criteria outlined in B-16, Alpha Spectrometry shall be used.

8.1.4 Tracer and/or Carrier:

8.1.4.a When used for isotope specific analysis by alpha spectrometry, initial sample preparation shall include treatment to ensure that tracer/carrier and analyte will undergo similar reactions during processing.

8.1.4.b All tracers/carriers shall be of the same element or of an element with the same chemistry as the isotopes of interest for the separations.

8.1.4.c All tracers used for alpha spectrometry shall be tested by the laboratory for contribution in the ROI of the analytes of interest. If a significant contribution is found, the method for correction shall be documented and records maintained.

8.1.5 Spectrum Evaluation:

8.1.5.a 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.

8.1.5.b Any manual integration or adjustment of ROIs shall be discussed in the case narrative.

8.1.5.c 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 the tracer and analyte peaks are resolved.

8.1.5.d If the sample analyte spectrum contains significant interferences with the analyte and/or tracer ROIs, reanalysis is required.

8.2 Gamma Spectrometry

The gamma detector system shall consist of any detector suitable for measuring the gamma isotopes of interest with the capacity to attain specified required limits and to meet 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.

In the absence of customer specified criteria, the criteria outlined in Table B-17, Gamma Spectrometry shall be used.

8.2.1 Calibration (Initial, Initial Verification and Continuing Verification)

8.2.1.a Energy Calibration Requirements:

8.2.1.a.i Germanium Detectors:

8.2.1.a.i.a The energy calibration measurements shall be made using at least six peaks which cover the energy range from 0.059 to approximately 2 MeV. Additional peaks may be used as deemed appropriate by the laboratory.

8.2.1.a.i.b 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).

8.2.1.a.ii Sodium Iodide Detectors:

8.2.1.a.ii.a Refer to ANSI N42.12, Section 4.3.2 for guidance on NaI detectors.

Note: 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.

8.2.1.b Efficiency Calibration Requirements

8.2.1.b.i Germanium Detectors:

8.2.1.b.i.a The efficiency calibration approach selected for broad spectrum gamma analysis shall cover the energy range of the gamma ray peaks used for nuclide quantification.

8.2.1.b.i.b When establishing an efficiency curve as a function of energy, the efficiency calibration measurements shall be at least six peaks which cover the typical energy range of approximately 0.059 to 2 MeV.

8.2.1.b.i.c Low Energy Response: If the detector is to be used for emissions below the lowest energy of a broad-spectrum calibration (e.g., below the 0.059 MeV criteria identified above), additional demonstration of acceptable calibration is required.

8.2.1.b.i.c.1 If manufacturer's information indicates that low-energy response below the lowest energy in the calibration standard is expected to be constant, use of the detector below that point requires check sources or LCS quality control checks that contain the isotope to be quantified (or other isotope with lower emission energies).

8.2.1.b.i.c.2 Acceptable recovery shall be demonstrated for every detector used in that lower range.

8.2.1.b.i.c.3 If low-energy response below the lowest energy calibration standard is not expected to be constant, use of a gamma detector at energies below the lowest calibration point requires that a single-isotope efficiency curve or separate low-energy curve bounding the energy of interest be established for that isotope.

8.2.1.b.i.c.4 In all cases, the laboratory shall be able to demonstrate that sample matrix effects (including potential attenuation from sample containers) on low energy emissions have been accounted for.

8.2.1.b.ii Sodium Iodide Detectors:

8.2.1.b.ii.a Efficiencies shall be determined when there is a change in resolution, geometry, or system configuration.

8.2.1.c Model Based Efficiency Calibration Requirements

Software may be used to generate efficiencies for 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 and shall be validated with a physical reference standard. When such software is used:

8.2.1.c.i The laboratory shall supply detailed records of the selection of parameters used to specify the efficiency calibration and sample models.

8.2.1.c.ii Each sample type selected for analysis using this model-based calibration

shall have a unique set of model parameters associated with it.

8.2.1.c.iii When such models are used, the closest model to the actual sample shall be selected.

8.2.1.c.iv The model selected for each sample shall be discussed 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.

8.2.1.d Initial Calibration Verification (when required):

8.2.1.d.i A minimum of 5,000 net counts shall be accumulated in each peak in at least four calibration verification peaks that bracket the range of use.

8.2.2 Background:

8.2.2.a Background Subtraction Count

8.2.2.a.i The counting interval for the long count shall be between one and four times the nominal counting interval of the test sources.

8.2.2.b Contamination Check

8.2.2.b.i Integrate spectrum from about 50 – 2,000 keV to check for gross contamination and retain information in laboratory records.

8.2.3 Batch Quality Control

8.2.3.a LCS matrices shall be consistent with the associated samples and contain representative nuclides within the energy ranges of those nuclides to be reported.

8.2.4 Detectors shall be calibrated for the specific geometry and matrix considerations used in the sample analysis.

8.2.5 Spectral Data Reference

8.2.5.a Records shall be maintained of the identification of the reference used for the half-life, abundance, and peak energy of all nuclides.

8.2.5.b The laboratory shall document, review, and provide configuration control for gamma spectrometry libraries.

8.2.5.c Assumptions made for libraries [i.e., half-lives based on supported/unsupported assumptions, inferential determinations (e.g., Thorium-234 = Uranium-238 because supported)] shall be maintained in laboratory records, and available upon request.

8.2.6 Spectrum Assessment

8.2.6.a 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.

8.2.6.b Any major contributor to the spectrum that is an unidentified peak (e.g., an isotopic peak not requested by the customer, not included in the library for identification, or not included in the client library used) shall be discussed in the case narrative.

8.3 Gas Flow Proportional Counting

In the absence of customer specified criteria, the criteria outlined in Table B-18, Gas Flow Proportional Counting shall be used.

8.3.1 Calibration (Voltage Plateau, Initial Efficiency, Cross-Talk, Self-Absorption, Initial Efficiency Verification, and Continuing Calibration Verification):

8.3.1.a The calibration sources shall provide adequate counting statistics over the period for which the source is to be counted.

8.3.1.b The source activity shall be in the range expected from routine samples and not be high enough to cause pulse pileups or dead time that impacts the analyses.

8.3.1.c The geometry of the calibration sources used for efficiency and self-absorption/cross-talk curves shall be the same planchets, including depth, shape (flat, flanged, ringed, etc.) and diameter as that of the prepared sample and QC sample planchets.

8.3.1.d The sources used for the determination of self-absorption and cross-talk shall be of similar isotope content to that of the target analytes. Normally, Americium-241; Polonium-210; or Thorium-230 are used for alpha and Caesium-137or Strontium-90/ Yttrium-90 are used for beta.

8.3.1.e The frequency, procedure, and criteria for calibration shall be described in the laboratory procedure or reference method.

8.3.1.f Cross-talk Factors

8.3.1.f.i A cross-talk curve shall be established for alpha to beta cross-talk versus residue weight for each nuclide using traceable calibration sources per matrix and method.

8.3.1.f.ii Beta to alpha cross-talk is not significantly affected by planchet residue weight and is generally constant over the applicable weight range. Therefore, this cross-talk correction does not require residue weight consideration.

8.3.1.f.iii The data used to generate cross-talk curves shall consist of at least seven points, well distributed throughout the mass range.

8.3.1.f.iv Crosstalk correction is not necessary when there Is no net activity in the opposing channel as is the case when counting chemically separated radionuclides.

8.3.1.g Self-Absorption (Mass Attenuation) Curves

8.3.1.g.i Self-absorption curves shall be determined for both alpha and beta counting.

8.3.1.h Initial and Continuing Efficiency Calibration Verification:

8.3.1.h.i Records shall be maintained of all calibration verifications.

8.3.2 Background Correction

8.3.2.a The background shall be sufficiently low to ensure that required detection limits are met.

8.3.2.b A Long Background Count shall be performed for subtracting background from blanks and test sources. The counting interval for the long count shall be at least the same as the normal counting time for blanks and test sources.

8.3.3 Batch Quality Control (Method Blank, Laboratory Control Sample, Matrix Spike, Matrix Duplicate):

In the absence of customer specified criteria, the criteria outlined in Table B-18, Gas Flow Proportional Counting shall be used.

8.3.4 Tracer and/or Carrier

When used for GPC analysis, initial sample preparation shall include treatment to ensure that tracer/carrier and analyte will undergo similar reactions during processing. All tracers/carriers shall have the same chemistry as the isotopes of interest for the separations.

8.3.5 Planchets

8.3.5.a Planchets shall be thoroughly cleaned before use to ensure that there are no interfering residues or contamination.

8.3.5.b All Planchets shall be prepared not to exceed sample weights in excess of the calibrated ranges of established self-absorption curves.

8.3.5.c 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 or flaming prove unsuccessful.

8.3.6 Sample Analyses

8.3.6.a Sample mass shall be recorded and demonstrated to be stable prior to counting.

8.3.6.b Any non-routine counting situations shall be discussed in the case narrative.

8.4 Liquid Scintillation Counting

In the absence of customer specified criteria, the criteria outlined in Table B-19, Liquid Scintillation Counter Analysis shall be used.

8.4.1 Tritium in Water

Water samples for tritium analysis and all associated QC samples shall be distilled prior to analysis unless specified otherwise by the customer. The applicable preparation procedure shall specify the fraction to be collected. The same fraction shall be collected for samples and all associated QC samples.

8.4.2 Counting Vial Preparation

8.4.2.a Samples shall be counted in low potassium glass vials or high-density polyethylene vials. Any other vials shall be accepted for use by the customer. Records of the acceptance criteria shall be maintained.

8.4.2.b 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. Analytical records shall contain sufficient information for this to be verified.

8.4.2.c Vials shall be prepared according to manufacturer's specification for the cocktail.

8.4.2.d The vials shall be "dark adapted" for a minimum of 30 minutes or according to the cocktail manufacturer's specifications before counting.

8.4.2.e The prepared vials shall be inspected to verify that the sample loaded properly in the cocktail and there is no visual evidence of phase separation before or after completion of the count.

8.4.2.f The sample volume to scintillation cocktail volume ratio shall be consistent for all standards and samples.

8.4.3 Calibration (Initial, Initial Verification, and Continuing Verification)

Any component of the cocktail that affects the energy transfer process and has a significant effect on the analysis and is referred to as quench. The quench of a cocktail can be affected by: color; turbidity; molecules of high electron affinity; solvent; acidity; and dissolved gases. Calibrations are typically established using an efficiency-response/quench curve.

8.4.3.a Calibration quench sets shall not be stored in direct sunlight or under fluorescent lights.

8.4.3.b For analysis methods using quench curves to determine individual sample detection efficiency or background, the quench curves shall be generated at a frequency defined by the laboratory and described in the laboratory procedure.

8.4.3.c A calibration shall be established for each radionuclide and liquid scintillation cocktail matrix.

8.4.4 Detector Response

8.4.4.a Calibration and window settings shall be specific for a radionuclide/method application.

8.4.5 Instrument Background

8.4.5.a The instrument background vial for all tritium matrices shall be prepared with low-tritium or “dead” water unless the laboratory can demonstrate suitably small background or blank effects from other sources of water.

8.4.5.b The instrument background vial shall be prepared with the same water to cocktail ratio as the samples are prepared.

8.4.5.c The type of water (dead water, DI water, etc.) used to prepare the instrument background vial shall be identified in the procedure.

8.4.5.d The instrument background shall be analyzed 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).

8.4.5.e The effect of quench on background shall be evaluated and corrected using a background quench curve if it is significant.

8.4.6 Batch Quality Control (Method Blank, Laboratory Control Sample, Matrix Spike, Matrix Duplicate):

In addition to the requirements in 7.1.5 b), d), if customer criteria are not specified, the criteria outlined in Table B-19, Liquid Scintillation Counter Analysis shall be used.

8.4.6.a Tracer and/or Carrier

8.4.6.a.i If a laboratory uses a tracer or carrier, its use and implementation shall be specified in the laboratory’s procedure.

8.4.6.a.ii When used for isotope specific analysis by liquid scintillation counter analyses, initial sample preparation shall include treatment to ensure that tracer/carrier and analyte will undergo similar reactions during processing.

8.4.6.a.iii All tracers used for liquid scintillation counter analysis shall be tested by the laboratory for contribution in the regions of interest (ROI) of the analytes of interest. If a significant contribution is found, the method for correction shall be documented and accepted by the customer prior to use. Records shall be maintained.

8.4.7 Spectrum Evaluation

8.4.7.a Sample spectra shall be reviewed and made available to the customer for each sample and QC sample.

8.4.7.b 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.

8.4.8 Sample-Specific Conditions

The following are additional conditions that require reanalysis for a particular sample and analyte, beginning with the preparation or recounting, as appropriate.

8.4.8.a 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. If this cannot be achieved, then it will be discussed in the case narrative.

8.5 Radon Scintillation (Lucas Cell)

Analyses by Lucas Cell shall incorporate EPA Method 903.1 (current version), *Radium-226 in Drinking Water Radon Emanation Technique*, or ASTM D3454. When references are updated, an implementation schedule shall be determined by the laboratory. Deviations from the method shall be documented in the laboratory's procedure and accepted by the customer.

In the absence of customer specified criteria, the criteria outlined in Table B-20, Radon Scintillation shall be used.

8.5.1 Calibration (Initial, Initial Verification, and Continuing Verification)

8.5.1.a Bubblers used for Radium-226 calibrations shall not be used for sample analysis.

8.5.1.b Initial Calibration shall be performed prior to use, following repair, loss of control, or upon incorporation of new or changed instrumentation.

8.5.1.b.i Establish the operating voltage plateau for each detector.

8.5.1.b.ii Verify manufacturer's specifications and confirm each selected operational voltage is within the slope range of <2%/100V.

8.5.1.b.iii Establish the Cell/Detector Efficiency for each Cell/Detector pair by using a traceable Radium-226 calibration source that matches the test sample configuration (type, size, and position relative to the detector).

8.5.1.c Continuing Calibration Verification

8.5.1.c.i Perform a detector response check each day of use using an appropriate test source. The results of this detector response check shall be within established laboratory-developed acceptance criteria.

8.5.1.c.ii Each Cell/Detector pair efficiency shall be verified at least annually. The continuing efficiency for each Cell/Detector pair shall be within + 25% of the initially determined efficiency.

8.5.1.d Any cell/detector pair removed from service will require re-calibration prior to

being returned to service.

8.5.2 Background Counts

8.5.2.a Each Cell shall be cleaned/purged, evacuated, and counted on its paired detector prior to the next use. Results of this background count shall be within the laboratory-developed acceptance criteria that does not impact sample analyses.

8.5.3 Batch Quality Control (Method Blank, Laboratory Control Sample, Matrix Spike, Matrix Duplicate):

8.5.3.a Method Blank

8.5.3.a.i Unless required by the program, quality control checks are not used to correct sample activities, but only to monitor contamination.

8.5.3.a.ii Prepared one method blank per preparatory batch.

8.5.3.a.iii Laboratories shall rotate batch method blank samples though cell/detector pairs to provide evidence of continuing process contamination control.

8.5.3.a.iv Count time for the method blanks shall be equal to or longer than associated sample count times.

8.5.3.a.v The acceptance criteria for the method blank shall be $|Z_{Blank}| < 3$ or within laboratory-developed criteria of + 3 standard deviations of the mean.

8.5.3.b Laboratory Control Sample

8.5.3.b.i Prepared one LCS per preparatory batch.

8.5.3.b.ii Laboratories shall rotate batch LCS samples though cell/detector pairs to provide evidence of continuing calibration.

8.5.3.b.iii The LCS shall meet customer specified requirements, acceptance criteria of $|Z_{LCS}| < 3$, or laboratory-developed acceptance criteria of + 3 standard deviations of the mean that are within 25% of the known LCS value.

8.5.3.c Matrix Spike Sample

8.5.3.c.i Prepare one MS per preparatory batch.

8.5.3.c.ii The MS shall meet customer specified requirements, an acceptance criterion of $Z_{MS} \leq 3$ if the activity level of the sample is greater than 5 times the spiking level or be within 40-140% recovery.

8.5.3.d Duplicate

8.5.3.d.i Prepare one duplicate per preparatory batch (one per 10 for drinking water sample groups).

8.5.3.d.ii The duplicate shall meet customer specified requirements, acceptance criteria

of $|Z_{Dup}| \leq 3$, a duplicate error ratio of ≤ 3, or a relative percent difference of $\leq$ 25%.

8.5.4 Carrier

8.5.4.a If carriers are used quantitatively, the laboratory shall follow the criteria in Table B-20 of Appendix B.

Module 7

Quality Systems for Toxicity Testing

1.0 Introduction

This module provides specific requirements for laboratories seeking accreditation for toxicity testing. Laboratories seeking accreditation for toxicity testing shall also meet the requirements presented in Module 1 and 2 of this standard. This module applies to laboratories measuring the toxicity and/or bioaccumulation of contaminants in effluents (whole effluent toxicity or WET), receiving waters, sediments, elutriates, leachates, and soils. In addition to the essential quality control (QC) standards described below, some methods may have additional or other requirements based on factors such as the type of organism evaluated and contain detailed QC requirements for toxicity testing activities.

2.0 Scope

This module provides quality assurance and QC requirements for laboratories seeking to obtain and maintain accreditation for toxicity testing methods. This module is not intended to relieve additional QC requirements that are specified by method, regulation, or customer.

3.0 Terms and Definitions

Definitions are considered requirements. Definitions are included in Module 2, Section 3.0. Additional relevant terms definitions related to this module are defined listed below.

Sensitivity: When referred to in this module, “sensitivity” relates to the meaning in the reference method.

4.0 Method Selection

The requirements in the Module 2 section on “Selection, Verification and Validation of Methods” apply.

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

The characteristics of validated methods (e.g., the uncertainty of the results, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the customer’s needs.

5.0 Method Validation

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

6.0 Demonstration of Capability (DOC)

6.1 General

Prior to acceptance and institution of any method for data reporting, satisfactory initial DOC is required.

Thereafter, ongoing DOC, as per the QC requirements in Section 7.1.2, is required.

In cases where a laboratory analyzes samples using a method that has been in use by the laboratory for at least one year prior to applying for accreditation, and there have been no significant changes in personnel or method, the ongoing DOC shall be acceptable as an initial DOC. The laboratory shall retain records to demonstrate that an initial DOC is not required.

For the initial DOC, appropriate records as discussed in Section 6.2.1 shall be completed. An initial DOC shall be completed each time there is a change in personnel, or method.

In general, this demonstration does not test the performance of the method in real world samples. However, before any results are reported, the initial DOC shall be performed. An initial DOC may be completed by a group of analysts and is for situations in which several individuals perform part of a set of activities that would produce a testing result.

All demonstrations shall be recorded. All data applicable to the demonstration shall be retained and readily available at the laboratory.

6.2 Initial DOC

An initial DOC shall be made prior to using any method, and at any time there is a significant change in personnel or method or any time that a method has not been performed by the laboratory or individual in a 12-month period.

6.2.1 The laboratory shall maintain records of each initial DOC in a manner such that the following information is available for each affected employee:

6.2.1.a individual(s) involved in preparation and/or analysis;

6.2.1.b matrix;

6.2.1.c species and endpoint(s);

6.2.1.d identification of method(s) performed;

6.2.1.e identification of laboratory-specific procedures used for analysis, including revision number;

6.2.1.f date(s) of analysis; and

6.2.1.g summary of analyses, including information outlined in Section 6.2.2.

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

Each analyst shall meet the QC requirements as specified in Section 7.1.2.

6.3 Ongoing DOC

The laboratory shall have a procedure describing ongoing DOC. The individual(s) shall demonstrate ongoing capability by meeting the QC requirements of the reference method, laboratory procedure, customer specifications, and/or this Standard. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate. This ongoing demonstration may include performing another initial demonstration of capability as per 6.2 or a documented process of analyst review using QC samples may serve as the annual ongoing DOC. QC samples shall be reviewed to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary.

7.0 Technical Requirements

7.1 Quality Control

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

7.1.a regular use of certified reference materials and/or internal QC using secondary reference materials;

7.1.b participation in inter-laboratory comparison or proficiency-testing program;

7.1.c replicate tests using the same or different methods;

7.1.d retesting of retained samples; and

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

7.1.1 Essential Quality Control Procedures

These general QC principles shall apply, where applicable, to all testing laboratories. The way they are implemented is dependent on the types of tests performed by the laboratory and are further described in this module. The standards for any given test type shall assure that the applicable principles are addressed:

7.1.1.a All laboratories shall have detailed written protocols in place to monitor the following QCs:

7.1.1.a.i positive and negative controls to monitor tests such as blanks, spikes, reference toxicants;

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

7.1.1.a.iii measures to evaluate method capability (e.g., percent minimum significant difference (PMSD));

7.1.1.a.iv selection of appropriate formulae to reduce raw data to final results such as regression and statistical analyses;

7.1.1.a.v selection and use of reagents and standards of appropriate quality;

7.1.1.a.vi measures to assure the selectivity of the test for its intended purpose; and

7.1.1.a.vii measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the reference method such as temperature, humidity, light, or specific equipment conditions.

7.1.1.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.

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

7.1.1.d The QC protocols specified by the laboratory's method manual shall be followed. The laboratory shall ensure that the essential standards outlined in this document or regulations (whichever are more stringent) are incorporated into their method manuals. When it is not apparent which is more stringent, the QC in the regulations is to be followed.

7.1.2 Positive and Negative Controls

7.1.2.a Positive Control. Reference toxicant tests demonstrate a laboratory's ability to obtain consistent results with the reference method and evaluate the overall health and sensitivity of test organisms over time.

7.1.2.a.i The laboratory shall demonstrate its ability to obtain consistent results with standard reference toxicants (SRT).

7.1.2.a.ii Ongoing laboratory performance shall be demonstrated by performing routine SRT testing for each method, species, and endpoint in accordance with the minimum frequency requirements specified in Section 7.1.2.a.iii.

7.1.2.a.iii The frequency of ongoing laboratory reference toxicant testing shall be as follows unless the reference method specifically requires less frequent SRT

tests (e.g., sediment tests).

For methods conducted at a frequency of monthly or greater, SRT tests shall be conducted monthly.

For methods and species commonly used in the laboratory, but which are tested at a frequency of less than monthly, SRT tests shall be conducted concurrently with the environmental test.

If the test organisms are obtained from an outside source, the sensitivity of each batch of organisms received from a supplier shall be determined via a concurrent SRT test unless the supplier can provide control chart data for the last five SRT tests using the same SRT and test conditions. Supplied SRT data may not be older than six months.

7.1.2.a.iv These standards do not currently specify a particular reference toxicant and dilution series. However, if the regulation identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements. All reference toxicant tests conducted for a given method and species shall use the same reference toxicant, test concentrations, dilution water and data analysis methods. A dilution factor of 0.5x or greater shall be used for both acute and chronic tests.

7.1.2.a.v The reference toxicant tests shall be conducted following the procedures required in the reference method.

7.1.2.b Negative Controls – Control, Brine Control, Control Sediment, Control Soil, and Dilution Water

7.1.2.b.i The standards for the use, type, and frequency of testing of negative controls are specified by the reference 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.

7.1.2.b.ii Appropriate additional negative controls shall be included when sample adjustments (for example addition of thiosulfate for dechlorination) or solvent carriers are used in the test.

7.1.3 Variability and/or Reproducibility

Intra-laboratory precision shall be determined on an ongoing basis using further reference toxicant tests and related control charts as described above.

7.1.4 Test Sensitivity

7.1.4.a The PMSD shall be calculated according to the formula specified by the reference method and reported with the test results.

7.1.4.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.

7.1.5 Selection and Use of Reagent and Standards

7.1.5.a The grade of all reagents used in toxicity tests is specified in the reference method except the reference standard. All reference standards shall be prepared from chemicals that are analytical reagent grade or better. Records shall be maintained of the preparation of all standards and reference toxicants.

7.1.5.b All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH, or specific conductance, shall comply with Module 4.

7.1.5.c Only reagent-grade water collected from distillation or de-ionization units is used to prepare reagents.

7.1.6 Constant and Consistent Test Conditions

7.1.6.a If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid cross-contamination.

7.1.6.b Laboratory space shall be adequate for the types and numbers of tests performed. The building shall provide adequate cooling, heating, and illumination for conducting testing and culturing. Hot and cold running water shall be available for cleaning equipment.

7.1.6.c Air used for aeration of test solutions, dilution waters and cultures shall be free of oil and fumes.

7.1.6.d The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s)) and the names(s) of the taxonomic expert(s) shall be kept on file at the laboratory. When organisms are obtained from an outside source, the supplier shall provide this same information.

7.1.6.e Equipment used for routine support measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, ammonia, and weight shall be calibrated, and/or standardized per manufacturer's instructions. Records shall be maintained of all measurements and calibrations.

7.1.6.f Test temperature shall be maintained as specified by the reference method. Temperature control equipment shall be adequate to maintain the required test temperature(s). The average daily temperature of the test solutions shall be maintained within method specified range. The minimum frequency of measurement shall be once per 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.

7.1.6.g Reagent grade water, prepared by any combination of distillation, reverse osmosis, ion exchange, activated carbon and particle filtration, shall meet the reference method requirements.

7.1.6.h The quality of the standard dilution water used for testing or culturing shall be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. Water used for culturing and testing shall be analyzed for toxic metals and organics whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified.

7.1.6.i The quality of the food used for testing or culturing shall be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. The laboratory shall have procedures for the evaluation of food acceptance.

7.1.6.j A subset of organisms used in bioaccumulation tests shall be analyzed at the start of the test (i.e., baseline) for the target compounds to be measured in the bioaccumulation test.

7.1.6.k Test chamber size and test solution volume shall be as specified in the reference method. All test chambers used in a test shall be identical.

7.1.6.l Test organisms shall be fed the quantity and type of food or nutrients specified in the reference method. The organisms shall also be fed at the intervals specified in the reference method.

7.1.6.m All organisms in a test shall be from the same source and lot. Where available, certified seeds shall be used for soil tests.

7.1.6.n All organisms used in tests or used as broodstock to produce neonate test organisms (e.g., cladocerans and larval fish), shall 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.

7.1.6.o All materials that contact test samples (e.g., test chambers, culture tanks, tubing, solutions, control water, sediment, soil, or food) shall be non-toxic and cleaned as described in the reference method. Materials shall not reduce or add to sample toxicity. Appropriate materials for use in toxicity testing and culturing are described in the reference methods.

7.1.6.p Light intensity shall be maintained as specified in the reference method. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the reference method and shall be checked and recorded at least quarterly. For algal and plant tests, the light intensity shall be measured and recorded at the start of each test.

7.1.6.q The laboratory shall maintain records of the health and culturing conditions of all organisms used for testing. These records 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 records of these water quality parameters and biological observations for each lot of organism received. These observations shall adequately address the 24-hour time period referenced in item 7.1.6 n) above. The laboratory shall

also record each of these observations and water quality parameters upon the arrival of the organisms at the testing laboratory.

7.1.6.r Age and the age range of the test organisms shall be as specified in the reference method. Supporting information, such as hatch dates and times, times of brood releases and metrics (for example, chironomid head capsule width) shall be recorded.

7.1.6.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 specified by the reference method and approved by the regulatory agency having authority for program oversight.

7.1.6.t All tests shall have at least the minimum number of replicates per treatment as specified by the reference method.

7.1.6.u The control population of Ceriodaphnia in chronic effluent or receiving water tests shall contain no more than 20% males.

7.1.6.v The culturing of C. dubia shall be adequate such that blocking by parentage can be established.

7.1.6.w Dissolved oxygen and pH in aquatic tests shall be within acceptable range at test initiation. Minimal aeration is provided to tests if acceptable dissolved oxygen concentrations cannot be otherwise maintained.

7.1.6.x Test soils or sediments shall be within the geochemical tolerance range of the test organism.

7.1.6.y An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH, or 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 reference method). The acceptability of the test shall depend on the experience and professional judgment of the technical director and the permitting authority.

7.2 Data Acceptance/Rejection Criteria

7.2.1 Positive Controls

A laboratory shall record the control performance and statistical endpoints for each method and species on control charts. The laboratory shall also evaluate precision (i.e., coefficient of variation, CV) for these tests against reference method or laboratory-derived criteria to determine validity of the testing result.

For endpoints that are point estimates (e.g., lethal concentration (LCp), inhibition concentration (ICp), or effective concentration (ECp)), control charts are constructed by plotting the cumulative mean and the control limits, which consist of the upper and lower 95% confidence limits (± 2 standard deviations). For endpoints from hypothesis tests (e.g., no observed effect concentration (NOEC) or no observed adverse effect concentration (NOAEC)) the values are plotted directly, and the control limits consist

of one concentration interval above and below the concentration representing the central tendency (i.e., the mode).

For endpoints that are point estimates the cumulative mean CV is calculated. For endpoints from hypothesis tests, the PMSD is calculated. These values are maintained on control charts.

Control chart limits are expected to be exceeded occasionally regardless of how well a laboratory performs. Acceptance limits for point estimates that are based on 95% confidence limits should theoretically be exceeded for one in twenty tests. Depending on the dilution factor and test sensitivity, control charts based on hypothesis test values (NOEC, NOAEC) may be expected to be exceeded on a similar frequency. Test results that fall outside of control chart limits at a frequency of 5% or less, or which fall just outside control chart limits (especially in the case of highly proficient laboratories which may develop relatively narrow acceptance limits over time), are not rejected de facto. Such data are evaluated in comparison with control chart characteristics including the width of the acceptance limits and the degree of departure of the value from acceptance limits.

Laboratories shall develop acceptance/rejection policies, consistent with the reference methods, for SRT data which considers source of test organisms, the direction of the deviation, test dilution factor, test sensitivity (for hypothesis test values), testing frequency, out-of-control test frequency, relative width of acceptance limits, inter-test CV, and degree of difference between test results and acceptance limits.

In the case of reference toxicant data which fail to meet control chart acceptance criteria, the test data are examined for defects, corrective action taken, and the test repeated if necessary, using a different batch of organisms or the data is qualified.

Intra-laboratory precision is determined on an ongoing basis using control charts. The control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC50 for acute tests, or as appropriate hypothesis test values, such as the NOEC or NOAEC, over time within a laboratory.

7.2.2 Negative Controls

The test acceptability criteria specified in the reference method shall be achieved for both the reference toxicant and the effluent or environmental sample toxicity test. The criteria shall be calculated and shall meet the reference method requirements for performing toxicity tests.

7.2.3 Selection of Appropriate Statistical Analysis Methods

7.2.3.a Methods of data analysis and reporting as specified by language in the regulation, permit, or the reference method shall be followed.

7.2.3.b Toxicity data shall be plotted on semi-logarithmic graph paper, relating time, mortality, and effluent concentration to verify computational results.

7.2.4 Sample Handling

All samples shall be chilled to 0-6 °C during or immediately after collection except as specified by the reference method and approved by the regulatory agency having authority for program oversight.

Module 8

Quality Systems for Industrial Hygiene Testing

1.0 Introduction

This module provides specific requirements for laboratories seeking accreditation for industrial hygiene (IH) testing. Laboratories seeking accreditation for IH testing shall also meet the requirements presented in Module 2 of this standard and the applicable IH requirements found in Appendix B. IH laboratories seeking accreditation for asbestos testing shall also meet the requirements presented in Module 3 of this standard. Accreditation for IH testing is to matrix-method-technology (instead of analyte-matrix-method) and proficiency testing is performed by field of testing (e.g., GC/ECD, GC/FID, IC, CVAA, GC/MS, ICP-AES, ICP/MS, HPLC, LC/MS, etc.).

2.0 Scope

This module provides quality assurance (QA) and quality control (QC) requirements for laboratories seeking to obtain and maintain accreditation for industrial hygiene testing methods. This module is not intended to relieve additional quality control requirements that are specified by method, regulation, or customer.

3.0 Terms and Definitions

Definitions are considered requirements. Definitions are included in Module 2, Section 3.0. Additional definitions related to this module are listed below.

Detection Limit: The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration.

Field of Testing: The field of testing is identified by the technology used for the testing.

Limit of Detection: The lowest concentration empirically determined to consistently provide a response meeting identification and minimum signal requirements for a specific analyte-matrix-method combination.

Limit of Quantitation (Reporting Limit): The smallest concentration that produces a quantitative result within documented acceptance criteria. The LOQ shall be set at or above the concentration of the lowest non-zero initial calibration standard and within the calibration range.

4.0 Proficiency Testing

For all Fields of Testing (FoT) in a laboratory's scope of accreditation, the laboratory shall demonstrate proficiency by completing one of the proficiency demonstrations listed below. The proficiency demonstrations are listed in priority order.

4.1 External Proficiency Testing Program

The laboratory shall participate in an external proficiency testing (PT) program in accordance with the American Industrial Hygiene Association Laboratory Accreditation Programs, LLC (AIHA LAP) policy module on proficiency testing.

4.1.1 Proficiency testing providers shall be accredited to ISO/IEC 17043. If an ISO/IEC 17043 accredited PT provider is not available, a non-accredited PT provider may be used with approval of the accrediting body.

4.1.2 Proficiency testing shall be performed using the same preparation, analytical procedure, and instrumentation combination used to test customer samples.

4.1.3 To obtain initial accreditation, the laboratory shall participate in and pass two consecutive rounds of PT per FoT.

4.2 External Proficiency Testing Program Not Available

4.2.1 When an external PT program is not available the laboratory shall meet PT requirements by participating in a round robin study in accordance with the AIHA LAP policy module on proficiency testing.

4.2.2 The laboratory shall submit in writing to its DoD-ELAP and/or DOECAP-AP accrediting body (AB) a list of items on its scope of accreditation for which an external PT program is not available.

4.2.3 The laboratory shall have procedures for participation in round robin studies. The procedure shall describe the schedule and acceptance criteria for round robin studies. When results are unacceptable, the laboratory shall implement its nonconforming work procedure.

4.2.4 The laboratory shall maintain records of the round robin studies for each FoT on its scope that uses round robin studies to meet PT requirements.

4.2.5 These requirements shall be met until an external PT program is available.

4.2.6 The laboratory shall achieve a history of two successful round robin studies out of the most recent three attempts for each FoT.

Note: If the laboratory has two consecutive acceptable round robin studies, a third study is not needed.

4.2.7 To obtain initial accreditation, the laboratory shall participate in and pass two consecutive round robin studies per FoT.

4.3 No Round Robin Studies

When an external PT program is not available and a round robin study is prohibited, proprietary, or impractical, then the laboratory shall meet PT requirements by participating in an internal PT program in accordance with the AIHA LAP policy module on proficiency testing.

4.3.1 The laboratory shall submit in writing to its DoD ELAP AB and/or its DOECAP-AP AB a list of items on its scope of accreditation for which an external PT is not available

and a round robin study is prohibited, proprietary, or impractical.

4.3.2 The laboratory shall have a procedure for the internal PT program that includes spiking procedures, frequency, responsibility for implementation, statistical treatment of resultant data, acceptance criteria, and actions to be taken in the event of an unacceptable result.

4.3.3 The laboratory shall maintain records of compliance with the internal PT program for each FoT on its scope that uses an internal PT program to meet PT requirements.

4.3.4 These requirements shall be met until a round robin study, or an external PT program, is available.

4.3.5 To obtain initial accreditation, the laboratory shall pass two consecutive rounds of internal PT per FoT.

4.4 No Internal PT Program

When an external PT program is not available, a round robin study is prohibited, proprietary, or impractical, and participating in an internal PT program is impractical then the laboratory may be permitted to meet PT requirements by demonstrating proficiency through the implementation of an internal QC program in accordance with the AIHA LAP policy module on proficiency testing.

4.4.1 The laboratory shall obtain concurrence from its DoD ELAP AB and/or its DOECAP-AP AB to meet PT requirements by demonstrating proficiency through the implementation of an internal QC program.

4.4.2 The laboratory shall have a procedure for the internal QC program that includes schedule and frequency of evaluation, identification of QC samples evaluated, acceptance criteria, and the actions to be taken in the event of an unacceptable result.

4.4.3 The laboratory shall maintain records of compliance with the internal QC program evaluation for each FoT on its scope that uses an internal QC program meet PT requirements.

4.4.4 To obtain initial accreditation, the laboratory shall pass two consecutive rounds of internal PT per FoT.

5.0 Method Selection

The requirements in the Module 2 section on “Selection, Verification and Validation of Methods” apply.

When adding a new analyte to a reference method, the inclusion of the analyte in the method shall meet all required calibration requirements and the QC requirements of the method to which the analyte is being added. If no QC requirements exist in the method, the laboratory shall adhere to the requirements outlined in a reference method of the same technology (when available). When adding an analyte, the laboratory shall follow the requirements of the relevant regulations to determine whether the addition of an analyte represents a method modification.

6.0 Method Validation

6.1 Validation of Methods

Before acceptance and institution of any method for which data will be reported, all methods shall be validated.

6.1.1 Method validation shall meet the requirements in the Module 2 Section on "Selection, Verification, and Validation of Methods" as well as all criteria in this Module.

6.1.2 The laboratory shall validate reference methods through the initial determinations of a detection limit (DL) if required, a limit of detection (LOD) if required, and a limit of quantitation (LOQ) as well as an Initial Demonstration of Capability (DOC). Requirements for DL, LOD and LOQ are contained in this module's section on "Detection Limits, Limits of Detection, and Limits of Quantitation." If the reference method has additional validation requirements, these requirements shall also be met.

6.1.3 In addition to the QC procedures for reference methods, the laboratory shall validate modified reference methods and non-reference methods (including laboratory-developed methods) using QC procedures and acceptance criteria that are consistent with those of similar reference methods or technologies, and the validation shall include the following:

6.1.3.a scope;

6.1.3.b calibration verification;

6.1.3.c interferences and cross-contamination;

6.1.3.d analyte identification;

6.1.3.e analyte quantitation;

6.1.3.f selectivity;

6.1.3.g sensitivity; and

6.1.3.h precision and bias.

6.1.4 The use of any modified or non-reference method shall be approved by the customer before use.

6.1.5 Methods shall be validated when substantive modifications are made to reference methods (e.g., stoichiometry, technology, mass tuning acceptance criteria, quantitation ions, compressing digestion or extraction timeframes, reducing reagent or solvent volumes, changing solvents, or compressing instrument runtimes).

6.1.6 When a modification of a method includes changes to bulk sample (e.g., soil, paint chips) preparation steps, the validation process shall include analysis of field samples in the matrix of concern. Validation shall include, where possible, parallel studies using the reference method versus the modified method.

6.1.6.a The field samples shall contain target analytes either found natively in the samples or spiked into the sample. Validation shall include multiple levels of target analyte concentrations. In this context, “matrix of concern” means samples that are like, or from specific sampling sites, in which the method will be used.

6.1.6.b Where modifications to only the analytical portion of the method are planned, the laboratory shall take into consideration any effects the matrix may have on the analysis as part of its risk assessment.

6.2 Detection Limit, Limit of Detection, and Limit of Quantitation

For each analyte in each field of testing, the laboratory shall have procedures for determining and verifying DL, LOD, and LOQ that reflect current operating conditions. DL, LOD, and LOQ determinations are not required for methods such as gravimetric or asbestos. DL and LOD determinations are not required if results are not reported below the LOQ, unless required by regulation or method.

For each preparation method listed on the scope of accreditation, the laboratory shall determine a DL, LOD, and LOQ, unless it falls within one of the stated exceptions. Although the laboratory is not required to determine a separate DL, LOD and LOQ for all possible combinations of preparation and cleanup techniques in use, the laboratory shall determine the DL, LOD, and LOQ using the combination of processes most likely to interfere with sensitivity (i.e., preparation method with all applicable cleanup/preparation steps).

The DL, LOD and LOQ shall be reported for each analyte in each field of testing unless it is not applicable to the test or specifically excluded by customer requirements.

Records of all supporting data for DL, LOD, and LOQ determinations shall be maintained.

6.2.1 Determination of the Detection Limit

When required to establish a DL, the laboratory shall use published methodologies from recognized entities such as USEPA, USDOE, ASTM, or NIOSH, or the DL may be established based on historical data.

6.2.2 Initial Determination of the Limit of Detection

The LOD determination procedure shall address the following requirements:

6.2.2.a After each DL determination, the laboratory shall establish the LOD by spiking a quality system matrix at a concentration greater than or equal to the DL. The LOD is equal to the concentration of this spike.

6.2.2.b The apparent signal to noise (S/N) ratio at the LOD shall be at least three and the results shall meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition).

6.2.2.c For data systems that do not provide a measure of noise, the signal produced by the LOD spike shall produce a response that is at least three standard deviations greater than the mean Method Blank concentration.

6.2.2.d The mean Method Blank is initially estimated based on a minimum of four Method Blank analyses and later established with a minimum of 20 Method Blank results.

6.2.2.e If the LOD spike response does not meet the requirements, the laboratory shall repeat the DL and/or LOD determination at a higher level, or implement its nonconforming work procedure, until the requirements are met.

6.2.3 Ongoing Verification of the Limit of Detection

The LOD verification procedure shall address the following requirements:

6.2.3.a The laboratory shall verify the LOD annually, at a minimum. The verification is made by repeating the LOD spike process at a concentration that is greater than or equal to half the current LOD and less than or equal to double the current LOD (i.e., 1/2x LOD ≤ Ongoing LOD Spike ≤ 2x LOD) provided the ongoing spike concentration is greater than or equal to the DL. The Ongoing LOD verification shall meet the same acceptance criteria as the Initial LOD verification for signal-to-noise and analyte identification. If the acceptance criteria are met, the Initial LOD is verified and may continue to be used.

6.2.3.b In the event the verification fails, the laboratory shall redetermine the LOD and, if necessary, the DL, or implement its nonconforming work procedure, until the requirements are met.

6.2.3.c If the method is altered in a way other than routine maintenance, and the change can be expected to elevate the detection limit, the LOD shall be reverified using the Ongoing LOD verification procedure.

6.2.3.d If there are multiple instruments that will be assigned the same LOD, then the LOD verification spike shall meet the requirements on each instrument.

6.2.3.e In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform ongoing LOD verifications on a one-per-batch basis, before sample analysis, in lieu of annual verification. The verification data shall meet the requirements of this section and be reported to the customer.

6.2.4 Initial and Ongoing Verification of the Limit of Quantitation

The LOQ verification procedure shall address the following requirements:

6.2.4.a For methods using multi-level calibration, the laboratory shall select an LOQ for each analyte that is greater than or equal to the LOD and the lowest non-zero calibration standard, but no greater than 10 times the LOD (i.e., LOD ≤ LOQ < 10x LOD). For methods using a single-point calibration, the LOQ shall be greater than or equal to the LOD and greater than or equal to the low-level calibration check standard, but no greater than 10 times the LOD.

6.2.4.b The LOQ shall be verified through analysis of verification samples. The LOQ verification sample consists of a spiked quality system matrix greater than or equal to the LOD or one-half the LOQ, whichever is less, and less than or equal to double the LOQ (i.e., LOD or 1/2 LOQ ≤ LOQ spike ≤ 2x LOQ). The LOQ verification shall meet the same criteria as the initial LOD verification for signal-to-noise and analyte identification and shall be within the laboratory's stated acceptance criteria. The laboratory acceptance criteria shall be determined based on a maximum of three standard deviations from the mean of historical data, but no wider than the laboratory control sample (LCS) acceptance criteria with an additional 20% allowance above and below. Additionally, the lower limit shall be greater than or equal to 10% recovery.

6.2.4.c In the event the verification fails, the laboratory shall redetermine the LOQ and, if necessary, the DL and/or LOD; or implement its nonconforming work procedure, until the requirements are met.

6.2.4.d If there are multiple instruments that will be assigned the same LOQ, then verification shall be performed on each instrument.

6.2.4.e The laboratory shall verify the LOQ annually, at a minimum.

6.2.5 The laboratory shall provide the following when DL, LOD, and LOQ summary information is requested:

6.2.5.a indication of which analyte/matrix/prep method/analytical method and instrument used;

6.2.5.b DL;

6.2.5.c claimed LOD;

6.2.5.d concentration of initial LOD spike and verification spike, if different;

6.2.5.e statement of compliance with analyte identification requirements;

6.2.5.f signal-to-noise value or statement of compliance with requirements;

6.2.5.g claimed LOQ;

6.2.5.h concentration of LOQ spike;

6.2.5.i recovery or result of LOQ spike;

6.2.5.j acceptance criteria at the LOQ; and

6.2.5.k if specifically requested, raw data to support parameters reported.

6.3 Evaluation of Method Precision and Bias

6.3.1 The laboratory shall evaluate the precision and bias of a method for each analyte of concern for each quality system matrix or follow a documented alternate procedure

when the analyte cannot be spiked into the sample matrix and QC samples are not commercially available.

6.3.2 The laboratory shall have a procedure for determining precision and bias.

6.3.3 The laboratory shall process the samples through the entire measurement system for each analyte of interest.

6.3.4 Precision and bias measurements shall evaluate the method across the analytical calibration range of the method.

6.3.5 The laboratory shall compare results of the precision and bias measurements with criteria established by the customer, criteria given in the reference method, and/or criteria established by the laboratory.

6.4 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, inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, and electrode response factors.

7.0 Demonstration of Capability (DOC)

7.1 General

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

7.1.2 Thereafter, the individual shall perform ongoing DOC.

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

7.1.4 All data applicable to the DOC shall be retained and readily available at the laboratory.

7.2 Initial DOC

Each individual shall successfully perform an initial DOC before using any method, any time there is a change in instrument type or method that could potentially affect the precision and bias, sensitivity, or selectivity of the output, or any time that a method has not been performed by the individual in a 12-month period.

Note: Examples of method changes that could potentially affect the precision and bias, sensitivity, or selectivity of the output include a change in the detector, column

type, matrix, method revision, or other components of the sample measurement system.

7.2.1 The laboratory shall have a procedure for performing an initial DOC.

7.2.2 The laboratory shall maintain records of each initial DOC in a manner such that the following information is readily available for each individual:

7.2.2.a individual(s) involved in preparation and/or analysis;

7.2.2.b matrix;

7.2.2.c analyte(s), class of analyte(s);

7.2.2.d identification of method(s) performed;

7.2.2.e identification of laboratory-specific procedure used for analysis, including revision number;

7.2.2.f date(s) of analysis; and

7.2.2.g summary of analyses.

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

7.2.3.a The analyte(s) shall be spiked in a volume of clean quality system matrix (i.e., a matrix in which no target analytes or interferences are present at concentrations that will impact the results of a specific method) sufficient to prepare four aliquots at the concentration specified in the reference method, or if unspecified, to a concentration of one to four times the LOQ.

7.2.3.b At least four aliquots shall be prepared and analyzed according to the method.

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

7.2.3.d Compare the information from 7.2.3.c above to the corresponding acceptance criteria for precision and accuracy in the method, if applicable, or in laboratory-generated acceptance criteria if there are not acceptance criteria in the method. If all analytes meet the acceptance criteria, the analysis of actual samples may begin. If any one of the analytes does not meet the acceptance criteria, the performance is unacceptable for that analyte.

7.2.3.e When one or more of the tested analytes fail at least one of the acceptance criteria, the analyst shall proceed according to i) or ii) below:

7.2.3.e.i Locate and correct the source of the failure and repeat the DOC procedure for

all analytes of interest.

7.2.3.e.ii Repeat the DOC procedure for all analytes that failed to meet criteria.

7.2.3.f Repeated failure, however, confirms a general problem with the measurement system. If repeated failure occurs, locate and correct the source of the failure, and repeat the DOC procedure for all analytes.

7.2.3.g When an analyte not currently found on the laboratory's list of accredited analytes is added to an existing accredited method, an initial demonstration shall be performed for that analyte.

7.3 Ongoing DOC

7.3.1 The laboratory shall have a procedure for ongoing DOC that includes how the laboratory will identify data associated with ongoing DOCs. The individual shall demonstrate on-going capability by routinely meeting the QC requirements of the reference method, laboratory procedure, customer requirements, and/or this standard. If the method has not been performed by the individual in a 12-month period, an initial DOC shall be performed. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

7.3.2 This on-going demonstration may be one of the following:

7.3.2.a acceptable performance of a blind sample (single blind to the individual) or blind PT sample on a similar method using the same technology;

Note: Acceptable results for both detected and non-detected analytes are considered acceptable performance.

7.3.2.b another initial DOC;

7.3.2.c at least four consecutive LCSs with acceptable levels of precision and accuracy. The laboratory shall determine the acceptable limits for precision and accuracy before analysis. The laboratory shall tabulate or be able to readily retrieve four consecutive passing LCSs or reference samples for each method for each individual performed within the last 12-month period;

7.3.2.d following a procedure for reviewing records of QC samples meeting the QC requirements of the method, laboratory procedure, customer requirements, and/or this standard. A review of these record may be used to identify patterns and determine if corrective action or retraining is necessary; or

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

8.0 Technical Requirements

8.1 Calibration

This section specifies the essential elements that shall define the procedures and required records for initial calibration and continuing calibration verification for methods that use calibration models including, but not limited to, average response factor or linear or quadratic regression, to ensure that the data shall be of known quality for the intended use.

Calibration requirements for auxiliary equipment are specified in Module 2. This section does not specify detailed procedural steps for calibration but does establish the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which requirements are more stringent, then the requirements of the regulation or mandated method shall be followed.

8.1.1 Initial Calibration

Each reported analyte shall be associated with an acceptable initial calibration. If the initial calibration is not acceptable, corrective actions shall be performed and all associated samples reanalyzed. The following items are required elements of initial calibration:

8.1.1.a The details of the initial calibration including calculations, integrations, acceptance criteria, and associated statistics shall be included or referenced in the procedure. When initial calibration procedures are referenced then the referenced procedures shall be retained by the laboratory.

8.1.1.b Sufficient raw data records shall be retained to permit reconstruction of the initial calibration (e.g., calibration date, method, unique instrument identification, analysis date, each analyte name, and individual initials or signature; concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration).

8.1.1.c The laboratory shall use the most recent initial calibration analyzed prior to the analytical batch, unless otherwise specified by the method.

8.1.1.d Standards used for calibration shall be Certified Reference Materials specifically identified as such in an accompanying Certificate of Analysis from a Reference Material Producer (RMP) accredited to ISO 17034 or Standard Reference Materials (SRM) from a National Metrology Institute (NMI), when commercially available. If standards are not commercially available from an United States of America or Canada-based RMP, then the laboratory shall use standards from an authoritative source.

8.1.1.e The laboratory shall have a written procedure addressing removal and replacement of calibration standards. The procedure shall comply with the following requirements:

8.1.1.e.i The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of individual analytes in interior levels is not permitted.

8.1.1.e.ii The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. A laboratory that chooses to remove a calibration level from the interior of the calibration shall remove that calibration level for all analytes. Removal of calibration level from the interior of the curve shall not to be used to compensate for lack of maintenance or repair to the instrument.

8.1.1.e.iii The laboratory shall adjust the LOQ and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.

8.1.1.e.iv The laboratory shall ensure that the remaining initial calibration levels are sufficient to meet the minimum requirements for the number of initial calibration levels as mandated by this standard, the method, and/or regulatory requirements.

8.1.1.e.v The laboratory may replace a calibration level provided that:

8.1.1.e.v.a the laboratory analyzes the replacement standard within 24 hours of the original calibration standard analysis for that particular calibration level;

8.1.1.e.v.b the laboratory replaces all analytes of the replacement calibration standard if a level within the interior of the calibration is replaced; and

8.1.1.e.v.c the laboratory shall limit the replacement to one calibration level.

8.1.1.e.vi The laboratory shall record a technically valid reason for either removal or replacement of any interior calibration level.

8.1.1.f For regression or average response/calibration factor calibrations, the minimum number of non-zero calibration standards shall be as specified in the table below;

Type of Calibration Curve	Minimum Number of Calibration Standards[b,c]
Threshold Testing[a]	1
Average Response	5
Linear Fit	5
Quadratic Fit	6

[a]The initial one-point calibration shall be at the threshold level provided by the customer and results shall be reported qualitatively with uncertainty, and in compliance with a decision rule.

[b]Fewer calibration standards may be used only if equipment firmware or software cannot accommodate the specified number of standards. Records detailing that limitation shall be maintained by the laboratory.

[c]Ion-selective electrode analyses (e.g., pH, ammonia) are not covered by this table. The laboratory shall use the minimum number of standards as recommended or

required in the reference method, or manufacturer's instructions.

8.1.1.g The lowest non-zero calibration standard shall be at or below the lowest concentration for which quantitative data are to be reported without qualification.

8.1.1.h The highest calibration standard shall be at or above the highest concentration for which quantitative data are to be reported without qualification. When sample responses exceed the calibration range, the laboratory shall dilute and reanalyze the sample to bring results within the calibration range, if sufficient sample volume and holding time permit. If results are outside the calibration range and reanalysis is not possible, report with appropriate data qualifiers.

8.1.1.h.i For methods utilizing inductively coupled plasma analysis, the laboratory may report a sample result with a response above the calibration range if the laboratory analyzes and passes (within 10% of the true value) a high-level check standard that exceeds the sample concentration but is within the linear dynamic range (provided the high-level check standard is analyzed in the same manner as the sample and evaluated with the same calibration).

8.1.1.i Sample results shall be quantitated from the initial calibration and may not be quantitated from any continuing calibration verification unless otherwise required by regulation, method, or program.

8.1.1.j Criteria for the acceptance of an initial calibration shall be documented (e.g., correlation coefficient or relative standard deviation).

8.1.1.k When procedures are employed that specify calibration with a single calibration standard and a zero point (blank or zero, however specified by the procedure), the following shall occur:

8.1.1.k.i The zero point and single calibration standard within the linear range shall be analyzed at least daily and used to establish the slope of the calibration.

8.1.1.k.ii To verify adequate sensitivity a standard shall be analyzed at or below the lowest concentration for which quantitative data are to be reported without qualification. This standard shall be analyzed before sample analysis with each calibration and shall meet the recovery acceptance criteria at the LOQ established by the method. If no criteria exist, the laboratory shall specify criteria in the procedure.

8.1.1.l For analysis of Aroclors which use a linear through origin model (or average response factor) the laboratory shall perform an initial multi-level calibration for a subset of Aroclors (e.g., a mixture of 1016/1260) and use a one-point initial calibration for pattern recognition for the remaining Aroclors. If one of the remaining Aroclors is identified, a multi-level calibration shall be performed for the specific Aroclor detected, and the sample extract reanalyzed using the multi-level calibration for quantitation.

8.1.1.m The laboratory shall verify all initial calibrations with an initial calibration verification standard (ICV) prepared from materials from an authoritative, independent, second source before analyzing samples. The use of a calibration

material from a second lot obtained from the same manufacturer, independently prepared from different source materials, is acceptable for use as a second-source ICV. The concentration of the second-source ICV shall be at the midrange or lower. When using neat materials for calibration, the second-source ICV may be an independent preparation of the neat material used for calibration. The calibration verification shall meet the acceptance criteria of the reference method, or if not specified in the reference method, the acceptance criteria for continuing calibration verification shall be used.

8.1.1.n For those methods where reporting non-detected analytes based on successful completion of a sensitivity check is allowed (similar to threshold testing but only for non-detects), the requirements of this standard shall not prohibit the practice.

8.1.2 Continuing Calibration Verification

The validity of the initial calibration shall be verified before sample analyses by a calibration verification with each analytical batch. The following items are essential elements of continuing calibration verification.

8.1.2.a The details of the continuing calibration verification procedure, calculations, and associated statistics shall be included or referenced in the procedure.

8.1.2.b Calibration shall be verified for each compound, element, or other discrete chemical species, except for multi-component analytes such as Aroclors, chlordane, total petroleum hydrocarbons, or toxaphene, where a representative chemical, related substance or mixture may be used.

8.1.2.c The concentration of the continuing calibration verification sample (CCV) shall be greater than or equal to the low calibration standard and less than or equal to the mid-range.

8.1.2.d Instrument calibration verification shall be performed at the beginning and end of each analytical batch, and at the frequency defined in the method except:

8.1.2.d.i a second source initial calibration verification that passes the continuing calibration verification criteria may be used in place of CCV;

8.1.2.d.ii a LCS may be used in place of a CCV (but not as a replacement for a failing CCV) for methods where the calibration goes through the same process as the LCS (using the continuing calibration verification acceptance criteria).

8.1.2.e Sufficient raw data records shall be retained to permit reconstruction of the calibration verification (e.g., method, unique instrument identification, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations). Continuing calibration verification records shall explicitly connect the continuing calibration verification data to the initial calibration.

8.1.2.f Criteria for the acceptance of a continuing calibration verification shall be established. If the continuing calibration verification results obtained are outside

the established acceptance criteria, the following steps shall be taken:

8.1.2.f.i If a cause for the calibration verification failure is identified that impacts only the CCV (e.g., a missed autosampler injection), then analysis may proceed if a second CCV is analyzed immediately (within one hour and no samples analyzed) and the result is within acceptance criteria (i.e., passing). Samples analyzed previously shall be considered valid if bracketed by a passing CCV. The laboratory shall maintain records of the cause for the failure of the first calibration verification result.

8.1.2.f.ii If a cause for the calibration verification failure is not isolated to the CCV or not identified, then the laboratory shall implement its nonconforming work procedure and repeat the CCV and all associated samples since the last successful CCV.

8.1.2.f.iii Qualifying data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory shall notify the customer before reporting data associated with a failed CCV.

8.1.2.f.iv Data associated with an unacceptable CCV shall be qualified if reported but shall not be reported if prohibited by the customer, a regulatory program or regulation. Data associated with CCVs that fail under the following special conditions shall still be qualified, but may use a different qualifier when:

8.1.2.f.iv.a the acceptance criteria for the CCV are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with a data qualifier. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated, and accepted.

8.2 Quality Control

The laboratory shall have QC procedures for monitoring the validity of environmental tests undertaken as specified in this Section.

8.2.1 Negative Control – Method Performance: Method Blank

For many IH analyses a media blank takes the place of a method blank. For media blanks, subtraction shall be applied as described in the reference method.

8.2.1.a The method blank is used to assess the samples in the preparation batch for possible contamination during the preparation and processing steps.

8.2.1.b The method blank shall be processed along with and under the same conditions as the associated samples to include all steps.

8.2.1.c Procedures shall be in place to determine if a method blank is contaminated.

8.2.1.d Any affected samples associated with a contaminated method blank shall be reprocessed for analysis or the results reported with appropriate data qualifiers.

8.2.1.e The method blank shall be analyzed at a minimum of one per preparation batch.

8.2.1.f When no separate preparation method is used (e.g., 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, not including method blanks, LCS, matrix spikes and matrix duplicates.

8.2.1.g 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.

8.2.1.h Method blanks are not applicable for certain analyses, (e.g., pH, conductivity, flash point, and temperature).

8.2.1.i For chromatographic analyses, when samples that are extracted together are analyzed on separate instruments or in separate analytical shifts, the method blank associated with those samples (e.g., extracted with the samples) shall be analyzed on at least one of those instruments. A method blank, solvent blank, or instrument blank shall be analyzed on all other instruments on which the set of samples was analyzed to demonstrate the instrument is not contributing contaminants to the samples.

8.2.2 Positive Control – Method Performance: Laboratory Control Sample

8.2.2.a The LCS is used to evaluate the performance of the total measurement system. The LCS shall be processed along with and under the same conditions as the associated samples and include all steps.

8.2.2.b The LCS shall be analyzed at a minimum of one per preparation batch.

8.2.2.c Exceptions are allowed for those analytes for which no spiking solutions are available (e.g., pH, color, odor, temperature, dissolved oxygen, or turbidity).

8.2.2.d In instances for which no separate preparation method is used (e.g., 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, not including method blanks, LCS, matrix spikes, and matrix duplicates.

8.2.2.e The LCS shall be a quality system matrix similar to the associated samples, known to be free of analytes of interest, spiked with known concentrations of analytes.

8.2.2.f Alternatively, the LCS may consist of a media containing known and verified concentrations of analytes or a Certified Reference Material. All analyte concentrations shall be within the calibration range of the methods.

8.2.2.g The components to be spiked shall be as specified by the reference method or regulation, or as requested by the customer. In the absence of specified spiking components, the laboratory shall spike as follows:

8.2.2.g.i All reported analytes shall be spiked in the LCS (with the exception of Aroclor analysis, which is spiked per the method). This may require the preparation of multiple LCSs to avoid interferences.

8.2.2.g.ii The concentration of the spiked compounds shall be at or below the mid-range of the calibration if customer-provided concentrations are not specified.

Note: The matrix spike may be used in place of the LCS if the acceptance criteria are as stringent as the LCS acceptance criteria.

8.2.3 Sample-Specific Controls

The laboratory shall document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of quality system matrix specific QC samples and are designed as data quality indicators for a specific sample using the designated method. These controls alone are not used to judge laboratory performance.

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

8.2.3.a Matrix Spikes and Matrix Spike Duplicates

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. A MS and MSD are usually not required in IH methods.

8.2.3.a.i Each preparation batch of samples shall contain an associated MS and MSD where required by method or regulation using the same matrix collected for the specific project unless specifically exempt by the applicable method or the applicable B-Table. The requirements for MS/MSD are not applicable to all methods. If adequate sample material is not available, then the lack of MS/MSDs shall be noted in the case narrative, and a LCS Duplicate (LCSD) shall be used to determine precision. Additional MS/MSDs may be required by a customer.

8.2.3.a.ii The MS and MSD shall be spiked with all reported analytes (except for Aroclor analysis, which is spiked per the method).

8.2.3.b Matrix Duplicates

8.2.3.b.i Each preparation batch of samples shall contain a MD when precision is not monitored through the analysis of a MS/MSD pair. MDs 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 MD may provide a usable measure of sample homogeneity. It may also provide a measure of precision when target analytes are present. A MD is usually not required in IH methods.

8.2.3.b.ii The frequency of the analysis of a MD is as specified by the customer or method.

8.2.3.b.iii Matrix duplicates shall be performed on replicate aliquots of actual samples. The composition is usually not known.

8.2.4 Data Reduction

The laboratory shall have a procedure for data reduction (e.g., linear regression), and records shall be maintained.

8.2.5 Reagent Quality, Water Quality, and Checks

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

8.2.5.b The quality of water sources shall meet method specified documented requirements. The laboratory shall monitor that the water sources meet the requirements and shall maintain records.

8.2.5.c The laboratory shall verify the concentration of titrants in accordance with written laboratory procedures and shall maintain records.

8.2.5.d The quality (e.g., purity) specifications for all standards and reagents (including water) shall be included or referenced in procedures.

8.2.6 Selectivity

The laboratory shall validate selectivity by following the checks established within the method and/or the Appendix B IH Tables.

8.2.6.a For chromatography methods where confirmation is recommended or required in the reference method or by the customer, all results greater than the DL shall be confirmed. Confirmation techniques include further analysis using a second column with dissimilar stationary phase, using a second detector type, or by other recognized confirmation techniques. HPLC UV-Diode Array detectors are not considered confirmation for a UV detector.

8.2.6.a.i Confirmation techniques using the same detector type (e.g., second-column confirmation) shall meet the same calibration and QC criteria as the initial or primary analysis.

8.2.6.a.ii The RPD of results from the primary and confirmation technique using the same detector type shall be less than or equal to 40%.

8.2.6.a.iii If using a second column for confirmation, the laboratory shall identify the primary column for each target analyte. If results are reported from the second column due to interference, QC failure, or customer requirements, the laboratory shall discuss in the case narrative.

8.2.6.a.iv If using a mass spectrometer for confirmation, the laboratory shall have a procedure that includes acceptance criteria for selectivity and sensitivity.

8.2.6.a.v When reporting data for methods that require analyte confirmation, customer reporting requirements shall be followed. If customer requirements are not available, the laboratory shall follow the reporting requirements in the method. If the method does not include reporting requirements, the laboratory shall report the results from the primary column or detector, unless there is a scientifically valid and documented reason for not doing so, and concurrence is obtained from the customer.

8.2.6.a.vi The customer shall be notified of any results that are unconfirmed (e.g., confirmation was not performed, or confirmation was obscured by interference) and the results shall be identified in the test report using data qualifiers and described in the case narrative. Analyte presence shall only be reported if both original and confirmation signals are positive or if confirmation signal cannot be discerned from interference.

8.2.7 Desorption Efficiency

8.2.7.a The laboratory shall correct results for desorption efficiency as recommended or required in the reference method for each type of adsorbent media used in the laboratory before or concurrent with sample analysis.

8.2.7.b Unless prohibited by the reference method, the laboratory may meet this requirement by preparing its initial calibration standards on the media used for samples. When this option is used, the laboratory shall quantitate sample results directly from the initial calibration.

8.2.7.c The laboratory shall use the same lot of media as samples, where possible.

8.2.7.d Where use of the same lot of media as samples or determination of the desorption efficiency is not possible, the laboratory shall qualify results and explain in the case narrative.

8.3 Data Acceptance/Rejection Criteria

8.3.1 Negative Control – Method Performance: Method Blank

8.3.1.a While the goal is to have no detectable contaminants, each method blank exhibiting potential contamination shall be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. A method blank shall be considered contaminated if the concentration of any target analyte (chemical of concern) in the blank exceeds the LOQ or 1/10th the amount measured in any associated sample, whichever is greater.

8.3.1.b When a method blank is contaminated and background contamination is not subtracted from field sample results, the laboratory shall reprepare and analyze all affected QC and field samples processed with the contaminated blank, if sufficient sample material is available. Samples are affected if they have any detections less than 10X the amount detected in the MB. If the affected samples cannot be reprepared and analyzed, report results with a data qualifier applied to specific analytes in all samples in the associated preparatory batch.

8.3.2 Positive Control – Method Performance: Laboratory Control Sample

8.3.2.a The laboratory shall calculate results of the individual batch LCS in %R or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall include the calculation in the procedure.

8.3.2.b The laboratory shall evaluate the LCS recovery against acceptance criteria provided by the customer or listed in the reference method. For analytes that are not listed in the reference method, the laboratory shall evaluate the LCS against its laboratory-developed acceptance criteria.

8.3.2.c When LCS results are outside of acceptance criteria, the laboratory shall reprepare and analyze the LCS and all affected QC samples and field samples in the associated preparation batch for failed analytes, if sufficient material is available. If the samples cannot be reanalyzed, results may be reported with appropriate data qualifiers applied to specific analytes in all samples in the associated preparatory batch.

8.3.2.c.i When the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detect results may be reported with a data qualifier and sample repreparation and analysis is not necessary.

8.3.2.d Regardless of which limits are used for LCS evaluation, the laboratory shall develop acceptance criteria for all analytes on its scope of accreditation that:

8.3.2.d.i are statistically derived based on the laboratory's historical data, using scientifically valid and documented procedures;

8.3.2.d.ii meet the limits within the reference method if available;

8.3.2.d.iii are updated on at least an annual basis or as stated in the reference method, whichever is more frequent, and re-established after major changes in the measurement system (e.g., new instrumentation);

8.3.2.d.iv are based on at least 20 data points generated under the same measurement system;

8.3.2.d.v do not exclude failed LCS recovery data and statistical outliers from the calculation, unless there is a scientifically valid and documented reason (e.g., incorrectly made standard, instrument malfunction);

8.3.2.d.vi are not outside ± 3 times the standard deviation of the mean LCS recovery;

8.3.2.d.vii are used for trend analysis; and

8.3.2.d.viii are used for batch control, if applicable.

8.3.2.e Control charts or data analysis software shall be maintained and used to detect trends and prevent out-of-control conditions. Control limits shall be monitored at least quarterly for shifts in mean recovery, changes in standard deviation, and development of trends. The laboratory may choose representative compounds for control charts for the purpose of trend analysis. The basis for selecting representative compounds shall be documented and shall be scientifically valid.

8.3.2.f The laboratory shall review control charts at a specified frequency for out-of-control conditions and initiate corrective actions when appropriate. Data analysis software may also be used for the statistical evaluation of data for trends.

8.3.2.g The laboratory shall use its laboratory-developed LCS control limits for the purpose of trend analysis and may use those control limits as a component in estimating measurement uncertainty.

8.3.2.h Laboratory Control Sample Duplicates

8.3.2.h.i The laboratory shall evaluate the LCSD recovery and RPD using acceptance criteria provided by the customer, or if customer requirements are not provided, using the appropriate Appendix B Table. If these acceptance criteria are not available, the laboratory shall evaluate the LCSD recovery and RPD using the reference method, or if not specified in the reference method, using laboratory-developed limits.

8.3.2.h.ii If LCSD results are outside of acceptance criteria, the data shall be evaluated to determine if the source of the failure is analytical error. If so, reprepare and analyze for failed analytes in all affected QC samples and field samples in the associated preparation batch, if sufficient material is available. Otherwise, qualify specific analytes in all samples in the associated preparatory batch.

8.3.3 Sample-Specific Controls

8.3.3.a Matrix Spike; Matrix Spike Duplicate

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), RPD, or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall include in a procedure the calculation for %R, RPD or other statistical treatment used.

8.3.3.a.i The laboratory shall evaluate MS/MSD recoveries using the same acceptance criteria used for the LCS.

8.3.3.a.ii The laboratory shall evaluate the MS/MSD RPD against customer requirements, or if customer requirements are not provided, the reference method, or if not provided by the customer or reference method, the

laboratory-developed limits.

8.3.3.a.iii If MS or MSD results or MS/MSD RPD are outside the acceptance criteria, the data shall be evaluated to determine if the source(s) of failure is analytical error. If so, reprepare and analyze the MS and MSD if sufficient sample material is available. Otherwise, qualify specific analytes in the parent sample.

8.3.3.b Matrix Duplicates

The results from matrix duplicates are primarily designed to assess the homogeneity of the particular sample chosen. If that sample is homogenous, it may also describe the precision of analytical results in a given matrix. These may be expressed as RPD or another statistical treatment (e.g., absolute differences).

8.3.3.b.i The laboratory shall include in a procedure the calculation for RPD or other statistical treatments.

8.3.3.b.ii The laboratory shall evaluate the MD RPD against customer requirements, or if not specified, the reference method, or if not specified, with its laboratory-developed limits.

8.3.3.b.iii If the MD RPD is outside the acceptance criteria, the data shall be evaluated to determine if the source of failure is analytical error. If so, reprepare and analyze the MD if sufficient sample material is available. Otherwise, qualify specific analytes in all samples in the associated preparatory batch.

Appendix A

Appendix A has been removed from the document.

Appendix B

Quality Control Requirements

Table B-1. Organics Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Breakdown check (Endrin/DDT Organochlorine Pesticide Methods only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin shall each be ≤ 15%. No samples shall be run until degradation of DDT and Endrin are each ≤ 15%.	Correct problem, then repeat breakdown checks. Qualification of data is not appropriate.
Other Performance Checks If the reference method includes additional performance checks such as peak tailing evaluation, the checks shall be performed and evaluated as described in the reference method.	As recommended or required in the reference method.	As recommended or required in the reference method.	Correct problem, then repeat performance checks. Qualification of data is not appropriate.

Table B-1. Organics Analysis by Gas Chromatography (GC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 20%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors shall be performed using a 5-point (minimum) calibration. Results shall not be quantitated using a single point.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 50%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) or Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical batch. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL when ICAL is performed or on days when ICAL is not performed, the initial CCV for the batch shall be used.	NA.

Table B-1. Organics Analysis by Gas Chromatography (GC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater. For analytes reported across a RT range (e.g., TPH variations, chlordane), the RT window of the method-defined marker compounds are established as stated above. The RT range is calculated based on the lower limit of the RT window for the first marker compound and the upper limit of the RT window for the last marker compound.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA

Table B-1. Organics Analysis by Gas Chromatography (GC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	At the beginning of each analytical batch before sample analysis when ICAL is not performed, after every 10 field samples, and at the end of the analytical batch. CCVs for multi-component analytes (e.g., Toxaphene, Chlordane and Aroclors other than 1016 and 1260) are only required before and after analysis of samples in which these compounds are present. Per the reference method, when these analytes are detected the sample(s) are reanalyzed with the appropriate CCVs included.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value, unless the specific reference method has tighter criteria, in which case the reference method shall be followed.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If Used**	If used, every field sample, standard, and QC sample.	RT within ± 30 seconds from RT of the midpoint standard in the ICAL; Measured area within – 50% to +50% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used as the RT reference.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-1. Organics Analysis by Gas Chromatography (GC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all analytes to be reported.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-1. Organics Analysis by Gas Chromatography (GC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Surrogate Spike	All QC and samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-1. Organics Analysis by Gas Chromatography (GC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Confirmation of positive results (second column) The laboratory may also use mass spectrometry for confirmation.	All results > the DL must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not a requirement or in cases where confirmation is not technically possible). Samples without detected target analytes do not require second-column analysis.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%. Laboratory shall use project-specific reporting requirements if available. If not available, use the reference method requirements if available; otherwise report the result from the primary column. RPD evaluation between primary and secondary column does not apply if both results are below LOQ.	Apply qualifier to affected results and explain in the case narrative. The laboratory shall identify the primary column for each target analyte. If results are reported from the secondary column due to interference or QC outside acceptance criteria, the laboratory shall apply qualifier to affected analyte results and explain in the case narrative.

Table B-2. Organics Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A and 8321 by UV Detection)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Chromato-graphic Resolution	With each ICAL using a standard at or near the mid-range, CCV, and in each field sample.	The height from the baseline to the valley between two peaks is less than or equal to 50% of the height of the shorter peak.	If the required resolution cannot be achieved at ICAL, the compounds that are not resolved shall be reported as an aggregate of the compounds. If the required resolution can be achieved in the ICAL but cannot be achieved in a CCV, then correct problem. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific analyte(s) in all affected samples and explain in the case narrative. If the required resolution can be achieved in the calibration and the CCV but cannot be achieved in analysis of field samples, apply qualifier to the specific analyte(s) in all affected samples and explain in the case narrative.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: <u>Option 1:</u> %RSD for each analyte ≤ 20%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; <u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3:</u> non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-2. Organics Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A and 8321 by UV Detection)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 15% and 40%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) or Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.
Retention Time (RT) window width **Internal Standard Used**	With each sample.	RRT width is ± 0.06 from the established RRT.	NA.

Table B-2. Organics Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A and 8321 by UV Detection)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICAL is not performed; after every 10 field samples; and at the end of the analytical batch run.	All reported analytes and surrogates within their respective retention time window and within ± 20% of true value, unless the reference method recommends tighter requirements, in which case the reference method criteria shall be followed. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If Used**	Every field sample, standard, and QC sample.	RT within ± 10 seconds (0.17 minutes) from RT of the midpoint standard in the ICAL; EICP area within 30% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect HPLC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-2. Organics Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A and 8321 by UV Detection)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-2. Organics Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A and 8321 by UV Detection)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-2. Organics Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A and 8321 by UV Detection)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.
Confirmation of positive results (second column) The laboratory may also use mass spectrometry for confirmation.	All field sample results > the DL shall be confirmed (except for single-column methods where confirmation is not a requirement or in cases where confirmation is not technically possible). Spectral match confirmation of a UV detector with a UV diode array detector (or vice versa) is not considered an acceptable confirmation technique. Samples without detected target analytes do not require second-column analysis.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%. Laboratory shall use customer-provided reporting requirements if available. If not available, use the reference method requirements if available; otherwise report the result from the primary column. RPD evaluation between primary and secondary column does not apply if both results are below LOQ.	Apply qualifier to affected results and explain in the case narrative. The laboratory shall identify the primary column for each target analyte. If results are reported from the secondary column due to interference or QC outside acceptance criteria, the laboratory shall apply qualifier to affected analyte results and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Sample Preparation and Processing	Each field sample, PT, LCS, and blank when ISM is required.	Before drying and grinding, sample shall be sieved to 2 mm. Following 2 mm sieve, entire sample shall be dried and ground following the laboratory's grinding procedure for ISM.	NA.
Grinding Procedure for ISM	Initial method validation and any time major equipment is changed or when a reduction in the number or time of grinding cycles occur.	The laboratory shall demonstrate that grinding procedure is capable of reducing the particle size to <75 microns by passing representative portions of ground demonstration samples through a 200-mesh sieve.	NA.
Grinding for ISM	Each field sample, PT, LCS, and blank when ISM is required.	Grinding periods shall not exceed 60 seconds and shall be followed by a 2-minute or longer cool down period between the grind cycles.	NA.
Grinding Blank for ISM For batch preparation, the Grinding Blank may serve as the Method Blank.	One per batch using Ottawa sand or a verified clean soil. Grinding blank shall be processed immediately after a customer-identified sample with suspected high target analyte concentration or after the LCS.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated sample whichever is greater.	The laboratory may reprepare and analyze the ground grinding blank to confirm results outside of acceptance criteria. If the grinding blank is not reprepared and analyzed, or if contamination is confirmed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
ISM Laboratory Replicates	At the analytical subsampling step, ISM laboratory replicates shall be performed on one ISM sample in each preparation batch. ISM replicates shall not be performed on any sample identified as a QC or blank (e.g., LCS, Grinding Blank or other QC). The laboratory shall prepare and analyze three or more replicates, each consisting of a minimum of 30 increments of the same mass and depth.	RSD shall not exceed 20%. RSD does not apply if all results are below the LOQ.	The laboratory may reprepare and analyze laboratory replicates to confirm the RSD result outside of acceptance criteria. If the laboratory replicates are not reprepared and analyzed, or if the RSD outside of acceptance criteria is confirmed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Initial calibration (ICAL) for all analytes	At instrument set-up and when needed based on reference method requirements or QC results, before sample analysis.	Minimum 5 levels. Each analyte shall meet one of the two options below: Option 1: %RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 40%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed or on days when ICAL is not performed, the initial CCV for the sequence is used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within their respective retention time window and within ± 20% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If used**	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS) If a laboratory utilizes a self-spiked solid LCS, the fortification of ISM samples shall be performed before any preparation steps performed, such as drying, grinding, and sieving. For ISM, a solid reference material containing all reported analytes shall be prepared (e.g., ground and subsampled) and analyzed in exactly the same manner as a field sample.	One per preparatory batch. Shall contain all surrogates and all analytes to be reported. A Standard Reference Material (SRM) that is used for a solid LCS can be ground as a single batch and subsampled repeatedly as long as the SRM is within expiration date.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria (beginning at the subsampling step for ISM samples) if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 20% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike Surrogates shall be added to each field sample, blank, and LCS before any processing (e.g., before drying, grinding or extraction)	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.
Confirmation of positive results (second column) The laboratory may also use mass spectrometry for confirmation.	All results > the DL shall be confirmed. Samples without detected target analytes do not require second-column analysis.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%. RPD evaluation between primary and secondary column does not apply if both results are below LOQ.	Apply qualifier to affected results and explain in the case narrative. The laboratory shall identify the primary column for each target analyte. If results are reported from the secondary column due to interference or QC outside acceptance criteria, the laboratory shall apply qualifier to affected analyte results and explain in the case narrative.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Mass Calibration	At instrument set-up; at least annually or as specified by the manufacturer, whichever is more frequent; after major maintenance; and when needed based on method requirements or QC results, before analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As recommended or required by the instrument manufacturer.	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Tune Check	Before ICAL or as stated in the reference method, whichever is most frequent.	Specific ion abundance criteria of BFB or DFTPP from the reference method revision used for analysis and reporting.	Retune instrument and verify. If the method requires an ICAL after a change in tuning parameters, follow the reference method as written (e.g., 8260D, 8270E). Qualification of data is not appropriate.
Performance Check (SVOC, except when analyzing only PAHs or PCBs)	EPA Method 8270E: prior to initial calibration. EPA Methods 8270C, 8270D and any other SVOC GC/MS method: at the beginning of each 12-hour period, before analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol present at their normal responses and tailing factor ≤ 2.	Correct problem, then repeat performance check. Qualification of data is not appropriate.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 20%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$; If the specific version of a reference method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements shall also be met.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 50%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) **Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.**			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater. For analytes reported across a RT range (e.g., TPH variations, chlordane), the RT window of the method-defined marker compounds are established as stated above. The RT range is calculated based on the lower limit of the RT window for the first marker compound and the upper limit of the RT window for the last marker compound.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within their respective retention time window and within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If Used**	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used. Common contaminants (Methylene chloride, Acetone, 2-Butanone, and Phthalates) shall not be detected > LOQ.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) **Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.**			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) **Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.**			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike **If Used**	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified prior to reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-5 has been removed from the document.

Table B-6. Dioxins and Furans Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Resolving Power	Before ICAL and at the beginning and the end of each 12-hour period of analysis.	Static resolving power ≥ 10,000 (10% valley) for identified masses.	Retune instrument and verify. Rerun affected samples. Qualification of data is not appropriate.
Performance Check	Before ICAL or calibration verification. At the beginning of each 12-hr period during which samples or calibration solutions are analyzed	RT window width: Peak separation between 2,3,7,8-TCDD and other TCDD isomers: Resolved with a valley of ≤ 25%. Identification of all first and last eluters of the five homologue RT windows. Records shall include a labeled chromatogram showing compliance with this requirement. Absolute RT for switching from one homologous series to the next ≥ 10 sec. for all components of the mixture.	Correct problem, then repeat performance checks. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument setup and after CCV failure.	Ion abundance ratios in accordance with the method. S/N ratio ≥ 10 for all reported analyte ions. Minimum 5 levels when using evaluation by %RSD or linear regression. Each analyte shall meet one of the two options below: Option 1: %RSD for each analyte ≤ 20% and %RSD ≤ 20% for the RFs for labeled IS. Option 2 (allowed for EPA 1613 only): linear least squares regression for each analyte: r2 ≥ 0.99 and %RSD ≤ 20% for the RFs for labeled IS. Linear regression calibration shall not be forced through origin.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-6. Dioxins and Furans Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 50%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and isotope-labeled analog.	Position shall be set using the GC performance check solution or GC RT Window Defining Solution, as specified by reference method. The absolute RT criteria per reference method shall be met.	The qualification of data is not appropriate.

Table B-6. Dioxins and Furans Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV) or Calibration Verification (VER)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	The m/z abundance ratios for all analytes shall be within the reference method limits. S/N ≥ 10 for all reported analyte ions. EPA 8290 (CCV): Initial and 12-hr: For analytes, SRF within ± 20% D of RF established in ICAL; and for isotope-labeled analog standards: RF within ± 30%D of the mean of RF established in ICAL. Closing CCV: For analytes, RF within ± 25% D of RF established in ICAL; and for isotope-labeled analog standards, RF within ± 35%D of the mean of RF established in ICAL EPA 1613 (VER): Initial and 12-hr CCV: all compounds shall meet the reference method acceptance criteria. Closing CCV: All compounds shall meet the reference method acceptance criteria ± 5%.	Where an assignable cause isolated to only the CCV or VER is identified, one CCV or VER may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV or VER is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or VER, or recalibrate. All affected samples since last passing CCV or VER shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Extracted Internal Standard (EIS)	Every field sample, standard, and QC sample.	Retention time within ± 15 seconds from retention time of the midpoint standard in the ICAL. EPA 8290: Calculated recovery of each carbon-labeled internal standard within 40% - 135% of ICAL average. EPA 1613: The recovery of each stable isotopically-labeled analog shall be within the method limits. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. EPA 1613: water samples shall be diluted and smaller amounts of soils, sludges, sediments, and other matrices shall be reprepared and analyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the EIS outside acceptance criteria and explain in the case narrative.

Table B-6. Dioxins and Furans Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS) or Ongoing Precision and Recovery Sample (OPR)	One per preparatory batch. Shall contain all analytes to be reported.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria. EPA 1613: OPR method limits shall be met before sample analysis can proceed.	Where an assignable cause isolated to only the LCS or OPR is identified, the LCS or OPR may be reanalyzed. Otherwise, reprepare and analyze the LCS or OPR and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. If insufficient material, notify the customer before preparation. Shall be analyzed for all analytes reported in the parent sample.	RPD of all analytes ≤ 30% between sample and MD. RPD between sample and MD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-6. Dioxins and Furans Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Sample Estimated Maximum Possible Concentration (EMPC)	EPA 8290 only: Every field sample.	As recommended or required in the reference method.	NA.
Sample 2,3,7,8-TCDD TEQ concentration	EPA 8290 only: Sample 2,3,7,8-TCDD TEQ concentration.	As recommended or required in the reference method.	NA.

Table B-7. Mercury (Hg) Analysis by Cold Vapor Atomic Absorption (CVAA)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels and a calibration blank. Linear least squares regression: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 10% and 20%, respectively. The maximum allowable %RSE shall be 20%. Acceptable %RE at the low level also meets the requirement for the LLCCV.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Low-level Calibration Check Verification (LLCCV)	Daily before the analysis of any samples. The LLCCV shall be less than or equal to the LOQ.	All reported Hg values within ± 20% of the true value.	Correct problem and repeat ICAL. Qualification of data is not appropriate.

Table B-7. Mercury (Hg) Analysis by Cold Vapor Atomic Absorption (CVAA)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 10 field samples; and at the end of the analytical batch run.	Hg result within ± 10% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Continuing Calibration Blank (CCB)	Immediately after the ICAL and immediately after every CCV.	No Hg detected > ½ LOQ or > 1/10th the amount measured in the associated sample(s) whichever is greater. No Hg results < -½ LOQ.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-7. Mercury (Hg) Analysis by Cold Vapor Atomic Absorption (CVAA)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No Hg detected > ½ LOQ or > 1/10th the amount measured in the associated sample(s) whichever is greater. No Hg results < -½ LOQ.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-7. Mercury (Hg) Analysis by Cold Vapor Atomic Absorption (CVAA)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 20% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Linear Range Check	If results are reported above the calibration range, the recommendations or requirements for linear range determination and checks listed in the reference method shall be followed.	Within ± 10% of true value.	Data shall not be reported above the calibration range without a passing Linear Range check. Dilute samples until they are within the calibration range or verify the Linear Range according to the referenced method. If the samples cannot be reanalyzed, apply qualifier to analyte results exceeding the calibration range and explain in the case narrative.
Other Performance Checks	As recommended or required in the reference method.	If the reference method includes additional performance checks such as ICV, the checks shall be performed and evaluated as described in the reference method.	Correct problem, then repeat performance checks. Qualification of data is not appropriate.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum: one high standard and a Calibration Blank. If a multi-level calibration is used: Minimum of 5 standards and a calibration blank when evaluating with a linear regression, 6 standards and a calibration blank when evaluating with a quadratic regression. Single Point Calibration: The curve shall be verified with a low level (at or below the LOQ) and a mid-level calibration verification standard. Low level calibration verification: Within 80-120% of the true value. This also meets the requirement for the LLCCV. Mid-level calibration verification: Within 90-110% of the true value. Multi-level calibration: Option 1: RSE for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 10% and 20%, respectively. The maximum allowable %RSE shall be 20%. Acceptable %RE at the low level also meets the requirement for the LLCCV.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	After ICAL, before sample analysis, every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Lower Limit of Quantitation (LLOQ) Verification	Daily. The LLOQ verification shall be less than or equal to the LOQ.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL. Qualification of data is not appropriate.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Blank (CCB)	Immediately after the ICAL and immediately after every CCV.	The absolute values of all analytes < ½ LOQ or < 1/10th the amount measured in any sample, whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Interference Check Solutions (ICS) or Multi-Element Spectral Interference Check (SIC)	After ICAL and before sample analysis. The laboratory shall perform an ICS or a multi-element SIC at the concentrations and frequency as recommended or required in the reference method.	ICS-A or SIC: Absolute value of concentration for all non-spiked project analytes < 1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ± 20% of true value. ICS-AB is not required if the instrument is able to read negative responses.	Correct problem, repeat ICS or SIC. Qualification of data is not appropriate.
Additional Spectral Interference Check (SIC)	Any additional SICs listed in the reference method shall be followed.	As recommended or required in the reference method.	Correct problem, repeat SIC. Qualification of data is not appropriate.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all reported analytes. MD: Shall be analyzed for all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Dilution Test	One per preparatory batch if MS or MSD fails and if target analyte concentration of the parent sample is within the linear range and > 25x the LOQ before dilution or as in referenced method.	Five-fold dilution shall agree within the percentage recommended or required in the reference method.	None, unless required by the project. Apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Post-Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails and if target analyte concentration of the parent sample is not sufficiently high to perform the dilution test.	As recommended or required in the reference method.	None, unless required by the project. Apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Linear Range Check	If results are reported above the calibration range, the recommendations or requirements for linear range determination and checks listed in the reference method shall be followed.	Within ± 10% of true value.	Data shall not be reported above the calibration range without a passing Linear Range check. Dilute samples until they are within the calibration range or verify the Linear Range according to the referenced method. If the samples cannot be reanalyzed, apply qualifier to analyte results exceeding the calibration range and explain in the case narrative.
Tune Check	Before ICAL.	Mass calibration < 0.1 Da from the true value; Resolution < 0.9 Da full width at 10% peak height. Follow instrument manufacturer's recommendations for additional tuning criteria.	Retune instrument and verify. Qualification of data is not appropriate.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum: one high standard and a Calibration Blank. If a multi-level calibration is used: Minimum of 5 standards and a calibration blank when evaluating with a linear regression, 6 standards and a calibration blank when evaluating with a quadratic regression. Single Point Calibration: The curve shall be verified with a low level (at or below the LOQ) and a mid-level calibration verification standard. Low level calibration verification: Within 80-120% of the true value. This also meets the requirement for the LLCCV. Mid-level calibration verification: Within 90-110% of the true value. Multi-level calibration: <u>Option 1:</u> RSE for each analyte ≤ 20%; <u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3:</u> non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at the mid-range and low level of the calibration shall be 10% and 20%, respectively. The maximum allowable %RSE shall be 20%. Acceptable %RE at the low level also meets the requirement for the LLCCV.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	After ICAL, before sample analysis, every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Lower Limit of Quantitation (LLOQ) Verification	Daily. The LLOQ verification shall be less than or equal to the LOQ.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL. Qualification of data is not appropriate.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Internal Standard (IS)	Every field sample, standard and QC sample.	IS intensity in the samples is evaluated as recommended or required in the reference method.	Perform a 5X dilution, reanalyze sample. Repeat dilutions until IS intensities are acceptable. Qualification of data is not appropriate.
Continuing Calibration Blank (CCB)	Immediately after the ICAL and immediately after every CCV.	The absolute values of all analytes < ½ LOQ or < 1/10th the amount measured in any sample, whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Interference Check Solutions (ICS) or Multi-Element Spectral Interference Checks (SIC)	Daily, after ICAL and before sample analysis or every 12 hours of continuing sample analysis, whichever is more frequent.	ICS-A or SIC: Absolute value of concentration for all non-spiked project analytes < 2X LOQ (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of true value.	Correct problem, repeat ICS. Correct problem; reanalyze ICS or SIC, reanalyze all samples following the last SIC within acceptance criteria. Qualification of data is not appropriate.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all reported analytes. MD: Shall be analyzed for all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Dilution Test	One per preparatory batch if MS or MSD fails and if target analyte concentration of the parent sample is within the linear range and > 25x the LOQ before dilution or as in referenced method.	Five-fold dilution shall agree within the percentage recommended or required in the reference method.	None, unless required by the project. Apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Post-Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails and if target analyte concentration of the parent sample is not sufficiently high to perform the dilution test.	As recommended or required in the reference method.	None, unless required by the project. Apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-10. Hexavalent Chromium Analysis by Colorimetry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL)	Daily, before sample analysis.	Minimum 5 levels for when using evaluation by linear regression and 6 levels for evaluation by quadratic regression. Each result shall meet one of the two options below: Option 1: linear least squares regression for each result: $r^2 \geq 0.99$; Option 2: non-linear least squares regression (quadratic) for each result: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 10% and 30%, respectively. The maximum allowable %RSE shall be 15%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 10 samples; and at the end of the analytical batch run.	Hexavalent chromium within ± 10% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-10. Hexavalent Chromium Analysis by Colorimetry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No hexavalent chromium detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-10. Hexavalent Chromium Analysis by Colorimetry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for a MS and MSD or MD, one LCSD shall be included in the preparatory batch.	Recovery: Same as the LCS acceptance criteria. Precision: RPD between LCS and LCSD ≤ 20%.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	Solids (or any samples requiring digestion): One soluble and one insoluble spike per matrix type (as described in the reference method) per preparatory batch if sufficient material is provided. Aqueous: One soluble spike per preparatory batch if sufficient material is provided.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to result in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	Solid Matrix: One MD per preparatory batch if sufficient material is provided. Aqueous Matrix: One MSD or MD per every ten field samples if sufficient material is provided.	Recovery: Same as the LCS acceptance criteria. Precision: RPD of all results ≤ 20% between MS and MSD or sample and MD. RPD between sample and MD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to result in the parent sample and explain in the case narrative.

Table B-10. Hexavalent Chromium Analysis by Colorimetry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Post-digestion Matrix Spike	One per matrix type (as described in the reference method) per preparatory batch.	Recovery within 85 – 115%.	Explain failure in case narrative. Dilute sample, reprepare and analyze with and without post-digestion matrix spike. If results are still outside acceptance criteria after dilution and reanalysis, contact the customer and/or apply qualifier to result in the parent sample and explain in the case narrative.

Table B-11. Cyanide Analysis (CN) by Colorimetry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL)	Daily before sample analysis.	Minimum of 6 levels plus a reagent blank. Linear least squares regression for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Distillation Verification	Once after each ICAL, before sample analysis. Evaluate one distilled standard above the mid-range concentration of the calibration curve and one below the mid-range.	Within ± 10% of non-distilled true value.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at the mid-level and low level of the calibration shall be 10% and 25%, respectively. The maximum allowable %RSE shall be 15%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-11. Cyanide Analysis (CN) by Colorimetry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	Cyanide within ± 10% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Continuing Calibration Blank (CCB)	Immediately after the ICAL and immediately after every CCV.	The absolute values of all cyanide results must not be > ½ LOQ or > 1/10th the amount measured in the associated sample(s) whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-11. Cyanide Analysis (CN) by Colorimetry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	The absolute values of all cyanide results must not be > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-11. Cyanide Analysis (CN) by Colorimetry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch.	Recovery: Same as the LCS acceptance criteria. Precision: RPD between LCS and LCSD ≤ 20%.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to result in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided.	Recovery: Same as the LCS acceptance criteria. Precision: RPD of all results ≤ 20% between MS and MSD or sample and MD. RPD between sample and MD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to result in the parent sample and explain in the case narrative.

Table B-12. Common Anions and Hexavalent Chromium Analysis by Ion Chromatography (IC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 15%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 10% and 40%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) establishment	Once per ICAL and at the beginning of the analytical sequence.	RT shall be set using the midpoint standard of the ICAL when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA.

Table B-12. Common Anions and Hexavalent Chromium Analysis by Ion Chromatography (IC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 10 samples; and at the end of the analytical run.	All reported analytes within ± 10% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-12. Common Anions and Hexavalent Chromium Analysis by Ion Chromatography (IC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria. Hexavalent Chromium: Laboratory-developed acceptance criteria no wider than ± 10%.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 10% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-12. Common Anions and Hexavalent Chromium Analysis by Ion Chromatography (IC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all reported analytes. MD: Shall be analyzed for all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 15% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Mass Calibration	At instrument set-up, at least annually or as specified by the manufacturer, whichever is more frequent, after major maintenance, and when needed based on method requirements or QC results, prior to sample analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As per instrument manufacturer	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Mass Accuracy Verification	Following each mass calibration Mass calibration shall be verified with the mid-level perchlorate standard.	Perchlorate ions shall be within ± 0.3 Da of mass 99, 101, and 107 or their respective daughter ion masses (83, 85, and 89), depending on which ions are quantitated.	Correct problem, then repeat verification or mass calibration as needed. Qualification of data is not appropriate.
Conductivity Limit Study	At initial setup and when major changes occur in the method's operating procedures (e.g., addition of cleanup procedures, column changes, mobile phase changes).	Reported perchlorate concentration within ± 20% of true value., and the internal recovery and calibration standard (IRCS) recovery within ± 50% of the mid-level standard.	Decrease anion concentrations in the dissolved salt solution and repeat Conductivity Limit Study. Qualification of data is not appropriate.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Conductivity Limit Check Sample (CLCS)	Daily before sample analysis and after every 20th field sample. The CLCS shall be prepared with the dissolved salt solution at the conductivity limit and perchlorate concentration at the LOQ and undergo the same filtration as aqueous field samples.	Reported perchlorate concentration within ± 20% of true value, and the internal recovery and calibration standard (IRCS) recovery within ± 50% of the CCV.	Correct problem. Reanalyze all samples and QC samples since last passing CLCS. If column degradation is suspected, a new column must be calibrated before the samples can be reanalyzed. If poor recovery from the cleanup filters is suspected, a different lot of filters shall be used to reprepare and analyze all samples since last passing CLCS.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum of 6 calibration levels shall be used. The calibration shall meet one of the options below: Option 1: linear least squares regression: $r^2 \geq 0.99$; Option 2: non-linear least squares regression (quadratic): $r^2 \geq 0.995$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 15% and 50%, respectively. The maximum allowable %RSE shall be 15%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Retention Time (RT)	Every field sample, standard, and QC sample.	Retention times of the native perchlorate peak and the IRCS perchlorate peak shall be within ± 0.2 minutes.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 10th field sample; and at the end of the analytical batch run.	Reported perchlorate concentration within ± 15% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Recovery and Calibration Standard (IRCS) **^{18}O-labeled perchlorate**	Every field sample, standard, and QC sample.	Measured IRCS area within ± 50% of the value from the average of the IRCS area counts of the ICAL if the calibration is performed on the same day as the analysis, or otherwise, the first CCV of the analytical batch. Measured IRCS area in the first CCV of the analytical batch within ± 50% of the value from the average of the IRCS area counts of the ICAL.	Inspect instrument for malfunctions, verify conductivity is below the conductivity limit, and correct problem. All affected samples shall be reanalyzed. If samples cannot be reanalyzed, apply qualifier to results associated with IRCS outside acceptance criteria and explain in the case narrative.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria. If QSM Appendix C Limits are not available, then laboratory-developed acceptance criteria shall be no wider than ± 20%.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 15% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Confirmation of positive results **Isotope Ratio $^{35}Cl/^{37}Cl$**	Every field sample, standard, and QC sample.	Evaluate the relative abundances of the m/z 83/85 or m/z 99/101 ions in the chromatogram depending on which ions are quantitated. Peak area counts ratio within ± 30% of the average peak area count ratio of the mid-range calibration standard, if the calibration is performed on the same day as the analysis, or otherwise, within the average peak area count ratios of all the CCV runs of the analytical batch.	If Isotope Ratio is not within acceptance criteria, the sample shall be reanalyzed. If the sample was not pretreated, the sample shall be reprepared using cleanup procedures and analyzed. If the Isotope Ratio remains outside acceptance criteria after cleanup, use alternative techniques to confirm presence of perchlorate, e.g., a post spike sample or dilution to reduce any interference, and apply qualifier to result and explain in the case narrative. The use of cleanup procedures, post spike samples, and dilutions, and the disposition of results of alternate techniques used to confirm presence of perchlorate shall be discussed in the case narrative.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	DFTPP mass range from 51-443 m/z using acceptance criteria from Method 8271.	Correct problem, then repeat tune check. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 15%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at the mid-level and low level of the calibration shall be 20% and 50%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater. For analytes reported across a RT range (e.g., TPH variations, chlordane), the RT window of the method-defined marker compounds are established as stated above. The RT range is calculated based on the lower limit of the RT window for the first marker compound and the upper limit of the RT window for the last marker compound.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICAL not performed; after every 10 samples or 12 hours of analysis time, whichever is sooner; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 25% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Internal Standard (IS) **If Used**	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Surrogate Spike	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-15 has been removed from the document.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) **(Energy, efficiency and FWHM peak resolution)**	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits, indicating a change in performance since the previous calibration. Shall use traceable calibration source (CS) that matches sample test source (STS) configuration (type, size, and position relative to the detector).	Verify manufacturer's specifications for point source efficiency; At least three isotopes within the energy range of 3 to 6 MeV. Energy vs. channel slope equation < 15 keV per channel. Full Width – Half Maximum (FWHM) < 100 keV for each peak used for calibration. Minimum of 3,000 net counts in each peak. Peak energy positions of all calibration isotopes are within 40 keV of reference peak energies.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV) **(check source or pulser check)**	Weekly source check or pulser check verification before analysis of samples. Pulser check may be used to verify energy calibration when using radiotracers during analysis. Source check may be used to verify energy, FWHM and efficiency.	CCV energy response shall be monitored by one of the following: Option 1: Energy response checks shall have a tolerance limit or control chart set at ± 3% or 3σ. Option 2: Pulser check observed peak centroid falls ≤ 20 keV from reference energy. Check source FWHM ≤ 100 keV and within 40 keV of corresponding calibration peaks in initial energy calibration.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required if the reanalyzed CCV passes. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all affected samples and explain in the case narrative.
Background Subtraction Count (BSC) Measurement	At least monthly, before initial use or after initial calibration. BSC test source shall match STS configuration (type, size, and position relative to the detector).	Count time equal to or longer than associated sample count time. Each region of interest (ROI) activity sufficiently low enough to ensure required detection limits are met. Background shall be monitored by one of the following: Option 1: Use a statistical test to determine a change in the background count rate value. Option 2: Within ± 3σ of mean activity of recent BSCs for total ROI for all isotopes of interest (minimum of 3 BSC values).	Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC. All affected samples since last passing BSC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected samples results and explain in the case narrative.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Contamination Check (ICC)	Performed weekly and after counting high activity samples.	Instrument contamination shall be monitored by one of the following: Option 1: Use a statistical test to determine a change in the background count rate value. Option 2: Within ± 3σ of mean activity of recent BSCs (minimum of 3 BSCs).	Check control chart for trends and recount. If still out of control, determine cause and correct problem. If background activity has changed, re-establish BSC. All affected samples since last passing ICC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected samples results and explain in the case narrative.
Method Blank (MB)	One per preparatory batch. Blank matrices shall be consistent with the associated samples (e.g., radon-free distilled or deionized water, representative solid material, or physically and chemically identical filter media).	Count time shall be equal to or longer than associated sample count time. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$.	If not within the control limit, recount the MB to confirm results, unless all sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. LCS matrices shall be consistent with the associated samples and contain representative nuclides within the energy ranges of those nuclides to be reported.	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Laboratory-developed control limits shall be within 25% of the true value. Option 2: Control limit for $\|Z_{LCS}\| \leq 3$.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch. (MS not required when chemical yield tracers or carriers are employed).	MS shall be monitored by one of the following: Option 1: Within 60-140% recovery if customer or reference method requirements are not specified. Option 2: If activity of the sample > 5X the spiking level, then $\|Z_{MS}\| \leq 3$.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.
Matrix Duplicate (MD)	One per preparatory batch.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; or the RPD is ≤ 25%. Option 2: Control limit for $\|Z_{Dup}\| \leq 3$.	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Tracers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Isotopic yield within 30-110% if customer or reference method requirements are not specified. FWHM < 100 keV and peak energy within ± 50 keV of known peak energy.	The data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all affected samples in the associated preparatory batch and explain in the case narrative.
Carriers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Chemical yield within 30-110% if customer or reference method requirements are not specified.	The data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all affected samples in the associated preparatory batch and explain in the case narrative.

Table B-17. Gamma Isotopes by Gamma Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) **(Energy, efficiency and FWHM peak resolution)**	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits, indicating a change in performance since the previous calibration. Shall use a traceable calibration source (CS) that matches sample test source (STS) configuration (type, size, geometry and position relative to the detector).	Minimum of 10,000 net counts in each reference peak for at least six calibration peaks that bracket the range of use. For standard broad spectrum detectors, follow one of the two options: Option 1: Peak energy difference is within 0.1 keV of reference energy for all points. Peak Full Width at Half Maximum (FWHM) < 2.5 keV at 1332 keV. Energy vs. channel slope equation shall be linear and accurate to 0.5 keV. Option 2: Verify manufacturer's specifications for gamma peak resolution. Efficiency vs. energy for each geometry/matrix. 95% confidence limit of the fitted function: ≤ 8% over energy range. Low energy or thin-window gamma systems shall be calibrated to an energy range in accordance with manufacturer's specifications or data sheets.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-17. Gamma Isotopes by Gamma Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily or before use. When working with long count times or batch sequences that run more than a day, the CCV shall be performed at the beginning and end of each analytical batch. The maximum elapsed time between CCVs shall not exceed seven days.	Response checks shall meet one of the following: Option 1: Responses shall meet acceptance criteria of ± 3% or 3σ of the mean. Option 2: Peak Energy/Efficiency: low, mid, and high energies shall be within 10% of the initial calibration value; and FWHM: Low, mid, and high energies shall be within 10% of initial FWHM value.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required if the reanalyzed CCV passes. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all affected samples and explain in the case narrative.
Background Subtraction Count Measurement (BSC) **(Long count for subtracting background from all counts except the BSC and ICC.)**	Immediately after ICAL and at least monthly.	The counting interval for the long count shall be between one and four times the nominal counting interval of the test sources. Activity shall be low enough to meet customer-provided requirements and shall meet one of the following: Option 1: Statistical test of successive counts and count rates for identified background peaks show no significant difference. Option 2: Within ± 3σ of mean activity of recent BSCs (minimum of 3 BSCs).	Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC. All affected samples since last passing BSC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected samples results and explain in the case narrative.

Table B-17. Gamma Isotopes by Gamma Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Contamination Check (ICC)	Daily, or when working with long count times, before and after each analytical batch. Check after counting high activity samples.	No extraneous peaks identified (i.e., no new peaks in the short background spectrum compared to previous spectra); Integrate spectrum from ~50 – 2,000 keV to check for gross contamination. Activity shall be low enough to meet customer-provided requirements and the result shall be within the tolerance limit or control chart limits of ± 3% or 3σ of the mean activity.	Check control chart for trends and recount. If still out of control, determine cause and correct problem. If background activity has changed, re-establish BSC. All affected samples since last passing ICC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected samples results and explain in the case narrative.
Method Blank (MB)	One per preparatory batch. Blank matrices and geometry shall be consistent with the associated samples.	Count time shall be equal to or longer than associated sample count time. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$.	If not within the control limit, recount the MB to confirm results, unless all sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-17. Gamma Isotopes by Gamma Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. LCS matrices shall be consistent with the associated samples including geometry and contain representative nuclides within the energy ranges of those nuclides to be reported.	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Laboratory-developed control limits shall be within 25% of the true value. Option 2: Control limit for $\lvert Z_{LCS} \rvert \leq 3$.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; or the RPD is ≤ 25%. Option 2: Control limit for $\lvert Z_{Dup} \rvert \leq 3$.	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-18. Alpha and Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) – <u>Voltage Plateau</u> **(Separate plateaus determined for alpha and beta activity)**	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits, indicating a change in performance since the previous calibration. Shall use a traceable calibration source.	Perform a series of counts in ≤ 50V steps from approximately 300-1500V. Determine a usage range where slope of the plateau is < 5% over 100V change.	Correct problem, then repeat ICAL-Voltage Plateau determination. Qualification of data is not appropriate.
Initial Calibration (ICAL) – <u>Efficiency</u>	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits, indicating a change in performance since the previous calibration.	Detector's counting efficiency, using traceable calibration sources, shall be determined for each radionuclide used to analyze test sources. Verify manufacturer's specifications for detector efficiency for both alpha and beta counting modes using known sources. A 1σ counting uncertainty of ≤ 1% shall be achieved for all detector efficiency determinations.	Correct problem, then repeat ICAL-Efficiency determination. Qualification of data is not appropriate.

Table B-18. Alpha and Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) – Cross-Talk Factors	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits, indicating a change in performance since the previous calibration.	Determine cross-talk factors for each nuclide using a traceable calibration source per matrix and method. Verify manufacturer's specifications for cross- talk in alpha and beta channels.	Correct problem, then repeat ICAL-Cross Talk Factors determination. Qualification of data is not appropriate.
Initial Calibration (ICAL) – Self-Absorption Curve	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits, indicating a change in performance since the previous calibration.	Using traceable calibration sources, establish a mass attenuation curve with a minimum of seven mass-attenuated standards. For each radionuclide of interest, establish mathematical function (curve) of detector efficiency vs. source mass loading. 95% confidence limit of the fitted function (curve) over the calibration range to ≤ 10% and ≤ 5% uncertainty for alpha and beta, respectively.	Correct problem, then repeat ICAL-Self Absorption Curve determination. Qualification of data is not appropriate.

Table B-18. Alpha and Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily or before use and after a counting gas change. When working with long sample count times or batch sequences that run more than a day, CCV shall be performed at the beginning and end of each analytical batch if the elapsed time between CCVs is not longer than seven days.	Minimum of 2,000 net counts for each energy type (Alpha, Beta). Response checks shall be within a tolerance limit or control chart limits ≤ 3% or 3σ of the mean.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required if the reanalyzed CCV passes. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all affected samples and explain in the case narrative.
Background Subtraction Count (BSC) Measurement **(Long count for subtracting background from all counts except the BSC and ICC.)**	Determine alpha and beta background after efficiency calibration and performed at least monthly for each detector using a contamination-free source mount.	Count time ≥ the associated sample count time. Background shall be monitored by one of the following: Option 1: Use a statistical test to determine a change in the background count rate value. Option 2: Within ± 3σ of mean activity of recent BSCs for total ROI for all isotopes of interest (minimum of 3 BSC values).	Check control chart for trend and recount. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC. All affected samples since last passing BSC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected samples results and explain in the case narrative.

Table B-18. Alpha and Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Contamination Check (ICC) **(Short count for controlling gross contamination)**	Daily or when working with long count times, before and after each analytical batch and after counting high-activity samples.	Background shall be monitored by one of the following: Option 1: Use a statistical test to determine a change in the background count rate value. Option 2: Within ± 3σ of mean activity of recent BSCs for total ROI for all isotopes of interest (minimum of 3 BSC values).	Check control chart for trends and recount. If still out of control, determine cause and correct problem. If background activity has changed, re-establish BSC. All affected samples since last passing ICC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected samples results and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	Count time shall be ≥ the associated sample count time. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$.	If not within the control limit, recount the MB to confirm results, unless all sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-18. Alpha and Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch.	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Laboratory-developed control limits shall be within 25% of the true value. Option 2: Control limit for $\|Z_{LCS}\| \leq 3$.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch. (MS not required when yield tracers/carriers are employed, or when performing a direct/non-destructive measurement)	MS shall be monitored by one of the following: Option 1: Within 60-140% recovery if customer or method requirements are not specified. Option 2: If activity of the sample > 5X the spiking level, then $\|Z_{MS}\| \leq 3$.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; or the RPD is ≤ 25%. Option 2: Control limit for $\|Z_{Dup}\| \leq 3$.	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-18. Alpha and Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Tracers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Isotopic yield within 30-110% if customer or reference method requirements are not specified.	The data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all affected samples in the associated preparatory batch and explain in the case narrative.
Carriers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Chemical yield within 30-110% if customer or reference method requirements are not specified.	The data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all affected samples in the associated preparatory batch and explain in the case narrative.

Table B-19. Tritium in Water by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Performance Check	Before initial use, following repair or loss of control, and upon incorporation of new or changed instrument settings. Use a traceable calibration source (CS) radionuclide sources, typically unquenched Liquid Scintillation cocktails tagged with 3H or 14C.	Verify manufacturer's specifications for counting efficiency and establish energy ROIs (window settings) for nuclides of interest.	Correct problem, then repeat ICAL for Efficiency and ROI. Qualification of data is not appropriate
Initial Calibration (ICAL) **(Quench curve)**	Before initial use, following repair or loss of control, upon incorporation of new or changed instrument settings, matrix, or cocktail type. Shall use a traceable CS.	Analyze a minimum of five different quench factor sources. Obtain a minimum of 10,000 counts for each data point. The quench curve shall meet one of the following: Option 1: Correlation coefficient for quench curve ≥ 0.995. Option 2: ROI error < 1%, and 95% confidence limit for established curve is < 5% over expected quench range.	Correct problem, then repeat Quench Curve. Qualification of data is not appropriate

Table B-19. Tritium in Water by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis for short counting intervals. When working with long count times or batch sequences that run more than a day, the CCV shall be performed at the beginning and end of each analytical batch. The maximum elapsed time between CCVs shall not exceed seven days. ROI for unquenched traceable reference standards (typically 3H or 14C).	Response acceptance criteria shall be ± 3% or 3σ of the mean.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required if the reanalyzed CCV passes. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all affected samples and explain in the case narrative.
Long Background Count (Evaluation) **(Unquenched Blank)**	Before initial use and monthly.	Count time equal to or longer than associated sample count time. Background shall be monitored by one of the following: Option 1: Use a statistical test to determine a change in the unquenched background ROI count rate value. Option 2: Within ± 3σ of mean activity of recent BSCs for each ROI to be determined (minimum of 3 BSCs).	Check control chart for trend and recount. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC. All affected samples since last passing BSC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected samples results and explain in the case narrative.

Table B-19. Tritium in Water by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Background Evaluation **(Quenched Blank)**	One per preparatory batch. The Method Background test source shall match the associated sample configuration (type, size and position relative to the detector).	Count time shall be equal to or longer than associated sample count time. Background shall be monitored by one of the following: Option 1: Use a statistical test to determine a change in the quenched background ROI count rate value. Option 2: Within ± 3σ of mean activity of recent BSCs for each ROI to be determined (minimum of 3 BSCs).	Check control chart for trends and recount. Determine cause, correct problem. If background activity has changed, re-establish Method Background. All affected samples since last passing Method Background shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclide) where count rate < 10X that in the affected ROI in all affected samples and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	MB shall be counted for at least the same amount of time as samples. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$.	If not within the control limit, recount the MB to confirm results, unless all sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-19. Tritium in Water by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch.	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Laboratory-developed control limits shall be within 25% of the true value. Option 2: Control limit for $\|Z_{LCS}\| \leq 3$.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch. (MS not required when yield tracers or carriers are employed)	MS shall be monitored by one of the following: Option 1: Within 60-140% recovery if customer or reference method requirements are not specified. Option 2: If activity of the sample > 5X the spiking level, then $\|Z_{MS}\| \leq 3$.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; or the RPD is ≤ 25%. Option 2: Control limit for $\|Z_{Dup}\| \leq 3$.	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-19. Tritium in Water by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Tracers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Within 30-110% if acceptance criteria are not specified by the reference method or customer.	The data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all affected samples in the associated preparatory batch and explain in the case narrative.
Carriers (if used)	Every field sample and any associated batch QC samples as chemical yield monitor.	Chemical yield within 30-110% if customer or reference method requirements are not specified.	The data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all affected samples in the associated preparatory batch and explain in the case narrative.

Table B-20 Radium-226 (Radon) by Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) **Instrument Operational Voltage and Cell/Detector Efficiency**	Before initial use, following repair or loss of control and upon incorporation of new or changed instrument settings. Establish the operating voltage plateau for each detector. Use traceable Ra-226 calibration source (CS) that matches sample test source (STS) configuration (type, size and position relative to the detector).	Verify manufacturer's specifications. Confirm each selected operational voltage is within the slope range of < 2%/100V.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	Detector response check before use using an appropriate STS. Each Cell/Detector pair efficiency shall be checked at least annually.	The results of this detector response check shall fall within established laboratory-developed control limits. Continuing efficiency for each Cell/Detector pair shall be within less than ± 25% of the initial average.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required if the reanalyzed CCV passes. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all affected samples and explain in the case narrative.

Table B-20 Radium-226 (Radon) by Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Background Subtraction Count (BSC) Measurement, Cell Contamination Check	Before use for each Cell/Detector pair.	Results shall be within ±3σ of mean activity of recent BSCs (minimum of 3 BSC values).	Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC. All affected samples since last passing BSC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected samples results and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	Count time shall be equal to or longer than associated sample count time. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$.	If not within the control limit, recount the MB to confirm results, unless all sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-20 Radium-226 (Radon) by Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch.	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Laboratory-developed control limits shall be within 25% of the true value. Option 2: Control limit for $\|Z_{LCS}\| \leq 3$.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch. (MS not required when a chemical carrier is employed)	MS shall be monitored by one of the following: Option 1: Within 60-140% recovery if customer or method requirements are not specified. Option 2: If activity of the sample > 5X the spiking level, then $\|Z_{MS}\| \leq 3$.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; or the RPD is ≤ 25%. Option 2: Control limit for $\|Z_{Dup}\| \leq 3$.	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-20 Radium-226 (Radon) by Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Carriers (if used quantitatively)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Chemical yield within 30-110% if not specified by the customer or the reference method.	The data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all affected samples in the associated preparatory batch and explain in the case narrative.

Table B-21. Organics in Air Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Cannister Maintenance and Cleanliness Check	As recommended or required in the reference method.	No reported analytes detected > ½ LOQ.	Correct problem, then repeat cleaning of canister and recertify. Qualification of data is not appropriate.
Tune Check	Before ICAL and before each 24-hour period of sample analysis.	Specific ion abundance criteria of BFB from the reference method.	Correct problem, then repeat tune check. Qualification of data is not appropriate.
Calibration Blank	As recommended or required in the reference method.	As recommended or required in the reference method.	Correct problem, then repeat calibration blank. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, prior to sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 8 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 30%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.995$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.995$; Note: The requirement $r^2 \geq 0.995$ is intentional and consistent with method TO-15A.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-21. Organics in Air Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall determine the %RE at every point in the calibration. %RE shall be less ≤ 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and internal standard.	RT shall be set using the average retention times of the ICAL.	NA.
Retention Time (RT) Window Width	Every field sample, standard, and QC sample. Calculated for each analyte.	RT within ± 2 seconds from the established RT position.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	At the beginning and end of the analytical batch. Analysis of a known standard shall be in the lower third of the calibration curve.	All reported analytes within ± 30% of true value for initial and closing CCV.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-21. Organics in Air Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Internal Standard (IS)	Every field sample, standard, and QC sample.	RT within ± 2 seconds from the established RT position; area response for each IS compound shall be within ± 40% of the average response as determined from the ICAL. On days when ICAL is not performed, the daily initial CCV can be used as the reference for the area response.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-21. Organics in Air Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 25% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Mass Calibration	At instrument set-up; at least annually or as specified by the manufacturer, whichever is more frequent; after major maintenance; and when needed based on method requirements or QC results, before analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As recommended or required by the instrument manufacturer.	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Mass Accuracy Verification (according to manufacturer instructions)	Before each ICAL.	Masses within the mass range to be monitored shall be within ± 0.2 Da of the target mass.	Correct problem, then repeat verification. Qualification of data is not appropriate.
Instrument Sensitivity Check (ISC)	Daily. The ISC standard shall be less than or equal to the LOQ.	S/N ≥ 10:1 for all quantitation ions and S/N ≥ 3:1 for all other characteristic ions. All reported analytes and surrogates within ± 20% of true value	Correct problem, then repeat ISC. If problem persists, repeat ICAL, raising LOQ to a concentration where criteria are met. Qualification of data is not appropriate.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Characteristic Ions	Minimum of 3 structurally significant ions that are not isotopic clusters, when possible. IS and surrogates may use fewer ions.	The relative intensities of the characteristic ions of target analytes agree within 30% of the relative intensities in the reference spectrum and the relative intensities shall be > 0.	Qualification of data is not appropriate.
Chromatography Performance Checks (e.g., DDT Tailing factor in 8270)	As recommended or required by the full-scan reference method.	As recommended or required by the full-scan reference method	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, prior to sample analysis.	Average response factor ≥ 0.01 for each reported analyte. Minimum 5 levels when calibrating by %RSD or linear regression and 6 levels when calibrating by quadratic regression. Each analyte shall meet one of the three options below: <u>Option 1:</u> %RSD for each analyte ≤ 20%, unless the reference method has tighter criteria, in which case the method shall be followed; <u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3:</u> non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 50%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) or Relative Retention Time (RRT) Width	With each sample.	Characteristic ions shall maximize in the same scan or within one scan of each other. Within each SIM descriptor window, characteristic ions for the earliest eluting target analyte shall begin at baseline and latest eluting target analyte shall end at baseline. Option 1: The RT shall be within ±10 seconds of the established RT. Option 2: RRT window width is ± 0.06 from the established RRT. Note: After maintenance is performed which may affect RT, RRTs may be updated based on the daily CCV providing that each peak or peak cluster starts and ends at baseline, within the SIM descriptor window.	Correct problem, then rerun ICAL and any affected samples. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	Response factor ≥ 0.01 for each reported analyte All reported analytes and surrogates within their respective retention time window and within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method recommends or requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS)	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 20% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike	Every field sample and QC sample, spiked at a concentration no greater than the mid-range of the calibration. PAH analysis: Surrogates required for polycyclic aromatic hydrocarbon (PAH) target analytes shall include: fluoranthene-d_{10} and 2-methylnaphthalene-d_{10}. If samples are prepared by ISM the surrogate requirements for PAH analysis are identified in Table B-30.	Same as the LCS acceptance criteria. RF for PAH surrogates ≥ 0.40.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-23 has been removed from the document.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
AFFF samples	Each AFFF sample. Note: This does not include AFFF samples that are to be evaluated for MIL-PRF-14385 compliance. Those AFFF samples shall be evaluated in compliance with DoD AFFF01, not Method 1633. A copy of the latest version of DoD AFFF01 can be found at https://denix.osd.mil/edqw/	AFFF samples shall be subsampled in duplicate for analysis in accordance with DoD AFFF01, Section 11.2.1 through 11.2.9. All AFFF samples shall be prepared and analyzed in duplicate in the same manner as aqueous samples (e.g., solid phase extraction, extracted internal standards, carbon cleanup, etc.).	NA.
Instrument Blank (IB)	At the beginning of the analytical sequence and after the analysis of high concentration samples and standards (e.g., customer samples, highest calibration standard, calibration verification standard).	No analytes detected > ½ LOQ.	If acceptance criteria are not met after the highest calibration standard, calibration shall be performed using a lower concentration for the highest standard until acceptance criteria is met. If field sample analyte concentrations exceed the highest calibration standard and the same analytes in the following field sample or in consecutive following field samples also exceed the IB acceptance criteria (i.e., > 1/2 LOQ), the affected samples shall be reanalyzed using a fresh aliquot of the sample extract. If the extract cannot be reanalyzed and re-extraction is not possible, apply qualifier to affected results and explain in the case narrative.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Extracted Internal Standard (EIS)	Every field sample, QC sample, and standard.	Isotopically labeled analogs of analytes shall be used when they are commercially available. Where Method 1633 does not provide EIS recovery acceptance criteria for the sample matrix under evaluation, a laboratory shall use laboratory-developed recovery acceptance criteria no wider than any acceptance criteria provided by the customer. Preliminary laboratory-developed acceptance criteria of 20-150% shall be used until laboratory acceptance criteria are developed in accordance with Method 1633. Where Method 1633 does not provide EIS recovery acceptance criteria for the sample matrix under evaluation, the lower limit of the laboratory-developed acceptance criteria cannot be < 20%.	Follow the requirements listed in Method 1633, Section 15.3.2 for all sample matrices. If EIS recoveries still fall outside of the acceptance range, the customer shall be contacted for additional measures to be taken. Apply qualifier to affected analyte results and explain in the case narrative.
Method Blank (MB)	One per preparatory batch	No analytes detected > ½ LOQ.	Correct problem. If required, reprepare and analyze MB and all QC samples and affected field samples processed with the contaminated blank if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (LLLCS) Method 1633 equivalent to the LCS is the Ongoing Precision and Recovery Standard (OPR). Method 1633 equivalent to the LLLCS is Low-Level Ongoing Precision and Recovery Standard (LLOPR).	One set per preparatory batch. Shall contain all EIS, NIS, and all analytes to be reported.	Where Method 1633 does not provide LCS and LLLCS recovery acceptance criteria for the sample matrix under evaluation, a laboratory shall use laboratory-developed recovery acceptance criteria no wider than any acceptance criteria provided by the customer. Preliminary acceptance criteria of 40-150% shall be used until acceptance criteria are developed by the laboratory in accordance with Method 1633. Where Method 1633 does not provide LCS and LLLCS recovery acceptance criteria for the sample matrix under evaluation, the laboratory-developed acceptance criteria shall not be < 40%.	Correct problem. If required, reprepare and analyze the LCS and/or LLLCS and all affected QC samples and field samples in the associated preparatory batch for failed analytes if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Duplicate (MD) Method 1633 equivalent to the MD is the Laboratory Duplicate	Each AFFF sample prepared using an aliquot of the field sample shall be prepared in duplicate. For all other matrices, one per preparatory batch.	RPD of all analytes ≤ 30% between sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Mass Calibration	At instrument set-up; at least annually or as specified by the manufacturer, whichever is more frequent; after major maintenance; and when needed based on method requirements or QC results, before analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As recommended or required by the instrument manufacturer.	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 20%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 40%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Instrument Sensitivity Check (ISC)	Before analysis and at least once every 12 hours. The ISC standard shall be less than or equal to the LOQ.	All reported analytes and surrogates within ± 40% of true value	Correct problem, then repeat ISC. If problem persists, repeat ICAL, raising LOQ to a concentration where criteria are met. Qualification of data is not appropriate.
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater. For analytes reported across a RT range (e.g., TPH variations, chlordane), the RT window of the method-defined marker compounds are established as stated above. The RT range is calculated based on the lower limit of the RT window for the first marker compound and the upper limit of the RT window for the last marker compound.	NA.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICAL not performed; after every 10 samples or 12 hours of analysis time, whichever is sooner; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 25% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS)	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and LC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike	All field and QC samples.	The laboratory shall use the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-26. IH Methods for Organics Analysis by Gas Chromatography (GC) or High-Performance Liquid Chromatography (HPLC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Canister or Thermal Desorption Unit (TDU) tube Cleanliness Certification	Batch certification for canisters or TDU tubes is permitted before shipment unless the customer requires individual certification. Individual canisters or TDU tubes shall be certified for SIM analysis.	As recommended or required in the reference method. If not specified in the reference method, no reported analytes detected ≥ LOQ.	Correct problem, then repeat performance checks. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 30%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors shall be performed using a 5-point (minimum) calibration. Results may not be quantitated using a single point.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention time (RT) window position establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte.	RT shall be set using the midpoint standard of the ICAL curve when ICAL is performed or on days when ICAL is not performed, the initial CCV for the sequence is used.	NA.

Table B-26. IH Methods for Organics Analysis by Gas Chromatography (GC) or High-Performance Liquid Chromatography (HPLC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention time (RT) window width	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte.	The RT of the analyte shall be within ± 2 seconds of the established window position.	NA.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second-source standard before sample analysis	All reported analytes within the established RT windows. Recovery as recommended or required in the reference method. If not specified in the reference method: GC: all reported analytes within ± 20% of true value. HPLC: all reported analytes within ± 15% of true value.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.
Low-Level Continuing Calibration Verification (LLCCV) A standard containing all target analytes at or below the LOQ	Daily. LLCCVs for multi-component analytes (i.e., Toxaphene, Chlordane and Aroclors other than 1016 and 1260) are only required before and after analysis of samples in which these compounds are present.	GC: All reported analytes within ± 30% of true value. HPLC: All reported analytes within ± 25% of true value.	Correct problem and repeat ICAL. Qualification of data is not appropriate.

Table B-26. IH Methods for Organics Analysis by Gas Chromatography (GC) or High-Performance Liquid Chromatography (HPLC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICV is not performed; after every 10 field samples; and at the end of the analytical batch run. CCVs for multi-component analytes (i.e., Toxaphene, Chlordane and Aroclors other than 1016 and 1260) are only required before and after analysis of samples in which these compounds are present.	All reported analytes within their respective RT windows. GC: All reported analytes within ± 20% of true value for initial and closing CCV. HPLC: All reported analytes within ± 15% of true value for initial and closing CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Continuing Calibration Blank (CCB)	As recommended or required in the reference method.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated sample(s) whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-26. IH Methods for Organics Analysis by Gas Chromatography (GC) or High-Performance Liquid Chromatography (HPLC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	As recommended or required in the reference method. If not specified in the reference method, one per preparatory batch.	If subtraction of background contamination is not specified in the reference method, then no analytes detected ≥ LOQ or > 1/10th the amount measured in any sample(s) whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all analytes to be reported.	A laboratory shall use laboratory-developed acceptance criteria if project specific or reference method limits are not specified.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-26. IH Methods for Organics Analysis by Gas Chromatography (GC) or High-Performance Liquid Chromatography (HPLC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	One per preparatory batch. Shall contain all analytes to be reported.	Recovery: Same as LCS acceptance criteria. LCSD: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Confirmation of positive results (second column)	When required by the reference method or customer, or when recommended by the lab.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%. Laboratory shall use project-specific reporting requirements if available. If not available, use method requirements if available; otherwise report the result from the primary column. RPD evaluation between primary and secondary column does not apply if both results are below LOQ.	Apply qualifier to affected results and explain in the case narrative. The laboratory shall identify the primary column for each target analyte. If results are reported from the secondary column due to interference or QC outside acceptance criteria, the laboratory shall apply qualifier to affected analyte results and explain in the case narrative.
Field Blank (FB)	As recommended or required in the reference method. If not specified, two per preparatory batch, if provided.	Report concentrations on analytical report. Explain in the case narrative on report if results ≥ LOQ.	NA

Table B-27. IH Methods for Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) or Liquid Chromatography/Mass Spectrometry (LC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Canister or Thermal Desorption Unit (TDU) tube Cleanliness Certification	Batch certification for canisters or TDU tubes is permitted before shipment unless the customer requires individual certification. Individual canisters or TDU tubes shall be certified for SIM analysis.	As recommended or required in the reference method. If not specified in the reference method, no reported analytes detected ≥ LOQ.	Correct problem, then repeat performance checks. Qualification of data is not appropriate.
Tune Check	Before ICAL and before each 24-hour period of sample analysis.	As recommended or required in the reference method.	Retune instrument and verify. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 30%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) and Relative Retention Time (RRT) establishment	As recommended or required in the reference method.	As recommended or required in the reference method.	NA.

Table B-27. IH Methods for Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) or Liquid Chromatography/Mass Spectrometry (LC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention time (RT) window width **No Internal Standards Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte.	The RT of the analyte shall be within ± 2 seconds of the established window position.	NA.
Relative Retention time (RT) window width **Internal Standards Used**	With each sample or per reference method requirements.	RRT window width is ± 0.06 RRT units from the established RRT.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second-source standard before sample analysis	All reported analytes within established RT windows. Recovery as recommended or required in the reference method. If not specified in the reference method: Each target analyte shall be recovered within ± 30% of the true value.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.

Table B-27. IH Methods for Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) or Liquid Chromatography/Mass Spectrometry (LC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Low-Level Continuing Calibration Verification (LLCCV) A standard containing all target analytes at or below the LOQ	Daily.	All reported analytes within ± 40% of true value.	Correct problem and repeat ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICV is not performed; after every 24 hours of analysis; and at the end of the analytical batch when IS are not used.	All reported analytes within established RT windows. All reported analytes within ± 30% of true value. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-27. IH Methods for Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) or Liquid Chromatography/Mass Spectrometry (LC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Blank (CCB)	As recommended or required in the reference method.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated sample(s) whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If Used**	Every field sample, standard, and QC sample.	RT within ± 20 seconds from the RT of the mean RT of the ICAL. The area response shall be within ± 40% of the mean area response of the ICAL's. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-27. IH Methods for Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) or Liquid Chromatography/Mass Spectrometry (LC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	As recommended or required in the reference method. If not specified in the reference method, one per preparatory batch.	If subtraction of background contamination is not specified in the reference method, then no analytes detected ≥ LOQ or > 1/10th the amount measured in any sample(s) whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all analytes to be reported.	A laboratory shall use laboratory-developed acceptance criteria if project specific or reference method limits are not specified.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-27. IH Methods for Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) or Liquid Chromatography/Mass Spectrometry (LC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	One per preparatory batch. Shall contain all analytes to be reported.	Recovery: Same as LCS acceptance criteria. LCSD: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Field Blank (FB)	As recommended or required in the reference method. If not specified, two per preparatory batch, if provided.	Report concentrations on analytical report. Explain in the case narrative if results ≥ LOQ.	NA

Table B-28. IH Methods for Inorganics Analysis by Atomic Absorption (AA), Cold Vapor Atomic Absorption (CVAA), Inductively Coupled Plasma/Atomic Emission Spectroscopy (ICP/AES), Colorimetry, Ion Chromatography (IC), or Ion-Selective Electrode (ISE)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Linear Range Check (ICP/AES Only)	If results are reported above the calibration range, the requirements for linear range determination and checks listed in the referenced method shall be followed.	Within ± 10% of true value.	Data shall not be reported above the calibration range without a passing Linear Range check. Dilute samples until they are within the calibration range or verify the Linear Range according to the referenced method. If the samples cannot be reanalyzed, apply qualifier to analyte results exceeding the calibration range and explain in the case narrative.

Table B-28. IH Methods for Inorganics Analysis by Atomic Absorption (AA), Cold Vapor Atomic Absorption (CVAA), Inductively Coupled Plasma/Atomic Emission Spectroscopy (ICP/AES), Colorimetry, Ion Chromatography (IC), or Ion-Selective Electrode (ISE)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Multi-level calibration: Minimum of 5 standards and a calibration blank when evaluating with a linear regression, 6 standards and a calibration blank when evaluating with a quadratic regression. Option 1: linear least squares regression for each analyte: r2 ≥ 0.99; Option 2: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99. Single Point Calibration (ICP/AES): The curve shall be verified with a low level (at or below the LOQ) and a mid-level calibration verification standard. Low level calibration verification: Within 80-120% of the true value. This also meets the requirement for the LLCCV. Mid-level calibration verification: Within 90-110% of the true value. Ion Selective Electrode (ISE): Minimum of 3 standards or as per instrument manufacturers recommendation.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-28. IH Methods for Inorganics Analysis by Atomic Absorption (AA), Cold Vapor Atomic Absorption (CVAA), Inductively Coupled Plasma/Atomic Emission Spectroscopy (ICP/AES), Colorimetry, Ion Chromatography (IC), or Ion-Selective Electrode (ISE)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Retention time (RT) window position establishment (IC Only)	Once per multi-level calibration	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed or on days when ICAL is not performed, the initial CCV for the sequence is used.	NA.
Retention Time (RT) window width establishment (IC Only)	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte. Not applicable if internal standard calibration is used.	As recommended or required in the reference method	NA.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second-source standard before sample analysis	All reported analytes within established RT windows and within ± 10% of true value.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.
Low-Level Continuing Calibration Verification (LLCCV) A standard containing all target analytes at or below the LOQ	Daily. Analyze a standard prepared at or below the analyte's LOQ concentration.	AA, CVAA, and ICP/AES: All reported analytes within ± 30% of the true value. Colorimetric, IC, and ISE: as recommended or required in the reference method, if specified otherwise, laboratory-developed acceptance criteria.	Correct problem and repeat ICAL. Qualification of data is not appropriate.

Table B-28. IH Methods for Inorganics Analysis by Atomic Absorption (AA), Cold Vapor Atomic Absorption (CVAA), Inductively Coupled Plasma/Atomic Emission Spectroscopy (ICP/AES), Colorimetry, Ion Chromatography (IC), or Ion-Selective Electrode (ISE)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICV is not performed; after every 10 field samples; and at the end of the analytical batch run. Colorimetric: After every 15 field samples and at the end of the analysis sequence.	All reported analytes within established RT windows and ± 10% of true value. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Continuing Calibration Blank (CCB) (AA, CVAA, and ICP/AES Only)	Immediately after every ICV and immediately after every CCV.	The absolute values of all analytes shall be < ½ LOQ or < 1/10th the amount measured in any sample, whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-28. IH Methods for Inorganics Analysis by Atomic Absorption (AA), Cold Vapor Atomic Absorption (CVAA), Inductively Coupled Plasma/Atomic Emission Spectroscopy (ICP/AES), Colorimetry, Ion Chromatography (IC), or Ion-Selective Electrode (ISE)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes shall be < ½ LOQ or < 1/10th the amount measured in any sample, whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Interference Check Solution (ICS) or Multi-Element Spectral Interference Check (SIC)	After ICAL and before sample analysis. The laboratory shall perform an ICS or a multi-element SIC at the concentrations and frequency as recommended or required by the referenced method	ICS-A or SIC: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value. ICS-AB is not required if the instrument is able to read negative responses.	Correct problem, repeat ICS or SIC. Qualification of data is not appropriate.

Table B-28. IH Methods for Inorganics Analysis by Atomic Absorption (AA), Cold Vapor Atomic Absorption (CVAA), Inductively Coupled Plasma/Atomic Emission Spectroscopy (ICP/AES), Colorimetry, Ion Chromatography (IC), or Ion-Selective Electrode (ISE)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all analytes to be reported.	A laboratory shall use laboratory-developed acceptance criteria if project specific or reference method limits are not specified.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	One per preparatory batch. Shall contain all analytes to be reported.	Recovery: Same as LCS acceptance criteria. LCSD: RPD of all analytes ≤ 20% between LCS and LCSD. IC LCSD: RPD of all analytes ≤ 15% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Field Blank (FB)	As recommend or required in the reference method. If not specified, two per preparatory batch, if provided.	Report concentrations on analytical report. Explain in the case narrative if results ≥ LOQ.	NA

Table B-29. IH Methods for Inorganics Analysis in Ambient Air by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Linear Range Check	If results are reported above the calibration range, the requirements for linear range determination and checks listed in the referenced method shall be followed.	Within ± 10% of true value.	Data shall not be reported above the calibration range without a passing Linear Range check. Dilute samples until they are within the calibration range or verify the Linear Range according to the referenced method. If the samples cannot be reanalyzed, apply qualifier to analyte results exceeding the calibration range and explain in the case narrative.
Tune Check	Before ICAL and as per the reference method.	As recommended or required in the reference method.	Retune instrument and verify. Qualification of data is not appropriate.

Table B-29. IH Methods for Inorganics Analysis in Ambient Air by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Multi-level calibration: Minimum of 5 standards and a calibration blank when evaluating with a linear regression, 6 standards and a calibration blank when evaluating with a quadratic regression. Option 1: linear least squares regression for each analyte: r2 ≥ 0.99; Option 2: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99. Single Point Calibration: The curve shall be verified with a low level (at or below the LOQ) and a mid-level calibration verification standard. Low level calibration verification: Within 80-120% of the true value. This also meets the requirement for the LLCCV. Mid-level calibration verification: Within 90-110% of the true value.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second-source standard before sample analysis	All reported analytes within ± 10% of true value.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.

Table B-29. IH Methods for Inorganics Analysis in Ambient Air by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Low-Level Continuing Calibration Verification (LLCCV) A standard containing all target analytes at or below the LOQ	Daily. Analyze a standard prepared at or below the analyte's LOQ concentration.	All reported analytes within ± 30% of the true value	Correct problem and repeat ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICV not performed; after every 10 field samples; and at the end of the analytical batch run.	All reported analytes within established ± 10% of the true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-29. IH Methods for Inorganics Analysis in Ambient Air by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Blank (CCB)	Immediately after every ICV and immediately after every CCV.	The absolute values of all analytes shall be < ½ LOQ or < 1/10th the amount measured in any sample, whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS)	Every field sample, standard and QC sample.	The area response shall be within 30-120% of the IS in the ICAL Blank. On days when ICAL is not performed, the daily initial CCV may be used.	Perform a 5X dilution, reanalyze sample. Repeat dilutions until IS intensities are acceptable. Qualification of data is not appropriate.
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes shall be < ½ LOQ or < 1/10th the amount measured in any sample, whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-29. IH Methods for Inorganics Analysis in Ambient Air by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Spectral Interference Check (SIC)	Daily, after ICAL and before sample analysis.	Absolute value of concentration for all non-spiked project analytes <2X LOQ (unless they are a verified trace impurity from one of the spiked analytes).	Correct problem; reanalyze SIC, reanalyze all samples following the last SIC within acceptance criteria. Qualification of data is not appropriate.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all analytes to be reported.	A laboratory shall use laboratory-developed acceptance criteria if project specific or reference method limits are not specified.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	One per preparatory batch. Shall contain all analytes to be reported.	Recovery: Same as LCS acceptance criteria. LCSD: RPD of all analytes ≤ 20% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-29. IH Methods for Inorganics Analysis in Ambient Air by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Field Blank (FB)	As recommended or required in the reference method. If not specified, two per preparatory batch, if provided.	Report concentrations on analytical report. Explain in the case narrative if results ≥ LOQ.	NA

Table B-30. Polycyclic Aromatic Hydrocarbon (PAH) Sample Processing by Incremental Sampling Methodology (ISM)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Grinding	Each field sample.	The entire field sample shall be ground. Drying is typically performed to aid in grinding and is therefore optional for dry soils. The need for grinding is a project decision. Removing a portion of the field sample, not to exceed 10 g, is only allowed to determine the percent moisture for surrogate spikes. The field sample shall be well mixed before removing a portion.	The laboratory shall explain in the case narrative whether grinding was performed.
Grinding Procedure for Incremental Sampling Methodology (ISM)	When sample grinding is performed, initial method validation and any time major equipment is changed or when a reduction in the number or time of grinding cycles occur.	The laboratory shall demonstrate that the grinding procedure reduces the particle size to < 75 microns by passing representative portions of ground demonstration samples through a 200-mesh sieve.	Correct problem. Qualification of data is not appropriate.
Grinding Cycle	When sample grinding is performed using a puck mill, each field sample.	When a puck mill is used to grind the samples, grinding cycles shall not exceed 60 seconds and shall be followed by a 2-minute or longer cool down period between the grind cycles.	Correct problem and reprepare samples. Qualification of data is not appropriate.
Grinding Blank For batch preparation, the Grinding Blank may serve as the Method Blank.	When sample grinding is performed, a grinding blank is required. One per preparatory batch using Ottawa sand or a verified clean soil. A Grinding Blank shall be performed immediately after a customer-identified sample with suspected high target analyte concentration or after the LCS.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	The laboratory may reprepare and analyze the ground grinding blank to confirm results outside of acceptance criteria. If the grinding blank is not reprepared and analyzed, or if contamination is confirmed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-30. Polycyclic Aromatic Hydrocarbon (PAH) Sample Processing by Incremental Sampling Methodology (ISM)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
ISM Laboratory subsampling Replicates The laboratory shall prepare and analyze three or more replicates, each consisting of a minimum of 30 increments of the same mass and depth.	At the analytical subsampling step prior to extraction. When grinding is not performed, each ISM field sample. When grinding is performed, one ISM sample in each preparation batch.	RSD shall not exceed 30%.	RSD failure indicates the sample is not representative. If available, reprepare an additional field sample utilizing grinding procedures. Otherwise, apply qualifier and explain in the case narrative.

Table B-30. Polycyclic Aromatic Hydrocarbon (PAH) Sample Processing by Incremental Sampling Methodology (ISM)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS) A laboratory-prepared solid LCS may be used. The fortification shall be performed prior to any preparation steps. The LCS shall be prepared and analyzed in exactly the same manner as the field samples, including all drying and grinding steps.	One per preparatory batch. Shall contain all surrogates and all analytes to be reported. A Standard Reference Material (SRM) that is used for a LCS can be ground as a single batch and subsampled repeatedly as long as the SRM is within expiration date.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS) Spiking is performed after ISM preparation prior to extraction.	One per preparatory batch if sufficient material is provided.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-30. Polycyclic Aromatic Hydrocarbon (PAH) Sample Processing by Incremental Sampling Methodology (ISM)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike Surrogates shall be added to each field sample, blank, and LCS. A solid surrogate fortification standard shall be added prior to any processing (e.g. prior to drying/grinding or extraction). If drying/grinding is not performed, surrogates may be added after analytical subsampling.	All field samples, blanks and LCS.	The laboratory shall use the following PAH surrogates and respective recovery criteria if the corresponding native analytes are reported unless customer-provided limits are specified. Recovery: Acenaphthylene-d8 within 10% to 120%. Benz[a]anthracene-d12 within 36% to 120%. Benzo[a]pyrene-d12 within 14% to 120% Fluorene-d10 within 29% to 120%	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Ion Transitions (Parent-> Product)	Before method implementation.	The chemical derivation of the ion transitions shall be documented.	NA.
Mass Calibration	At instrument set-up; at least annually or as specified by the manufacturer, whichever is more frequent; after major maintenance; and when needed based on method requirements or QC results, before analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As recommended or required by the instrument manufacturer.	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Mass Accuracy Verification (as recommended or required by the method; otherwise according to manufacturer instructions)	Before each ICAL.	Masses within the mass range to be monitored shall be within 0.2 Da of the target mass.	Correct problem, then repeat verification. Qualification of data is not appropriate.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Sensitivity Check (ISC)	Before analysis and at least once every 12 hours. The ISC standard shall be less than or equal to the LOQ.	S/N ≥10:1 for all quantitation ions and S/N ≥3:1 for all other characteristic ions in the lowest calibration standard used to define the quantitative range. All reported analytes and surrogates within ± 30% of true value.	Correct problem, then repeat ISC. If problem persists, repeat ICAL, raising LOQ to a concentration where criteria are met. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 20%, unless the reference method has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 30%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 10 field samples; and at the end of the analytical batch run.	All reported analytes and surrogates within their respective retention time window and within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Continuing Calibration Blank (CCB)	Immediately after the ICAL and immediately after the CCV.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated sample(s) whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Internal Standard (IS)	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL; EICP area within -30% to +30% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 20% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Surrogate Spike	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Appendix C

Laboratory Control Sample (LCS) Control Limits and Requirements

1.0 Introduction

The DoD Environmental Data Quality Workgroup (EDQW) determined that both DoD and DOE would benefit from updating the existing Laboratory Control Sample (LCS) control limits that were established as a result of a study conducted in 1999 and reported in the 2004 LCS study. The initial study in 2004 was based on a limited data set and did not include all the laboratories and methods that are now a part of DoD ELAP and DOECAP-AP. The objective of the new study was to develop updated LCS limits and provide values for an expanded scope of methods.

The new LCS study, conducted in the summer of 2012, incorporated the contributions from approximately 50 DoD ELAP and DOECAP-AP accredited/approved laboratories. In all, 6.5 million records were analyzed, and LCS limits were set for 23 methods and approximately 1,280 matrix-method-analyte combinations. Based on the laboratory LCS sample data, control limits were calculated for all matrix-method-analyte combinations that met the criteria (a minimum of 100 records) for having sufficient data. Control limits were calculated as the sample mean (percent recovery) $\pm$ 3 sample standard deviations.

2.0 LCS Limit Tables

Table C-1. Method 1668 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2051-60-7	PCB 1	148	91.7	14.6	48	136
56558-16-8	PCB 104	152	99.4	6.2	81	118
32598-14-4	PCB 105	179	105.6	7.2	84	127
74472-37-0	PCB 114	177	105.4	6.2	87	124
31508-00-6	PCB 118	180	107.7	9.6	79	137
65510-44-3	PCB 123	188	107.2	8.8	81	134
57465-28-8	PCB 126	181	100.8	7.3	79	123
2050-68-2	PCB 15	151	106	13.9	64	148
33979-03-2	PCB 155	153	98.7	7.5	76	121
38380-08-4	PCB 156	176	104.5	6.9	84	125
52663-72-6	PCB 167	181	106.8	8.3	82	132
32774-16-6	PCB 169	181	98.8	7.3	77	121

Table C-1. Method 1668 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
74487-85-7	PCB 188	150	97.5	6.4	78	117
39635-31-9	PCB 189	176	102.2	5.7	85	119
38444-73-4	PCB 19	151	99.5	8.6	74	125
2136-99-4	PCB 202	150	97.1	7.1	76	118
74472-53-0	PCB 205	150	100	9.4	72	128
40186-72-9	PCB 206	183	97.5	7.8	74	121
52663-77-1	PCB 208	150	100.2	6.6	80	120
2051-24-3	PCB 209	181	107.6	8.4	83	133
2051-62-9	PCB 3	126	97.4	13.2	58	137
38444-90-5	PCB 37	152	104.3	14.4	61	148
13029-08-8	PCB 4	144	98	13.8	57	140
15968-05-5	PCB 54	150	95.9	9.5	67	124
32598-13-3	PCB 77	152	96.5	7	75	118
70362-50-4	PCB 81	150	100.6	7.7	78	124

Table C-2. Method 1668 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2051-60-7	PCB 1	206	86.7	9.4	58	115
37680-73-2	PCB 101	107	103.8	9.5	75	132
56558-16-8	PCB 104	206	99.4	6.9	79	120
32598-14-4	PCB 105	258	104.7	9.3	77	133
74472-37-0	PCB 114	246	106.5	8.7	81	133
31508-00-6	PCB 118	212	104.9	7.7	82	128
65510-44-3	PCB 123	252	106.8	10.2	76	138
57465-28-8	PCB 126	242	98.4	6.8	78	119
38380-07-3	PCB 128	103	102.3	7.8	79	126
2050-68-2	PCB 15	211	103.5	9.8	74	133
33979-03-2	PCB 155	208	97.4	9.5	69	126
38380-08-4	PCB 156	248	107.6	9.9	78	137
52663-72-6	PCB 167	249	110.4	11	78	143
32774-16-6	PCB 169	247	96.9	8.7	71	123

Table C-2. Method 1668 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
35065-30-6	PCB 170	108	108	10	78	138
74487-85-7	PCB 188	207	95.7	6.5	76	115
39635-31-9	PCB 189	248	102.4	7.2	81	124
38444-73-4	PCB 19	196	98.7	6.5	79	118
2136-99-4	PCB 202	205	95.5	6.2	77	114
74472-53-0	PCB 205	208	95.5	8.8	69	122
40186-72-9	PCB 206	210	93.6	6.6	74	113
52663-77-1	PCB 208	210	98.6	6.4	79	118
2051-24-3	PCB 209	212	103.7	8	80	128
2051-62-9	PCB 3	208	93.6	9.8	64	123
38444-90-5	PCB 37	206	97	12.3	60	134
13029-08-8	PCB 4	207	95	10.9	62	128
15968-05-5	PCB 54	204	95	9.4	67	123
32598-13-3	PCB 77	208	94.1	6.2	75	113
70362-50-4	PCB 81	208	100.6	8	77	125

Table C-3. Method 6010 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	6258	96.7	7.5	74	119
7440-36-0	Antimony	5997	96.4	5.7	79	114
7440-38-2	Arsenic	9530	96.2	4.9	82	111
7440-39-3	Barium	9236	98.3	5	83	113
7440-41-7	Beryllium	6799	97.8	5.1	83	113
7440-42-8	Boron	2312	93	7.1	72	114
7440-43-9	Cadmium	9466	97.5	5.3	82	113
7440-70-2	Calcium	6347	98.1	5.8	81	116
7440-47-3	Chromium	9598	98.9	4.6	85	113
7440-48-4	Cobalt	6725	98.7	4.5	85	112
7440-50-8	Copper	7839	99.1	6	81	117
7439-89-6	Iron	5746	99.7	6.1	81	118
7439-92-1	Lead	10160	96.8	5.1	81	112

Table C-3. Method 6010 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7439-93-2	Lithium	551	98.8	4.5	85	112
7439-95-4	Magnesium	6283	96.1	6.1	78	115
7439-96-5	Manganese	6732	99.1	4.9	84	114
7439-98-7	Molybdenum	4424	98.7	5.7	82	116
7440-02-0	Nickel	7412	98.1	4.9	83	113
7723-14-0	Phosphorus	189	103.1	3.8	92	114
7440-09-7	Potassium	6574	98.3	5.8	81	116
7782-49-2	Selenium	8862	94.5	5.6	78	111
7440-22-4	Silver	9105	97.3	5	82	112
7440-23-5	Sodium	5825	100.1	5.8	83	118
7440-24-6	Strontium	2573	98.5	5	83	114
7440-28-0	Thallium	6416	96.8	4.6	83	111
7440-31-5	Tin	2780	100.1	6.6	80	120
7440-32-6	Titanium	2107	98.2	5.2	83	114
7440-61-1	Uranium	109	97.4	5.2	82	113
7440-62-2	Vanadium	6934	98.3	5.4	82	114
7440-66-6	Zinc	7882	97.4	5	82	113

Table C-4. Method 6010 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	11532	100	4.8	86	115
7440-36-0	Antimony	10737	100.2	4.2	88	113
7440-38-2	Arsenic	14123	99.9	4.3	87	113
7440-39-3	Barium	14476	100.3	4.1	88	113
7440-41-7	Beryllium	11552	100.4	4	89	112
7440-69-9	Bismuth	147	95.8	3.2	86	105
7440-42-8	Boron	3871	98.8	4.8	85	113
7440-43-9	Cadmium	13922	100.8	4.1	88	113
7440-70-2	Calcium	11382	100	4.2	87	113
7440-47-3	Chromium	15027	101.1	3.9	90	113
7440-48-4	Cobalt	11824	101.2	4.2	89	114

Table C-4. Method 6010 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7440-50-8	Copper	12910	100.2	4.6	86	114
7439-89-6	Iron	13797	100.7	4.7	87	115
7439-92-1	Lead	14391	99.3	4.4	86	113
7439-93-2	Lithium	938	100.7	5.3	85	117
7439-95-4	Magnesium	11423	98.8	4.8	85	113
7439-96-5	Manganese	12767	101.9	4.1	90	114
7439-98-7	Molybdenum	8251	101.1	4	89	113
7440-02-0	Nickel	12699	100.5	4.1	88	113
7440-05-3	Palladium	492	99.8	4	88	112
7723-14-0	Phosphorus	203	100.5	4.2	88	113
7440-09-7	Potassium	11006	99.9	4.7	86	114
7782-49-2	Selenium	13264	98.5	5.2	83	114
7440-21-3	Silicon	1525	100.6	6.1	82	119
7440-22-4	Silver	13770	99.1	5.1	84	115
7440-23-5	Sodium	10893	100.9	4.7	87	115
7440-24-6	Strontium	3782	101.3	3.8	90	113
7704-34-9	Sulfur	145	100.7	3.9	89	112
7440-28-0	Thallium	10063	99.5	4.7	85	114
7440-31-5	Tin	4502	101.3	4.4	88	115
7440-32-6	Titanium	5625	101.1	3.4	91	111
7440-61-1	Uranium	223	101.3	5.8	84	119
7440-62-2	Vanadium	12032	100.2	3.6	90	111
7440-66-6	Zinc	13549	100.6	4.6	87	115

Table C-5. Method 6020 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	919	101	7.7	78	124
7440-36-0	Antimony	1911	98.2	8.7	72	124
7440-38-2	Arsenic	3686	99.8	6	82	118
7440-39-3	Barium	2598	100.6	5	86	116
7440-41-7	Beryllium	2457	100.3	6.6	80	120

Table C-5. Method 6020 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7440-42-8	Boron	581	101.1	9	74	128
7440-43-9	Cadmium	2893	99.6	5.4	84	116
7440-70-2	Calcium	835	102.2	5.4	86	118
7440-47-3	Chromium	2420	100.8	6	83	119
7440-48-4	Cobalt	2005	99.7	5.1	84	115
7440-50-8	Copper	2548	101.3	5.8	84	119
7439-89-6	Iron	1131	102.7	7.1	81	124
7439-92-1	Lead	3228	101	5.7	84	118
7439-93-2	Lithium	162	97.8	7.5	75	120
7439-95-4	Magnesium	868	101.6	7.1	80	123
7439-96-5	Manganese	1830	100.3	5.1	85	116
7439-97-6	Mercury	226	99.9	8.8	74	126
7439-98-7	Molybdenum	1188	98.1	5.1	83	114
7440-02-0	Nickel	2617	101.4	5.8	84	119
7440-09-7	Potassium	803	102.3	5.7	85	119
7782-49-2	Selenium	3104	99.2	6.6	80	119
7440-22-4	Silver	2488	100.1	5.9	83	118
7440-23-5	Sodium	818	102.2	7.7	79	125
7440-24-6	Strontium	676	101.7	8.9	75	129
7440-28-0	Thallium	2589	100.1	5.9	83	118
7440-29-1	Thorium	341	98.4	5.7	81	116
7440-31-5	Tin	886	101.3	6.6	82	121
7440-32-6	Titanium	512	100.2	5.7	83	117
7440-61-1	Uranium	833	101.1	6.1	83	120
7440-62-2	Vanadium	1677	99.1	5.7	82	116
7440-66-6	Zinc	2352	100.1	6.2	82	119

Table C-6. Method 6020 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	3145	100.6	5.4	84	117
7440-36-0	Antimony	5172	100.9	5.3	85	117

Table C-6. Method 6020 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7440-38-2	Arsenic	6404	100.1	5.3	84	116
7440-39-3	Barium	4452	99.9	4.8	86	114
7440-41-7	Beryllium	4297	102	6.3	83	121
7440-42-8	Boron	1460	101.5	9.6	73	130
7440-43-9	Cadmium	5699	100.8	4.7	87	115
7440-70-2	Calcium	2085	102.3	5.2	87	118
7440-47-3	Chromium	5569	100.6	5.1	85	116
7440-48-4	Cobalt	3885	100.7	4.7	86	115
7440-50-8	Copper	5092	101.4	5.4	85	118
7439-89-6	Iron	3135	102.4	5.2	87	118
7439-92-1	Lead	6868	101.7	4.5	88	115
7439-93-2	Lithium	461	102.3	8	78	126
7439-95-4	Magnesium	2399	100.4	5.9	83	118
7439-96-5	Manganese	4330	101.1	4.7	87	115
7439-97-6	Mercury	328	97.2	9	70	124
7439-98-7	Molybdenum	2908	99.3	5.4	83	115
7440-02-0	Nickel	5095	100.8	5.3	85	117
7440-09-7	Potassium	2154	101.2	4.7	87	115
7782-49-2	Selenium	5797	100.1	6.7	80	120
7440-22-4	Silver	4956	100.8	5.1	85	116
7440-23-5	Sodium	2313	100.7	5.3	85	117
7440-24-6	Strontium	1170	99.9	5.9	82	118
7440-28-0	Thallium	5352	99.3	5.6	82	116
7440-29-1	Thorium	313	103.7	5.7	87	121
7440-31-5	Tin	1509	100.6	4.8	86	115
7440-32-6	Titanium	1538	98.6	5.3	83	115
7440-33-7	Tungsten	130	103.5	6.2	85	122
7440-61-1	Uranium	1860	103.3	5.4	87	120
7440-62-2	Vanadium	3375	100.5	5	86	115
7440-66-6	Zinc	4253	101	6	83	119

Table C-7. Method 6850 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
14797-73-0	Perchlorate	575	102.5	6.1	84	121

Table C-8. Method 6850 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
14797-73-0	Perchlorate	790	101.6	5.8	84	119

Table C-9. Method 7196 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
18540-29-9	Hexavalent Chromium [Cr (VI)]	2688	96.7	4.3	84	110

Table C-10. Method 7196 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
18540-29-9	Hexavalent Chromium [Cr (VI)]	1576	100.5	3.6	90	111

Table C-11. Method 7470 - 7471 Series Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7439-97-6	Mercury	6471	102	7.5	80	124

Table C-12. Method 7470 - 7471 Series Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7439-97-6	Mercury	10530	100.5	6.3	82	119

Table C-13. Method 8015 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	1263	100.7	11.1	67	134
303-04	Diesel Range Organics (DRO)	2184	85.2	15.7	38	132
307-27	Gasoline Range Organics (GRO)	1134	100.3	7.2	79	122
307-51	Motor Oil	658	72.2	11.2	39	106
84-15-1	o-Terphenyl	314	87.4	14.1	45	130

Table C-14. Method 8015 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	756	101	10.8	69	133
303-04	Diesel Range Organics (DRO)	1757	83.7	16	36	132
307-27	Gasoline Range Organics (GRO)	971	99.9	7.3	78	122
307-51	Motor Oil	573	76.9	12.1	41	113
84-15-1	o-Terphenyl	299	90.5	11.4	56	125
630-02-4	Octacosane	130	101.1	13.8	60	142

Table C-15. Method 8081 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
789-02-6	2,4'-DDT	110	100.1	11.9	64	136
53-19-0	2,4-DDD	111	102.8	9.2	75	130
3424-82-6	2,4-DDE	111	102.2	9.5	74	131
72-54-8	4,4'-DDD	2995	97.7	13.9	56	139
72-55-9	4,4'-DDE	2938	95.3	13	56	134
50-29-3	4,4'-DDT	2470	95.8	15.1	50	141
309-00-2	Aldrin	2985	90.5	15.2	45	136
319-84-6	alpha-BHC	3021	90.9	15.3	45	137
5103-71-9	alpha-Chlordane	2681	93.7	13.2	54	133
319-85-7	beta-BHC	2989	93.1	14.3	50	136
57-74-9	Chlordane	229	95.7	17.7	43	149
319-86-8	delta-BHC	2943	93.3	15.3	47	139
60-57-1	Dieldrin	2987	95.7	13.4	56	136
959-98-8	Endosulfan I	984	92.2	13.2	53	132
33213-65-9	Endosulfan II	2913	93.1	13.5	53	134
1031-07-8	Endosulfan sulfate	2954	95.9	13.5	55	136
72-20-8	Endrin	3076	98.1	13.9	57	140
7421-93-4	Endrin Aldehyde	3004	86	17	35	137
53494-70-5	Endrin Ketone	2953	95.5	13.5	55	136
58-89-9	gamma-BHC [Lindane]	3153	92.1	14.4	49	135
5566-34-7	gamma-Chlordane	2749	94.3	13.7	53	135
76-44-8	Heptachlor	3144	91.6	14.9	47	136
1024-57-3	Heptachlor Epoxide	3093	93.9	13.9	52	136
118-74-1	Hexachlorobenzene	319	91.6	11.4	57	126
72-43-5	Methoxychlor	3021	97.6	15.2	52	143
2385-85-5	Mirex	303	96.4	10.6	65	128
877-09-8	Tetrachloro-m-xylene	1482	85.3	14.6	42	129
8001-35-2	Toxaphene	532	86.7	17.9	33	141

Table C-16. Method 8081 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
72-54-8	4,4'-DDD	3112	99.6	14.4	56	143
72-55-9	4,4'-DDE	3062	96	12.9	57	135
50-29-3	4,4'-DDT	2681	97	15.3	51	143
309-00-2	Aldrin	3021	89.5	14.7	45	134
319-84-6	alpha-BHC	3070	95.8	13.9	54	138
5103-71-9	alpha-Chlordane	2736	94.3	11.6	60	129
319-85-7	beta-BHC	3068	96.3	13.3	56	136
57-74-9	Chlordane	150	101.2	13	62	140
319-86-8	delta-BHC	3035	97.2	15	52	142
60-57-1	Dieldrin	3078	98	12.6	60	136
959-98-8	Endosulfan I	968	93.8	10.7	62	126
33213-65-9	Endosulfan II	3047	93.4	13.7	52	135
1031-07-8	Endosulfan sulfate	3013	97.2	11.9	62	133
72-20-8	Endrin	3635	98.7	13	60	138
7421-93-4	Endrin aldehyde	3018	91.1	13.5	51	132
53494-70-5	Endrin Ketone	2908	95.9	12.6	58	134
58-89-9	gamma-BHC [Lindane]	3693	96.4	12.5	59	134
5566-34-7	gamma-Chlordane	3008	95.8	13.2	56	136
76-44-8	Heptachlor	3597	91.9	12.8	54	130
1024-57-3	Heptachlor Epoxide	3574	96.9	12.1	61	133
118-74-1	Hexachlorobenzene	134	82.1	18.1	27.8	136.5
72-43-5	Methoxychlor	3569	99	15.2	54	145
2385-85-5	Mirex	340	88.8	12.6	51	127
877-09-8	Tetrachloro-m-xylene	1510	84.1	13.3	44	124
8001-35-2	Toxaphene	421	83.9	16.8	33	134

Table C-17. Method 8082 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
12674-11-2	Aroclor 1016	6847	90.1	14.5	47	134
11097-69-1	Aroclor 1254	406	101.2	11.4	67	135
11096-82-5	Aroclor 1260	7975	96.6	14.4	53	140

Table C-17. Method 8082 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
877-09-8	Tetrachloro-m-xylene	2379	86.7	14.4	44	130

Table C-18. Method 8082 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
12674-11-2	Aroclor 1016	3356	87.1	13.8	46	129
11097-69-1	Aroclor 1254	184	80.1	15.4	34	127
11096-82-5	Aroclor 1260	3538	89.4	14.8	45	134

Table C-19. Method 8141 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
86-50-0	Azinphos-methyl	325	96.7	19.6	38	156
35400-43-2	Bolstar [Sulprofos]	270	93.5	15.1	48	139
786-19-6	Carbophenothion	237	96.6	12.5	59	134
2921-88-2	Chlorpyrifos	333	93.3	15.5	47	140
56-72-4	Coumaphos	321	98.4	20.5	37	160
8065-48-3	Demeton	254	80.2	12.4	43	117
333-41-5	Diazinon	328	87.9	15.2	42	134
62-73-7	Dichlorvos [DDVP]	322	90.6	17.2	39	142
60-51-5	Dimethoate	264	77.5	20.6	16	139
298-04-4	Disulfoton	332	86	19.5	28	145
2104-64-5	EPN	300	90.6	15.5	44	137
563-12-2	Ethion	160	99.3	13.5	59	140
13194-48-4	Ethoprop	325	87.8	13.5	47	128
52-85-7	Fampphur	192	90.6	14.6	47	134
115-90-2	Fensulfothion	324	87.1	20	27	147
55-38-9	Fenthion	325	88.7	14.9	44	134
121-75-5	Malathion	322	91.2	15.2	46	137
298-00-0	methyl Parathion	330	93.6	14.8	49	138

Table C-19. Method 8141 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
126-68-1	O,O,O-Triethyl phosphorothioate	186	79.8	13.3	40	120
56-38-2	Parathion	313	94.3	14.9	50	139
298-02-2	Phorate	330	82.6	19.8	23	142
299-84-3	Ronnel	328	91.6	15.5	45	138
122-34-9	Simazine	120	93	16.3	44	142
22248-79-9	Stirophos [Tetrachlorovinphos, Gardona]	153	91.2	16.3	42	140
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	238	89	12.2	52	126
297-97-2	Thionazine	192	83.5	13.3	44	124
34643-46-4	Tokuthion [Protothiofos]	320	90.7	15.1	45	136
327-98-0	Trichloronate	326	88.3	17.2	37	140

Table C-20. Method 8141 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
1912-24-9	Atrazine	262	82.1	12.5	45	120
86-50-0	Azinphos-methyl	689	88.9	15.4	43	135
35400-43-2	Bolstar [Sulprofos]	561	91.2	14.6	47	135
786-19-6	Carbophenothion	418	94.4	14.1	52	137
2921-88-2	Chlorpyrifos	644	90	14.2	47	133
56-72-4	Coumaphos	684	89.9	15.1	45	135
8065-48-3	Demeton	591	76.2	17.1	25	128
126-75-0	Demeton-S	134	91.4	23.6	21	162
333-41-5	Diazinon	684	86	14.4	43	129
62-73-7	Dichlorvos [DDVP]	682	88.3	16.4	39	138
60-51-5	Dimethoate	597	75.2	16.5	26	125
298-04-4	Disulfoton	753	85.1	16.3	36	134
2104-64-5	EPN	623	90	14.3	47	133
563-12-2	Ethion	345	93.3	17.1	42	145
13194-48-4	Ethoprop	620	88.8	12.2	52	125

Table C-20. Method 8141 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
55-38-9	Fenthion	712	89.7	15.8	42	137
121-75-5	Malathion	635	87.8	14.6	44	132
150-50-5	Merphos	704	79.6	17.8	26	133
298-00-0	Methyl parathion	795	91.9	14.2	49	134
126-68-1	O,O,O-Triethyl phosphorothioate	295	94.2	17.5	42	147
56-38-2	Parathion	713	92.9	13.7	52	134
298-02-2	Phorate	675	79.8	19	23	139
139-40-2	Propazine [Milogard]	241	86.7	11.8	51	122
299-84-3	Ronnel	740	87.1	15.1	42	133
22248-79-9	Stirophos [Tetrachlorovinphos, Gardona]	310	94.8	15.8	48	142
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	584	86.5	13.1	47	126
297-97-2	Thionazine	366	85.1	13.4	45	125
34643-46-4	Tokuthion [Protothiofos]	696	87.8	14.8	43	132
327-98-0	Trichloronate	556	82.8	18.2	28	137

Table C-21. Method 8151 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
93-76-5	2,4,5-T	1106	84.6	17.7	31	138
93-72-1	2,4,5-TP [Silvex]	1179	86.1	14.3	43	129
94-75-7	2,4-D	1256	86	19.3	28	144
94-82-6	2,4-DB	1030	88.2	17.9	34	142
19719-28-9	2,4-Dichlorophenylacetic Acid	1041	74	15.9	27	122
100-02-7	4-Nitrophenol	208	76.7	20	17	137
50594-66-6	Acifluorfen	206	79.8	18	26	134
1861-32-1	Dacthal (DCPA)	147	72.5	15.6	26	119
1918-00-9	Dicamba	1070	85.2	15.7	38	132
120-36-5	Dichloroprop	1033	91.4	21	28	155
94-74-6	MCPA	935	81.5	17.8	28	135
93-65-2	MCPP	807	88.7	18	35	143

Table C-22. Method 8151 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
93-76-5	2,4,5-T	1758	94.8	17.5	42	147
93-72-1	2,4,5-TP [Silvex]	2289	92.9	13.8	51	134
94-75-7	2,4-D	2396	98.4	17.7	45	152
94-82-6	2,4-DB	1427	94.1	19.7	35	153
19719-28-9	2,4-Dichlorophenylacetic Acid	905	85	17.7	32	138
100-02-7	4-Nitrophenol	245	89.8	17.4	38	142
50594-66-6	Acifluorfen	262	95.5	16.2	47	144
133-90-4	Chloramben	230	79.5	18.5	24	135
1861-32-1	Dacthal (DCPA)	160	76.2	13.6	36	117
75-99-0	Dalapon	1220	79	20	19	139
1918-00-9	Dicamba	1434	95.3	15.2	50	141
120-36-5	Dichloroprop	1404	102	18.8	46	159
94-74-6	MCPA	1284	89.2	18.2	35	144
93-65-2	MCPP	1137	95.2	20.7	33	157
7085-19-0	Mecoprop	126	97.4	21.2	34	161
87-86-5	Pentachlorophenol	1149	97.5	13.8	56	139

Table C-23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	11115	101.1	7.8	78	125
71-55-6	1,1,1-Trichloroethane	12156	101.6	9.4	73	130
79-34-5	1,1,2,2-Tetrachloroethane	11670	97	8.9	70	124
79-00-5	1,1,2-Trichloroethane	11772	99.7	7.2	78	121
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	9760	100.8	11.7	66	136
75-34-3	1,1-Dichloroethane	11856	100.4	8.1	76	125
75-35-4	1,1-Dichloroethene	12352	100.3	10.1	70	131
563-58-6	1,1-Dichloropropene	10793	100.5	8.3	76	125
87-61-6	1,2,3-Trichlorobenzene	10572	97.8	10.6	66	130

Table C-23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
96-18-4	1,2,3-Trichloropropane	10925	99.1	8.8	73	125
526-73-8	1,2,3-Trimethylbenzene	1948	99.8	6	82	118
120-82-1	1,2,4-Trichlorobenzene	10980	98	10.4	67	129
95-63-6	1,2,4-Trimethylbenzene	11085	98.7	7.9	75	123
96-12-8	1,2-Dibromo-3-chloropropane	11380	96.6	11.7	61	132
106-93-4	1,2-Dibromoethane	11408	100.1	7.3	78	122
95-50-1	1,2-Dichlorobenzene	11785	99.1	7.2	78	121
107-06-2	1,2-Dichloroethane	12328	100.5	9.2	73	128
17060-07-0	1,2-Dichloroethane-d4	5951	103.1	10.8	71	136
540-59-0	1,2-Dichloroethene	7748	99.9	7.3	78	122
78-87-5	1,2-Dichloropropane	12145	99.5	7.8	76	123
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	1269	97.8	11.3	64	132
108-70-3	1,3,5-Trichlorobenzene	4723	99.4	9.6	71	128
108-67-8	1,3,5-Trimethylbenzene	11080	98.4	8.4	73	124
541-73-1	1,3-Dichlorobenzene	11619	98.9	7.4	77	121
142-28-9	1,3-Dichloropropane	10713	99.1	7.3	77	121
542-75-6	1,3-Dichloropropene	3714	101.6	8.1	77	126
106-46-7	1,4-Dichlorobenzene	11848	97.5	7.6	75	120
105-05-5	1,4-Diethylbenzene	1896	96.6	5.9	79	114
123-91-1	1,4-Dioxane	7698	96.4	13.7	55	138
544-10-5	1-Chlorohexane	2543	100.4	9.8	71	130
594-20-7	2,2-Dichloropropane	10703	99.7	11.1	67	133
78-93-3	2-Butanone [MEK]	11514	99.6	16.3	51	148
126-99-8	2-Chloro-1,3-butadiene	6667	99	11.3	65	133
110-75-8	2-Chloroethyl vinyl ether	6957	96.1	17.6	43	149
95-49-8	2-Chlorotoluene	10838	98.5	7.9	75	122
591-78-6	2-Hexanone	11004	99.1	15.4	53	145
79-46-9	2-Nitropropane	4969	98.3	17.1	47	150
67-63-0	2-Propanol [Isopropyl alcohol]	1696	99.8	13.4	60	140
460-00-4	4-Bromofluorobenzene	6267	98.9	6.8	79	119
106-43-4	4-Chlorotoluene	10785	98.3	8.6	72	124
108-10-1	4-Methyl-2-pentanone [MIBK]	11364	99.6	11.6	65	135

Table C-23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
67-64-1	Acetone	11089	99.6	21.4	36	164
75-05-8	Acetonitrile	5697	98.5	14.8	54	143
107-02-8	Acrolein [Propenal]	7528	101.1	18	47	155
107-13-1	Acrylonitrile	8293	99.7	11.4	65	134
107-05-1	Allyl chloride	6908	101.1	11.2	68	135
71-43-2	Benzene	12853	99.2	7.4	77	121
100-44-7	Benzyl chloride	2743	92.1	9.4	64	120
108-86-1	Bromobenzene	10974	99.3	7.3	78	121
74-97-5	Bromochloromethane	11023	101.4	7.8	78	125
75-27-4	Bromodichloromethane	11850	101	8.5	75	127
75-25-2	Bromoform	11890	99.1	10.8	67	132
74-83-9	Bromomethane	11416	98.3	15	53	143
75-15-0	Carbon disulfide	11132	97.9	11.5	63	132
56-23-5	Carbon tetrachloride	12090	102.3	10.7	70	135
108-90-7	Chlorobenzene	12382	99.7	6.9	79	120
124-48-1	Chlorodibromomethane	11852	100.2	8.7	74	126
75-00-3	Chloroethane	11444	98.8	13.3	59	139
67-66-3	Chloroform	12344	100.3	7.6	78	123
74-87-3	Chloromethane	11876	93.3	14.3	50	136
156-59-2	cis-1,2-Dichloroethene	11645	99.9	7.6	77	123
10061-01-5	cis-1,3-Dichloropropene	11805	99.8	8.7	74	126
1476-11-5	cis-1,4-Dichloro-2-butene	977	106	12.4	69	143
110-82-7	Cyclohexane	8827	98.9	10.6	67	131
108-94-1	Cyclohexanone	3764	93.2	20.9	30	156
1868-53-7	Dibromofluoromethane	2142	98.1	6.8	78	119
74-95-3	Dibromomethane	10913	101.1	7.9	78	125
75-71-8	Dichlorodifluoromethane [Freon-12]	11467	88.9	20.1	29	149
75-43-4	Dichlorofluoromethane	717	100.8	18	47	155
60-29-7	Diethyl ether	6283	99.6	9.6	71	129
108-20-3	Diisopropyl ether	8542	98.3	9.7	69	127
64-17-5	Ethanol	3958	102.2	18.9	45	159
141-78-6	Ethyl acetate	4516	95.4	14.5	52	139

Table C-23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
97-63-2	Ethyl methacrylate	7075	98.9	9.9	69	129
637-92-3	Ethyl tert-butyl ether	7514	98.9	9.1	72	126
100-41-4	Ethylbenzene	12427	99.1	7.7	76	122
462-06-6	Fluorobenzene	689	97.3	5.4	81	114
142-82-5	Heptane	5420	93.4	14.9	49	138
87-68-3	Hexachlorobutadiene	10264	98.1	12.4	61	135
67-72-1	Hexachloroethane	3265	102.5	10.1	72	133
110-54-3	Hexane	7116	93.6	16.1	45	142
74-88-4	Iodomethane	9457	100.9	10.1	71	131
78-83-1	Isobutyl alcohol	6162	97.5	12.6	60	135
108-21-4	Isopropyl acetate [Acetic acid]	2885	94.2	12.2	58	131
98-82-8	Isopropylbenzene	11596	100.8	11.1	68	134
179601-23-1	m/p-Xylene [3/4-Xylene]	10612	100.4	7.7	77	124
126-98-7	Methacrylonitrile	6736	99.2	11.1	66	132
79-20-9	Methyl acetate	8320	98.7	15.2	53	144
80-62-6	Methyl methacrylate	7050	98.4	11.9	63	134
1634-04-4	Methyl tert-butyl ether [MTBE]	11253	98.9	8.7	73	125
108-87-2	Methylcyclohexane	8565	99.4	11.2	66	133
75-09-2	Methylene chloride	12024	98.9	9.7	70	128
123-86-4	n-Butyl acetate	2981	95.1	11	62	128
71-36-3	n-Butyl alcohol	4800	92.9	12.6	55	131
104-51-8	n-Butylbenzene	10921	98.7	9.7	70	128
103-65-1	n-Propylbenzene	10947	98.9	8.8	73	125
91-20-3	Naphthalene	10602	95.6	11.2	62	129
95-47-6	o-Xylene	11940	100	7.7	77	123
99-87-6	p-Isopropyltoluene [p-Cymene]	10953	100.3	9	73	127
76-01-7	Pentachloroethane	5957	102	11.1	69	135
107-12-0	Propionitrile [Ethyl cyanide]	6734	101	11.1	68	134
135-98-8	sec-Butylbenzene	10960	99	8.8	73	126
100-42-5	Styrene	11809	100.2	8	76	124
994-05-8	tert-Amyl methyl ether [TAME]	7153	99.8	8.9	73	126
75-65-0	tert-Butyl alcohol	7492	100.5	10.7	68	133
98-06-6	tert-Butylbenzene	10974	98.8	8.6	73	125

Table C-23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
127-18-4	Tetrachloroethene	12091	100.5	9.2	73	128
109-99-9	Tetrahydrofuran	8039	98	12.4	61	135
108-88-3	Toluene	12499	99.3	7.3	77	121
2037-26-5	Toluene-d8	6232	100.7	5.2	85	116
156-60-5	trans-1,2-Dichloroethene	11849	99.2	8.6	74	125
10061-02-6	trans-1,3-Dichloropropene	11805	100.9	9.8	71	130
110-57-6	trans-1,4-Dichloro-2-butene	8307	98.6	12.3	62	136
79-01-6	Trichloroethene	12440	100.2	7.6	77	123
75-69-4	Trichlorofluoromethane [Freon-11]	11530	101	13.1	62	140
108-05-4	Vinyl acetate	7260	100.3	16.9	50	151
75-01-4	Vinyl chloride	12129	95.6	13.2	56	135
1330-20-7	Xylenes [total]	8623	100.7	7.7	78	124

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	24511	101.1	7.6	78	124
71-55-6	1,1,1-Trichloroethane	28223	102.7	9.6	74	131
79-34-5	1,1,2,2-Tetrachloroethane	27450	96.4	8.3	71	121
79-00-5	1,1,2-Trichloroethane	27338	99.5	6.5	80	119
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	21122	103	11.1	70	136
75-34-3	1,1-Dichloroethane	28154	101.3	8	77	125
75-35-4	1,1-Dichloroethene	29436	101	10	71	131
563-58-6	1,1-Dichloropropene	23631	102	7.8	79	125
87-61-6	1,2,3-Trichlorobenzene	24271	98.7	10.1	69	129
96-18-4	1,2,3-Trichloropropane	24525	97.5	8	73	122
526-73-8	1,2,3-Trimethylbenzene	2965	100.9	6.2	82	120
120-82-1	1,2,4-Trichlorobenzene	25290	99.8	10.1	69	130
95-63-6	1,2,4-Trimethylbenzene	27917	99.6	8	76	124
96-12-8	1,2-Dibromo-3-chloropropane	24955	94.9	11.1	62	128

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
106-93-4	1,2-Dibromoethane	29096	99	7.2	77	121
95-50-1	1,2-Dichlorobenzene	27583	99.4	6.5	80	119
107-06-2	1,2-Dichloroethane	32965	100.3	9.2	73	128
17060-07-0	1,2-Dichloroethane-d4	8673	99.5	6.1	81	118
540-59-0	1,2-Dichloroethene	18667	100.2	7.1	79	121
78-87-5	1,2-Dichloropropane	27787	100.1	7.2	78	122
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	3144	103.1	10.9	70	136
108-70-3	1,3,5-Trichlorobenzene	10037	102.1	9.2	75	130
108-67-8	1,3,5-Trimethylbenzene	27820	99.5	8.1	75	124
106-99-0	1,3-Butadiene	1202	100.6	19.2	43	158
541-73-1	1,3-Dichlorobenzene	26951	99.7	6.5	80	119
142-28-9	1,3-Dichloropropane	23811	99.1	6.5	80	119
542-75-6	1,3-Dichloropropene	9784	99.9	7.6	77	123
106-46-7	1,4-Dichlorobenzene	27715	98.3	6.5	79	118
105-05-5	1,4-Diethylbenzene	1980	98.4	6.4	79	118
123-91-1	1,4-Dioxane	17866	99	13.4	59	139
544-10-5	1-Chlorohexane	5790	99.6	8	76	124
540-84-1	2,2,4-Trimethylpentane [Isooctane]	5432	95.2	12.3	58	132
594-20-7	2,2-Dichloropropane	23775	99.7	13.2	60	139
75-85-4	2-Butanol	4332	92.7	9.1	66	120
78-93-3	2-Butanone [MEK]	26659	99.6	14.6	56	143
126-99-8	2-Chloro-1,3-butadiene	15673	100	11.7	65	135
110-75-8	2-Chloroethyl vinyl ether	18225	94.7	14.7	51	139
95-49-8	2-Chlorotoluene	23750	100	7.2	79	122
591-78-6	2-Hexanone	25368	97.9	13.5	57	139
91-57-6	2-Methylnaphthalene	3754	79.4	20.9	17	142
79-46-9	2-Nitropropane	10213	92.6	14.5	49	136
67-63-0	2-Propanol [Isopropyl alcohol]	2034	98.8	14.4	56	142
624-95-3	3,3-Dimethyl-1-butanol	6491	90.9	13.9	49	133
460-00-4	4-Bromofluorobenzene	9971	99.7	4.9	85	114
106-43-4	4-Chlorotoluene	23616	99.9	7.4	78	122

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
108-10-1	4-Methyl-2-pentanone [MIBK]	25796	98.5	10.6	67	130
67-64-1	Acetone	25006	99.5	20.1	39	160
75-05-8	Acetonitrile	13308	95.8	15.2	50	142
107-02-8	Acrolein [Propenal]	16380	96.8	19.3	39	155
107-13-1	Acrylonitrile	20173	99	11.9	63	135
107-05-1	Allyl chloride	15758	99	10.4	68	130
71-43-2	Benzene	34376	99.4	6.9	79	120
100-44-7	Benzyl chloride	10675	90.1	15.9	42	138
108-86-1	Bromobenzene	23762	99.7	6.7	80	120
74-97-5	Bromochloromethane	24356	100.8	7.5	78	123
75-27-4	Bromodichloromethane	26888	101.8	7.8	79	125
75-25-2	Bromoform	27675	97.8	10.8	66	130
74-83-9	Bromomethane	26717	97	14.7	53	141
75-15-0	Carbon disulfide	25719	98.8	11.5	64	133
56-23-5	Carbon tetrachloride	28870	103.8	10.7	72	136
108-90-7	Chlorobenzene	29802	100	6.1	82	118
124-48-1	Chlorodibromomethane	27424	100	8.5	74	126
75-45-6	Chlorodifluoromethane	7197	84.4	14.9	40	129
75-00-3	Chloroethane	27069	99	13	60	138
67-66-3	Chloroform	29373	101.1	7.5	79	124
74-87-3	Chloromethane	27697	94.5	15	50	139
156-59-2	cis-1,2-Dichloroethene	27935	100.1	7.5	78	123
10061-01-5	cis-1,3-Dichloropropene	27197	99.5	8	75	124
1476-11-5	cis-1,4-Dichloro-2-butene	1524	101.5	14.9	57	146
110-82-7	Cyclohexane	20438	100.4	10	71	130
1868-53-7	Dibromofluoromethane	5702	99.1	6.5	80	119
74-95-3	Dibromomethane	24473	101.1	7.3	79	123
75-71-8	Dichlorodifluoromethane [Freon-12]	25410	92	20.1	32	152
75-43-4	Dichlorofluoromethane	1504	101.5	9.8	72	131
60-29-7	Diethyl ether	17189	98.6	10.2	68	129
108-20-3	Diisopropyl ether	22989	97.5	10.3	67	128
64-17-5	Ethanol	9543	99.2	17.1	48	151

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
141-78-6	Ethyl acetate	9208	96.8	13.9	55	138
97-63-2	Ethyl methacrylate	16674	98.7	9	72	126
637-92-3	Ethyl tert-butyl ether	19841	98.3	9.4	70	127
100-41-4	Ethylbenzene	33325	99.8	7	79	121
462-06-6	Fluorobenzene	1373	97.9	6.1	80	116
142-82-5	Heptane	11878	94.4	15	49	140
87-68-3	Hexachlorobutadiene	23535	100.1	11.3	66	134
67-72-1	Hexachloroethane	8718	102.9	10.3	72	134
110-54-3	Hexane	15545	95.5	15.9	48	143
74-88-4	Iodomethane	20229	100	10.4	69	131
78-83-1	Isobutyl alcohol	14123	97.7	11.7	63	133
108-21-4	Isopropyl acetate [Acetic acid]	7216	97.8	11.6	63	133
98-82-8	Isopropylbenzene	28636	101.5	9.9	72	131
179601-23-1	m/p-Xylene [3/4-Xylene]	28168	100.5	6.9	80	121
126-98-7	Methacrylonitrile	15982	97.9	11.6	63	133
79-20-9	Methyl acetate	19698	96	13.2	56	136
80-62-6	Methyl methacrylate	16524	97.7	10.2	67	128
1634-04-4	Methyl tert-butyl ether [MTBE]	29660	97.3	8.8	71	124
108-87-2	Methylcyclohexane	20025	101.8	10.1	72	132
75-09-2	Methylene chloride	27659	99.4	8.3	74	124
123-86-4	n-Butyl acetate	7247	96.8	9.4	69	125
71-36-3	n-Butyl alcohol	10122	95.1	12	59	131
104-51-8	n-Butylbenzene	24088	101.1	8.8	75	128
109-60-4	n-Propyl acetate	602	100.8	8.3	76	126
103-65-1	n-Propylbenzene	24419	101	8.5	76	126
91-20-3	Naphthalene	27847	94.6	11.3	61	128
95-47-6	o-Xylene	31776	100	7.2	78	122
99-87-6	p-Isopropyltoluene [p-Cymene]	24335	102	8.5	77	127
76-01-7	Pentachloroethane	11688	101.1	10.7	69	133
109-66-0	Pentane	3915	74.8	19.7	16	134
107-12-0	Propionitrile [Ethyl cyanide]	15701	99.9	12	64	136
135-98-8	sec-Butylbenzene	24191	101.1	8.1	77	126
100-42-5	Styrene	26985	100.5	7.6	78	123

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
994-05-8	tert-Amyl methyl ether [TAME]	19726	98.1	10.1	68	128
75-65-0	tert-Butyl alcohol	21112	98.6	10.1	68	129
762-75-4	tert-Butyl formate	6651	98.1	11.1	65	132
98-06-6	tert-Butylbenzene	23919	101	7.7	78	124
127-18-4	Tetrachloroethene	29017	101.3	9.3	74	129
109-99-9	Tetrahydrofuran	18021	95	12.8	57	133
108-88-3	Toluene	33510	100.1	6.8	80	121
2037-26-5	Toluene-d8	9809	100.4	3.8	89	112
156-60-5	trans-1,2-Dichloroethene	27663	99.5	8.2	75	124
10061-02-6	trans-1,3-Dichloropropene	27134	100	8.9	73	127
110-57-6	trans-1,4-Dichloro-2-butene	19320	91.5	16.1	43	140
79-01-6	Trichloroethene	30150	101.1	7.3	79	123
75-69-4	Trichlorofluoromethane [Freon-11]	26108	103	12.8	65	141
108-05-4	Vinyl acetate	18941	100.2	15.3	54	146
75-01-4	Vinyl chloride	29472	97.4	13.2	58	137
1330-20-7	Xylenes [total]	23426	100.1	7	79	121

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	1645	78.5	13	40	117
95-94-3	1,2,4,5-Tetrachlorobenzene	1810	77.8	13.7	37	119
120-82-1	1,2,4-Trichlorobenzene	3577	75.7	13.9	34	118
95-50-1	1,2-Dichlorobenzene	3352	74.6	14	33	117
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	203	79.4	11.9	44	115
122-66-7	1,2-Diphenylhydrazine [Azobenzene]	2039	83	13.9	41	125
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	154	89.2	10.7	57	121
541-73-1	1,3-Dichlorobenzene	3288	72.6	14.1	30	115
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	598	84.6	14	43	127

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
106-46-7	1,4-Dichlorobenzene	3793	73.1	13.9	31	115
100-25-4	1,4-Dinitrobenzene	248	84.4	15.7	37	132
130-15-4	1,4-Naphthoquinone	150	81.2	8.8	55	108
90-13-1	1-Chloronaphthalene	119	81.1	11.1	48	115
90-12-0	1-Methylnaphthalene	3004	79.2	13.2	40	119
58-90-2	2,3,4,6-Tetrachlorophenol	1724	84.7	13.6	44	125
935-95-5	2,3,5,6-Tetrachlorophenol	227	75.9	11.9	40	112
608-27-5	2,3-Dichloroaniline	108	82.4	13	44	121
95-95-4	2,4,5-Trichlorophenol	4014	82.6	13.7	41	124
118-79-6	2,4,6-Tribromophenol	2930	85.7	15.4	39	132
88-06-2	2,4,6-Trichlorophenol	4183	82.1	14.5	39	126
120-83-2	2,4-Dichlorophenol	3794	80.9	13.7	40	122
105-67-9	2,4-Dimethylphenol	3886	78.4	16.2	30	127
121-14-2	2,4-Dinitrotoluene	4075	86.8	12.9	48	126
87-65-0	2,6-Dichlorophenol	1364	79.2	12.6	41	117
606-20-2	2,6-Dinitrotoluene	3706	85	13	46	124
53-96-3	2-Acetylaminofluorene	175	94	13.3	54	134
91-58-7	2-Chloronaphthalene	3569	77.5	12.1	41	114
95-57-8	2-Chlorophenol	3977	77.3	14.5	34	121
321-60-8	2-Fluorobiphenyl	3191	79.5	11.8	44	115
367-12-4	2-Fluorophenol	3008	75.2	13.3	35	115
91-57-6	2-Methylnaphthalene	5059	80.1	14	38	122
95-48-7	2-Methylphenol (o-Cresol)	4016	77	14.9	32	122
88-74-4	2-Nitroaniline	3639	85.4	13.8	44	127
119-75-5	2-Nitrodiphenylamine	279	88.1	11.6	53	123
88-75-5	2-Nitrophenol	3804	79.6	14.5	36	123
109-06-8	2-Picoline [2-Methylpyridine]	181	64.5	12.7	27	103
91-94-1	3,3'-Dichlorobenzidine	3521	71.3	16.5	22	121
56-49-5	3-Methylcholanthrene	188	95.1	13	56	134
99-09-2	3-Nitroaniline	3454	75.9	14.3	33	119
65794-96-9	3/4-Methylphenol [m/p-Cresol]	2900	76.5	14.1	34	119
534-52-1	4,6-Dinitro-2-methylphenol	3739	80.7	17.2	29	132
101-55-3	4-Bromophenyl phenyl ether	3708	85.1	13	46	124

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
59-50-7	4-Chloro-3-methylphenol	3880	83.3	12.9	45	122
106-47-8	4-Chloroaniline [p-Chloroanlinie]	3435	61.3	14.9	17	106
7005-72-3	4-Chlorophenyl phenyl ether	3673	83	12.7	45	121
106-44-5	4-Methylphenol [p-Cresol]	1555	84.1	14.1	42	126
100-02-7	4-Nitrophenol	3976	80.6	17	30	132
99-55-8	5-Nitro-o-toluidine [2-Amino-4-nitrotoluene]	187	69.8	15.8	23	117
57-97-6	7,12-Dimethylbenz(a)-anthracene	338	96.2	15.3	50	142
83-32-9	Acenaphthene	5300	81.3	13.7	40	123
208-96-8	Acenaphthylene	5194	81.8	16.8	32	132
98-86-2	Acetophenone	2101	73.9	13.6	33	115
120-12-7	Anthracene	5250	85.2	12.7	47	123
1912-24-9	Atrazine	1428	87.1	13.4	47	127
103-33-3	Azobenzene	378	82.1	14.2	39	125
56-55-3	Benz(a)anthracene	5385	87.4	12.9	49	126
50-32-8	Benzo(a)pyrene	5500	86.9	13.9	45	129
205-99-2	Benzo(b)fluoranthene	5323	88.3	14.5	45	132
191-24-2	Benzo(g,h,i)perylene	5263	88.5	15.1	43	134
207-08-9	Benzo(k)fluoranthene	5386	89.6	14.2	47	132
100-51-6	Benzyl alcohol	2895	75.7	15.6	29	122
111-91-1	bis(2-Chloroethoxy)methane	3705	78.4	14.2	36	121
111-44-4	Bis(2-chloroethyl) ether	3711	75.4	14.9	31	120
39638-32-9	bis(2-Chloroisopropyl) ether	769	82	16.3	33	131
117-81-7	Bis(2-ethylhexyl) phthalate	4018	91.9	13.7	51	133
103-23-1	bis(2-Ethylhexyl)adipate	156	90.8	10.1	61	121
85-68-7	Butyl benzyl phthalate	3956	90.3	14	48	132
105-60-2	Caprolactam	1203	81.3	11.9	46	117
86-74-8	Carbazole	3095	86.3	12	50	123
510-15-6	Chlorobenzilate	172	99.7	16.9	49	150
218-01-9	Chrysene	5395	87.1	12.2	50	124
84-74-2	Di-n-butyl phthalate	4041	89.4	12.8	51	128
117-84-0	Di-n-octyl phthalate	3985	92.4	16	45	140

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2303-16-4	Diallate [cis or trans]	173	93.7	12.7	56	132
53-70-3	Dibenzo(a,h)anthracene	5393	89.5	14.7	45	134
132-64-9	Dibenzofuran	3749	81.5	12.7	44	120
84-66-2	Diethyl phthalate	4012	87.2	12.3	50	124
60-51-5	Dimethoate	137	68	13.3	28	108
131-11-3	Dimethyl phthalate	4023	85.9	12.6	48	124
60-11-7	Dimethylaminoazobenzene	177	98.7	11.6	64	134
88-85-7	Dinoseb	123	67.3	17.1	16	119
101-84-8	Diphenyl ether	114	95.6	6	78	114
122-39-4	Diphenylamine	854	79.5	10.6	48	111
62-50-0	Ethyl methanesulfonate	174	85.1	16.9	34	136
206-44-0	Fluoranthene	5340	88.3	12.9	50	127
86-73-7	Fluorene	5150	84.2	13.8	43	125
118-74-1	Hexachlorobenzene	4138	83.5	13	45	122
87-68-3	Hexachlorobutadiene	4003	77.3	15.3	32	123
67-72-1	Hexachloroethane	4049	72.2	14.9	28	117
1888-71-7	Hexachloropropene	259	81.9	16.7	32	132
95-13-6	Indene	188	85.3	8.9	59	112
193-39-5	Indeno(1,2,3-cd)pyrene	5367	89.3	14.7	45	133
465-73-6	Isodrin	167	93.8	12.8	56	132
78-59-1	Isophorone	3787	75.9	15.2	30	122
120-58-1	Isosafrole	174	89.5	15.4	43	136
66-27-3	Methyl methanesulfonate	150	77.9	13.1	38	117
100-75-4	n-Nitorosopiperidine	232	89.4	9.8	60	119
924-16-3	n-Nitrosodi-n-butylamine	236	91.7	10.8	59	124
621-64-7	n-Nitrosodi-n-propylamine	3857	78.2	13.9	36	120
55-18-5	n-nitrosodiethylamine	421	82.1	13.8	41	124
62-75-9	n-Nitrosodimethylamine	3170	71.6	16.2	23	120
86-30-6	n-Nitrosodiphenylamine	2968	82.7	14.8	38	127
10595-95-6	n-Nitrosomethylethylamine	265	78.7	14.9	34	123
59-89-2	n-Nitrosomorpholine	172	91.3	13.8	50	133
930-55-2	n-Nitrosopyrrolidine	326	85.5	13.6	45	126
91-20-3	Naphthalene	5342	78.8	14.7	35	123

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
98-95-3	Nitrobenzene	4103	77.8	14.7	34	122
4165-60-0	Nitrobenzene-d5	3226	79.3	14.2	37	122
56-57-5	Nitroquinoline-1-oxide	177	91.3	24.5	18	165
126-68-1	O,O,O-Triethyl phosphorothioate	138	91.6	10.8	59	124
593-45-3	Octadecane	113	87.4	14.5	44	131
608-93-5	Pentachlorobenzene	346	89.7	11.8	54	125
76-01-7	Pentachloroethane	131	70.4	10.6	39	102
87-86-5	Pentachlorophenol	4161	78.7	18	25	133
82-68-8	Pentchloronitrobenzene	579	86.1	16	38	134
62-44-2	Phenacetin	185	95	12.5	57	133
85-01-8	Phenanthrene	5259	85.4	12	50	121
108-95-2	Phenol	4029	77.3	14.4	34	121
4165-62-2	Phenol-d5	1016	77.4	14.9	33	122
23950-58-5	Pronamide	179	93	12.4	56	130
129-00-0	Pyrene	5518	87.2	13.3	47	127
91-22-5	Quinoline	219	90	11.9	54	126
94-59-7	Safrole	176	87.8	13.6	47	129
1718-51-0	Terphenyl-d14	3111	90.5	12.3	54	127
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	136	94.4	14	52	137
297-97-2	Thionazine	139	94.6	10.7	62	127

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	2247	82.1	11.1	49	115
95-94-3	1,2,4,5-Tetrachlorobenzene	2326	77.9	14.5	35	121
120-82-1	1,2,4-Trichlorobenzene	4716	72.6	14.5	29	116
95-50-1	1,2-Dichlorobenzene	4442	71.4	13.3	32	111
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	112	83.9	8.3	59	109
122-66-7	1,2-Diphenylhydrazine [Azobenzene]	2244	85.4	12.2	49	122

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	241	89.1	16	41	137
541-73-1	1,3-Dichlorobenzene	4375	68.6	13.6	28	110
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	601	88.2	13.1	49	128
106-46-7	1,4-Dichlorobenzene	5433	70.4	13.9	29	112
90-13-1	1-Chloronaphthalene	211	84.5	8.8	58	111
90-12-0	1-Methylnaphthalene	3742	80	13.1	41	119
134-32-7	1-Naphthylamine	258	73.7	16.6	24	124
58-90-2	2,3,4,6-Tetrachlorophenol	2293	89	13	50	128
935-95-5	2,3,5,6-Tetrachlorophenol	266	85.6	11.7	50	121
608-27-5	2,3-Dichloroaniline	150	99.2	9.8	70	129
95-95-4	2,4,5-Trichlorophenol	5707	88.1	11.8	53	123
118-79-6	2,4,6-Tribromophenol	2059	91.5	16	43	140
88-06-2	2,4,6-Trichlorophenol	6136	87.2	12.4	50	125
120-83-2	2,4-Dichlorophenol	5330	84	12.2	47	121
105-67-9	2,4-Dimethylphenol	5298	77.5	15.6	31	124
51-28-5	2,4-Dinitrophenol	5127	82.9	20	23	143
121-14-2	2,4-Dinitrotoluene	6032	92.3	11.8	57	128
87-65-0	2,6-Dichlorophenol	1583	84	11.4	50	118
606-20-2	2,6-Dinitrotoluene	5107	90.7	11.2	57	124
53-96-3	2-Acetylaminofluorene	228	98.9	12.9	60	138
91-58-7	2-Chloronaphthalene	5084	78	12.8	40	116
95-57-8	2-Chlorophenol	5571	77.5	13.2	38	117
93951-73-6	2-Chlorophenol-d4	119	79.9	8.7	54	106
321-60-8	2-Fluorobiphenyl	2263	81.2	12.4	44	119
367-12-4	2-Fluorophenol	2022	68.8	16.6	19	119
91-57-6	2-Methylnaphthalene	6330	80.7	13.6	40	121
95-48-7	2-Methylphenol (o-Cresol)	5800	73	14.5	30	117
88-74-4	2-Nitroaniline	4855	90.8	12.1	55	127
119-75-5	2-Nitrodiphenylamine	272	97.3	11.3	64	131
88-75-5	2-Nitrophenol	5097	84.6	12.7	47	123
109-06-8	2-Picoline [2-Methylpyridine]	195	71.6	12.6	34	109
91-94-1	3,3'-Dichlorobenzidine	4815	77.9	16.9	27	129

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
56-49-5	3-Methylcholanthrene	237	94	12.8	56	133
99-09-2	3-Nitroaniline	4808	84.4	14.5	41	128
65794-96-9	3/4-Methylphenol [m/p-Cresol]	3472	69.7	13.6	29	110
534-52-1	4,6-Dinitro-2-methylphenol	5097	90.1	15.5	44	137
101-55-3	4-Bromophenyl phenyl ether	5074	89.1	11.5	55	124
59-50-7	4-Chloro-3-methylphenol	5338	85.5	11.3	52	119
106-47-8	4-Chloroaniline [p-Chloroanlinie]	4687	75.3	14	33	117
7005-72-3	4-Chlorophenyl phenyl ether	5071	86.7	11.3	53	121
106-44-5	4-Methylphenol [p-Cresol]	2798	72.5	15.8	25	120
99-55-8	5-Nitro-o-toluidine [2-amino-4-nitrotoluene]	260	82.1	14.6	38	126
57-97-6	7,12-Dimethylbenz(a)-anthracene	373	97.1	11.9	61	133
83-32-9	Acenaphthene	6952	84.5	12.3	47	122
208-96-8	Acenaphthylene	6662	85.3	14.7	41	130
98-86-2	Acetophenone	2877	82.1	12	46	118
120-12-7	Anthracene	6792	89.6	11	57	123
140-57-8	Aramite	100	82.8	16.3	34	132
1912-24-9	Atrazine	2328	92.8	16.4	44	142
103-33-3	Azobenzene	578	88.5	9.3	61	116
56-55-3	Benz(a)anthracene	6867	91.6	11.1	58	125
50-32-8	Benzo(a)pyrene	7045	90.8	12.4	54	128
205-99-2	Benzo(b)fluoranthene	6767	92	12.9	53	131
191-24-2	Benzo(g,h,i)perylene	6624	92	13.9	50	134
207-08-9	Benzo(k)fluoranthene	6803	93.2	12.1	57	129
100-51-6	Benzyl alcohol	3349	71.2	13.5	31	112
111-91-1	bis(2-Chloroethoxy)methane	5094	83.9	11.9	48	120
111-44-4	Bis(2-chloroethyl) ether	5139	80.8	12.6	43	118
39638-32-9	bis(2-Chloroisopropyl) ether	1140	83.4	15.4	37	130
117-81-7	Bis(2-ethylhexyl) phthalate	5288	95.2	13.3	55	135
85-68-7	Butyl benzyl phthalate	5173	93.3	13.5	53	134
86-74-8	Carbazole	4187	91.1	10.4	60	122
510-15-6	Chlorobenzilate	226	104.3	15.4	58	150

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
218-01-9	Chrysene	6779	91.3	10.7	59	123
124-18-5	Decane	126	66.9	12.8	29	105
84-74-2	Di-n-butyl phthalate	5329	93	11.4	59	127
117-84-0	Di-n-octyl phthalate	5222	95.5	15	51	140
2303-16-4	Diallate [cis or trans]	249	95.3	9.6	67	124
226-36-8	Dibenz(a,h)acridine	136	104.4	9.7	75	134
53-70-3	Dibenzo(a,h)anthracene	6840	92.7	13.8	51	134
132-64-9	Dibenzofuran	4963	85.3	10.8	53	118
84-66-2	Diethyl phthalate	5207	90.1	11.5	56	125
131-11-3	Dimethyl phthalate	4977	86	13.7	45	127
60-11-7	Dimethylaminoazobenzene	238	97.1	11.6	62	132
88-85-7	Dinoseb	144	93.4	10.8	61	126
101-84-8	Diphenyl ether	142	91.7	7.8	68	115
122-39-4	Diphenylamine	754	83	9.2	55	111
298-04-4	Disulfoton	122	92.5	12.5	55	130
62-50-0	Ethyl methanesulfonate	215	90.1	9.4	62	118
206-44-0	Fluoranthene	6826	92.6	11.9	57	128
86-73-7	Fluorene	6786	88.1	12	52	124
118-74-1	Hexachlorobenzene	6263	88.7	12.1	53	125
87-68-3	Hexachlorobutadiene	5878	73.1	16.9	22	124
67-72-1	Hexachloroethane	5904	68	15.7	21	115
95-13-6	Indene	253	93.8	13.7	53	135
193-39-5	Indeno(1,2,3-cd)pyrene	6880	92.6	13.6	52	134
465-73-6	isodrin	212	97.6	10	68	128
78-59-1	Isophorone	5190	83.3	13.7	42	124
120-58-1	Isosafrole	230	91.1	11.8	56	126
66-27-3	Methyl methanesulfonate	237	70.1	12.3	33	107
298-00-0	Methyl parathion	121	101.6	19	45	159
100-75-4	n-Nitorosopiperidine	299	88.6	10.8	56	121
924-16-3	n-Nitrosodi-n-butylamine	322	90.4	10.3	60	121
621-64-7	n-Nitrosodi-n-propylamine	5145	84	11.7	49	119
55-18-5	n-nitrosodiethylamine	488	81.8	12.9	43	121
86-30-6	n-Nitrosodiphenylamine	3743	86.8	11.9	51	123

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
10595-95-6	n-Nitrosomethylethylamine	311	78.7	12.7	41	117
59-89-2	n-Nitrosomorpholine	214	86.2	10.3	55	117
930-55-2	n-Nitrosopyrrolidine	716	80.8	10.8	48	113
91-20-3	Naphthalene	6953	80	13.5	40	121
98-95-3	Nitrobenzene	5955	83	12.8	45	121
4165-60-0	Nitrobenzene-d5	2223	82.1	12.6	44	120
126-68-1	O,O,O-Triethyl phosphorothioate	212	92.6	8.8	66	119
95-53-4	o-Toluidine	296	69.9	13.2	30	110
593-45-3	Octadecane	151	89	13.1	50	128
56-38-2	Parathion	152	102.6	12.3	66	140
608-93-5	Pentachlorobenzene	401	91.1	10.7	59	123
76-01-7	Pentachloroethane	139	60.9	10.4	30	92
87-86-5	Pentachlorophenol	6083	86.4	17.1	35	138
82-68-8	Pentchloronitrobenzene	618	94.5	13.4	54	135
62-44-2	Phenacetin	241	97.9	8.9	71	124
85-01-8	Phenanthrene	6822	89.6	10.2	59	120
298-02-2	Phorate	126	88.6	16.8	38	139
23950-58-5	Pronamide	249	97	10.5	65	129
129-00-0	Pyrene	7013	91.1	11.5	57	126
91-22-5	Quinoline	249	100.1	10.5	69	132
94-59-7	Safrole	233	90	9.7	61	119
1718-51-0	Terphenyl-d14	1893	91.7	13.9	50	134
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	200	96.7	11.9	61	133
297-97-2	Thionazine	196	102	10.1	72	132

Table C-27. Method 8270 Selected Ion Monitoring (SIM) Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
90-12-0	1-Methylnaphthalene	2267	76.6	11.3	43	111
95-95-4	2,4,5-Trichlorophenol	169	79.9	14.9	35	125

Table C-27. Method 8270 Selected Ion Monitoring (SIM) Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
91-58-7	2-Chloronaphthalene	615	76.7	10.5	45	108
321-60-8	2-Fluorobiphenyl	1961	80.6	11.6	46	115
91-57-6	2-Methylnaphthalene	2535	76.8	12.5	39	114
83-32-9	Acenaphthene	2813	77.7	11.2	44	111
208-96-8	Acenaphthylene	2761	77.1	12.8	39	116
120-12-7	Anthracene	2812	82.1	10.7	50	114
56-55-3	Benz(a)anthracene	2827	88	11.4	54	122
50-32-8	Benzo(a)pyrene	2789	87.3	12.5	50	125
205-99-2	Benzo(b)fluoranthene	2790	90.3	12.6	53	128
191-24-2	Benzo(g,h,i)perylene	2739	87.8	13	49	127
207-08-9	Benzo(k)fluoranthene	2761	89.3	11.2	56	123
111-44-4	Bis(2-chloroethyl) ether	192	65.4	15.8	18	113
117-81-7	Bis(2-ethylhexyl) phthalate	181	108.9	13.9	67	150
85-68-7	Butyl benzyl phthalate	144	103.5	10.6	72	135
86-74-8	Carbazole	183	79.3	14.6	36	123
218-01-9	Chrysene	2812	87.5	10.2	57	118
84-74-2	Di-n-butyl phthalate	150	106.5	12.9	68	145
117-84-0	Di-n-octyl phthalate	144	105.5	16.8	55	156
53-70-3	Dibenzo(a,h)anthracene	2778	89.2	13.2	50	129
132-64-9	Dibenzofuran	282	71.9	12.2	35	108
84-66-2	Diethyl phthalate	147	99.3	10.9	67	132
131-11-3	Dimethyl phthalate	149	99.3	9.3	71	127
206-44-0	Fluoranthene	2782	87.3	10.7	55	119
86-73-7	Fluorene	2795	80.6	11.2	47	114
118-74-1	Hexachlorobenzene	201	81.9	14.2	39	125
193-39-5	Indeno(1,2,3-cd)pyrene	2812	89.6	13.5	49	130
62-75-9	n-Nitrosodimethylamine	117	90.7	10.9	58	124
91-20-3	Naphthalene	2823	74.7	12.2	38	111
87-86-5	Pentachlorophenol	259	82.4	15.5	36	129
85-01-8	Phenanthrene	2792	80.8	10.6	49	113
129-00-0	Pyrene	2792	85.8	10.2	55	117

Table C-28. Method 8270 Selected Ion Monitoring (SIM) Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	106	77.3	7.3	56	99
90-12-0	1-Methylnaphthalene	2566	77.9	12.5	41	115
95-95-4	2,4,5-Trichlorophenol	488	84.1	13.4	44	124
118-79-6	2,4,6-Tribromophenol	164	83.7	12.7	46	122
606-20-2	2,6-Dinitrotoluene	118	67.2	15.8	20	115
91-58-7	2-Chloronaphthalene	717	72.4	12.7	34	111
321-60-8	2-Fluorobiphenyl	747	79.2	8.8	53	106
91-57-6	2-Methylnaphthalene	2984	76.5	12.6	39	114
83-32-9	Acenaphthene	3241	80.9	11.1	48	114
208-96-8	Acenaphthylene	3234	77.8	14.4	35	121
120-12-7	Anthracene	3224	85.8	11	53	119
56-55-3	Benz(a)anthracene	3277	89.3	10.1	59	120
50-32-8	Benzo(a)pyrene	3284	86.4	11.2	53	120
205-99-2	Benzo(b)fluoranthene	3248	89.7	12.3	53	126
191-24-2	Benzo(g,h,i)perylene	3178	86	14.1	44	128
207-08-9	Benzo(k)fluoranthene	3167	89.3	11.9	54	125
111-44-4	Bis(2-chloroethyl) ether	775	77.8	12.6	40	116
117-81-7	Bis(2-ethylhexyl) phthalate	275	114.1	19.6	55	173
85-68-7	Butyl benzyl phthalate	159	90.7	17.3	39	143
86-74-8	Carbazole	631	84	13.1	45	123
218-01-9	Chrysene	3215	88.3	10.4	57	120
84-74-2	Di-n-butyl phthalate	153	102.5	14.2	60	145
117-84-0	Di-n-octyl phthalate	157	103.3	19	46	160
53-70-3	Dibenzo(a,h)anthracene	3233	87.2	14.5	44	131
132-64-9	Dibenzofuran	864	77.5	14.1	35	120
84-66-2	Diethyl phthalate	142	94.5	13.5	54	135
206-44-0	Fluoranthene	3242	89.1	10.4	58	120
86-73-7	Fluorene	3232	84.1	11.3	50	118
118-74-1	Hexachlorobenzene	947	84.8	13	46	124
87-68-3	Hexachlorobutadiene	187	84.5	14.7	40	129
193-39-5	Indeno(1,2,3-cd)pyrene	3244	88.7	13.7	48	130
62-75-9	N-Nitrosodimethylamine	162	62.5	10	33	92
91-20-3	Naphthalene	3277	78.8	11.9	43	114

Table C-28. Method 8270 Selected Ion Monitoring (SIM) Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
4165-60-0	Nitrobenzene-d5	444	83.1	9.2	55	111
87-86-5	Pentachlorophenol	808	88.4	17.6	36	141
85-01-8	Phenanthrene	3240	83.6	10.3	53	115
129-00-0	Pyrene	3252	87.1	11.3	53	121
1718-51-0	Terphenyl-d14	642	95.1	12.4	58	132

Table C-29. Method 8290 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
3268-87-9	1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin	824	104.2	10.3	73	135
39001-02-0	1,2,3,4,6,7,8,9-Octachlorodibenzofuran	816	104.6	13	66	144
35822-46-9	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	813	100.7	8.1	76	125
67562-39-4	1,2,3,4,6,7,8-Heptachlorodibenzofuran	835	103.8	10.2	73	135
55673-89-7	1,2,3,4,7,8,9-Heptachlorodibenzofuran	823	101.1	9.8	72	131
39227-28-6	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	830	101.7	9.9	72	131
70648-26-9	1,2,3,4,7,8-Hexachlorodibenzofuran	835	103.1	8.9	77	130
57653-85-7	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	844	103.7	10	74	134
57117-44-9	1,2,3,6,7,8-Hexachlorodibenzofuran	837	103.6	10.3	73	134
19408-74-3	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	845	104.8	11.2	71	138
72918-21-9	1,2,3,7,8,9-Hexachlorodibenzofuran	895	104.6	10.1	74	135
40321-76-4	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	840	99.2	8.6	74	125
57117-41-6	1,2,3,7,8-Pentachlorodibenzofuran	803	103.7	8.9	77	131

Table C-29. Method 8290 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
60851-34-5	2,3,4,6,7,8-Hexachlorodibenzofuran	942	103.4	9.7	74	133
57117-31-4	2,3,4,7,8-Pentachlorodibenzofuran	912	101.4	8.9	75	128
1746-01-6	2,3,7,8-Tetrachlorodibenzo-p-dioxin	871	99	9.7	70	128
51207-31-9	2,3,7,8-Tetrachlorodibenzofuran	939	105.2	10.1	75	135

Table C-30. Method 8290 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
3268-87-9	1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin	539	107.7	9.1	81	135
39001-02-0	1,2,3,4,6,7,8,9-Octachlorodibenzofuran	553	107.9	14.1	66	150
35822-46-9	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	537	100.7	7.2	79	122
67562-39-4	1,2,3,4,6,7,8-Heptachlorodibenzofuran	574	105.2	8.1	81	130
55673-89-7	1,2,3,4,7,8,9-Heptachlorodibenzofuran	575	102.7	8.4	77	128
39227-28-6	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	568	102.9	7.7	80	126
70648-26-9	1,2,3,4,7,8-Hexachlorodibenzofuran	579	105	8.4	80	130
57653-85-7	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	585	105.7	9.4	78	134
57117-44-9	1,2,3,6,7,8-Hexachlorodibenzofuran	578	105.1	8.7	79	131
19408-74-3	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	585	106.6	10.1	76	137
72918-21-9	1,2,3,7,8,9-Hexachlorodibenzofuran	577	106.7	7.9	83	130
40321-76-4	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	579	98.6	7.5	76	121

Table C-30. Method 8290 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
57117-41-6	1,2,3,7,8-Pentachlorodibenzofuran	542	105.8	8	82	130
60851-34-5	2,3,4,6,7,8-Hexachlorodibenzofuran	597	105.5	8.1	81	130
57117-31-4	2,3,4,7,8-Pentachlorodibenzofuran	613	103.1	8.6	77	129
1746-01-6	2,3,7,8-Tetrachlorodibenzo-p-dioxin	635	97.9	9	71	125
51207-31-9	2,3,7,8-Tetrachlorodibenzofuran	641	104.9	11.1	72	138

Table C-31. Method 8310 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
90-12-0	1-Methylnaphthalene	740	88.3	16.1	40	137
91-57-6	2-Methylnaphthalene	742	87.3	15.7	40	135
83-32-9	Acenaphthene	826	87	13.2	47	127
208-96-8	Acenaphthylene	815	86.5	10.3	56	117
120-12-7	Anthracene	787	88.9	7.9	65	113
56-55-3	Benz(a)anthracene	838	97.3	9.5	69	126
50-32-8	Benzo(a)pyrene	838	91.3	9.6	63	120
205-99-2	Benzo(b)fluoranthene	838	95.8	8.2	71	120
191-24-2	Benzo(g,h,i)perylene	831	98.6	10	69	129
207-08-9	Benzo(k)fluoranthene	834	95	8.3	70	120
218-01-9	Chrysene	801	95.7	6.5	76	115
53-70-3	Dibenzo(a,h)anthracene	834	94.2	7.9	70	118
206-44-0	Fluoranthene	825	94.6	8.2	70	119
86-73-7	Fluorene	809	89.7	9.6	61	119
193-39-5	Indeno(1,2,3-cd)pyrene	675	98.9	11.6	64	134
91-20-3	Naphthalene	848	85.4	16.6	36	135
85-01-8	Phenanthrene	832	91.3	8.8	65	118
129-00-0	Pyrene	838	93.7	8.3	69	119

Table C-32. Method 8310 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
90-12-0	1-Methylnaphthalene	432	73.3	11	40	106
91-57-6	2-Methylnaphthalene	448	73.4	10.7	41	106
83-32-9	Acenaphthene	493	78.5	11.2	45	112
208-96-8	Acenaphthylene	478	80.5	9.1	53	108
120-12-7	Anthracene	453	85.8	9.2	58	113
56-55-3	Benz(a)anthracene	493	89	11.6	54	124
50-32-8	Benzo(a)pyrene	445	89.1	10.3	58	120
205-99-2	Benzo(b)fluoranthene	467	88.7	11.6	54	124
191-24-2	Benzo(g,h,i)perylene	428	88.6	11.3	55	122
207-08-9	Benzo(k)fluoranthene	460	88.4	11.8	53	124
218-01-9	Chrysene	469	90.3	9.6	61	119
53-70-3	Dibenzo(a,h)anthracene	452	87.2	10.5	56	119
206-44-0	Fluoranthene	485	86.9	10.6	55	119
86-73-7	Fluorene	483	82.2	9.7	53	111
193-39-5	Indeno(1,2,3-cd)pyrene	458	89.4	12.2	53	126
91-20-3	Naphthalene	440	73.3	10.5	42	105
85-01-8	Phenanthrene	489	85.2	9.5	57	114
129-00-0	Pyrene	472	86.3	9.3	58	114

Table C-33. Method 8321 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
99-35-4	1,3,5-Trinitrobenzene	228	92.4	7.6	69	115
99-65-0	1,3-Dinitrobenzene	234	102.4	6.5	83	122
118-96-7	2,4,6-Trinitrotoluene	222	99	11.4	65	133
121-14-2	2,4-Dinitrotoluene	229	100.7	6.1	82	119
606-20-2	2,6-Dinitrotoluene	225	99.7	4.6	86	113
35572-78-2	2-Amino-4,6-dinitrotoluene	230	102.2	9.2	75	130
88-72-2	2-Nitrotoluene	232	98.1	8.8	72	125
99-08-1	3-Nitrotoluene	235	96.8	9.5	68	125
19406-51-0	4-Amino-2,6-dinitrotoluene	230	101.2	8.1	77	125
99-99-0	4-Nitrotoluene	231	99.2	9.1	72	127

Table C-33. Method 8321 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
121-82-4	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (RDX)	231	100.2	7.6	77	123
98-95-3	Nitrobenzene	221	97.1	7.5	75	120
2691-41-0	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	225	89.3	8.1	65	114
78-11-5	PETN	229	102.3	13.6	62	143
479-45-8	Tetryl	214	78	13.9	36	120

Table C-34. Method 8321 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
99-35-4	1,3,5-Trinitrobenzene	452	88.6	7.4	66	111
99-65-0	1,3-Dinitrobenzene	460	98	6.5	78	118
118-96-7	2,4,6-Trinitrotoluene	413	98.4	10.1	68	129
121-14-2	2,4-Dinitrotoluene	458	96.4	6.9	76	117
606-20-2	2,6-Dinitrotoluene	447	93.7	4.7	80	108
35572-78-2	2-Amino-4,6-dinitrotoluene	456	97.9	9.6	69	127
88-72-2	2-Nitrotoluene	359	82	10.1	52	112
99-08-1	3-Nitrotoluene	356	83	9.7	54	112
19406-51-0	4-Amino-2,6-dinitrotoluene	459	96.7	9.7	68	126
99-99-0	4-Nitrotoluene	361	85.5	10.6	54	117
121-82-4	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	458	99.6	8.9	73	126
98-95-3	Nitrobenzene	353	84.6	7.7	61	108
2691-41-0	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	452	87	8.3	62	112
78-11-5	PETN	354	95	11	62	128
479-45-8	Tetryl	330	86.2	17.1	35	138

Table C-35. Method 8330 A/B Solid Matrix without Incremental Sampling Methodology (ISM)

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	339	105.7	5.7	89	123
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	607	101.9	7	81	123
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	602	104.2	6.7	84	124
118-96-7	2,4,6-Trinitrotoluene	618	100.2	8.4	75	125
121-14-2	2,4-Dinitrotoluene	600	102.3	6.9	82	123
606-20-2	2,6-Dinitrotoluene	556	102.4	5.4	86	119
35572-78-2	2-Amino-4,6-dinitrotoluene	562	103.8	5.7	87	121
88-72-2	2-Nitrotoluene	591	102	6	84	120
99-08-1	3-Nitrotoluene	614	103.3	8	79	127
19406-51-0	4-Amino-2,6-dinitrotoluene	594	104.2	6.7	84	124
99-99-0	4-Nitrotoluene	595	102.2	6.5	83	122
121-82-4	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	595	103.1	6.9	82	124
98-95-3	Nitrobenzene	598	103.9	7.9	80	128
55-63-0	Nitroglycerin	352	97.2	8.2	73	122
2691-41-0	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	581	99.1	7.5	77	122
78-11-5	PETN	326	100.9	7.5	78	123
479-45-8	Tetryl	584	101.8	11.9	66	138

Table C-36. Method 8330 A/B Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	978	101.1	6	83	119
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	1578	99	8.5	73	125
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	1572	98.7	7	78	120
118-96-7	2,4,6-Trinitrotoluene	1728	97	8.6	71	123
6629-29-4	2,4-Diamino-6-nitrotoluene	578	95	9.1	68	122
121-14-2	2,4-Dinitrotoluene	1563	98.9	7.1	78	120
59229-75-3	2,6-Diamino-4-nitrotoluene	577	96.6	8.3	72	122
606-20-2	2,6-Dinitrotoluene	1693	102	8.3	77	127

Table C-36. Method 8330 A/B Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
35572-78-2	2-Amino-4,6-dinitrotoluene	1568	99.4	6.8	79	120
88-72-2	2-Nitrotoluene	1630	98.4	9.6	70	127
618-87-1	3,5-Dinitroaniline	150	94.3	7.6	71	117
99-08-1	3-Nitrotoluene	1643	98.8	8.8	73	125
19406-51-0	4-Amino-2,6-dinitrotoluene	1586	100.3	8	76	125
99-99-0	4-Nitrotoluene	1654	99.1	9.3	71	127
121-82-4	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	1833	99.1	10.4	68	130
80251-29-2	Hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine (DNX)	109	92.8	8.8	66	119
5755-27-1	Hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine (MNX)	249	94.3	12.5	57	132
98-95-3	Nitrobenzene	1743	99.3	11.4	65	134
55-63-0	Nitroglycerin	1076	100.4	8.8	74	127
2691-41-0	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	1755	100	11.8	65	135
78-11-5	PETN	1079	100.2	9	73	127
479-45-8	Tetryl	1597	95.8	10.7	64	128

Table C-37. Method 8330B Solid Matrix with Incremental Sampling Methodology (ISM)

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
528-29-0	1,2-Dinitrobenzene [1,2-DNB]	283	98.9	6.8	78	119
99-35-4	1,3,5-Trinitrobenzene [1,3,5-TNB]	450	98	6.1	80	116
99-65-0	1,3-Dinitrobenzene [1,3-DNB]	461	96.3	7.7	73	119
118-96-7	2,4,6-Trinitrotoluene	443	95.8	8.2	71	120
121-14-2	2,4-Dinitrotoluene	457	98	7.5	75	121
606-20-2	2,6-Dinitrotoluene	430	98	6.3	79	117
35572-78-2	2-Amino-4,6-dinitrotoluene	455	96.5	8.7	71	123
88-72-2	2-Nitrotoluene	447	96.8	9.1	70	124
618-87-1	3,5-Dinitroaniline	115	101.6	5.3	86	118
99-08-1	3-Nitrotoluene	448	97.7	10.3	67	129
19406-51-0	4-Amino-2,6-dinitrotoluene	434	95.4	10.6	64	127

Table C-37. Method 8330B Solid Matrix with Incremental Sampling Methodology (ISM)						
99-99-0	4-Nitrotoluene	451	97.3	8.9	71	124
121-82-4	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	457	97.9	10.3	67	129
98-95-3	Nitrobenzene	440	97.9	10.4	67	129
55-63-0	Nitroglycerin	386	98.1	8.5	73	124
2691-41-0	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	422	99.1	8.2	74	124
78-11-5	PETN	376	100.1	9.4	72	128
479-45-8	Tetryl	377	101.3	11.1	68	135

Table C-38. Method 9010 - 9020 Series Solid Matrix						
CAS ID	**Analyte**	**N Records**	**Mean**	**Standard Deviation**	**Lower Control Limit**	**Upper Control Limit**
57-12-5	Cyanide, Total	842	98.2	7.4	76	120

Table C-39. Method 9010 - 9020 Series Aqueous Matrix						
CAS ID	**Analyte**	**N Records**	**Mean**	**Standard Deviation**	**Lower Control Limit**	**Upper Control Limit**
57-12-5	Cyanide, Total	1660	99	5.5	83	116

Table C-40. Method 9056 Solid Matrix						
CAS ID	**Analyte**	**N Records**	**Mean**	**Standard Deviation**	**Lower Control Limit**	**Upper Control Limit**
24959-67-9	Bromide	222	101	5.1	86	116
16887-00-6	Chloride	612	100.9	4.7	87	115
16984-48-8	Fluoride	300	100.3	9.1	73	128
14797-55-8	Nitrate	680	99.2	4	87	111
14797-65-0	Nitrite	419	100.3	4.9	86	115
14265-44-2	Phosphate	142	102.4	3.8	91	114
14808-79-8	Sulfate	305	100.9	4.7	87	115

Table C-41. Method 9056 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
24959-67-9	Bromide	2199	100.3	3.2	91	110
16887-00-6	Chloride	4948	98.5	4	87	111
16984-48-8	Fluoride	3251	99.7	4	88	112
14797-55-8	Nitrate	3192	99.7	3.9	88	111
14797-65-0	Nitrite	2583	98.9	3.9	87	111
14265-44-2	Phosphate	843	97.8	6.1	80	116
14808-79-8	Sulfate	4155	99.2	4.1	87	112

Table C-42. Method RSK-175 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
74-86-2	Acetylene	719	99.6	9.8	70	129
106-97-8	Butane	262	97.3	7.3	75	119
124-38-9	Carbon dioxide	441	100.8	6.9	80	122
74-84-0	Ethane	2240	102.6	9.6	74	131
74-85-1	Ethylene	2284	102.5	10.2	72	133
75-28-5	Isobutane	267	97.6	6.6	78	117
74-82-8	Methane	2459	99.2	8.7	73	125
74-98-6	Propane	900	98.1	8.2	74	123

Table C-43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	1344	97.9	10.5	67	129
71-55-6	1,1,1-Trichloroethane	5436	96.7	9.5	68	125
79-34-5	1,1,2,2-Tetrachloroethane	5273	95.9	10.4	65	127
79-00-5	1,1,2-Trichloroethane	5332	95.9	7.7	73	119
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	5351	96.1	10	66	126
75-34-3	1,1-Dichloroethane	5422	97	9.7	68	126
75-35-4	1,1-Dichloroethene	3503	97.3	11.9	61	133

Table C-43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
96-18-4	1,2,3-Trichloropropane	465	99.6	8	76	124
120-82-1	1,2,4-Trichlorobenzene	4545	98.5	14.5	55	142
95-63-6	1,2,4-Trimethylbenzene	4699	99.2	11.1	66	132
106-93-4	1,2-Dibromoethane	4655	98.2	7.9	74	122
76-14-2	1,2-Dichloro-1,1,2,2-tetrafluoroethane	4572	92.4	9.7	63	121
95-50-1	1,2-Dichlorobenzene	4739	95.7	11	63	129
107-06-2	1,2-Dichloroethane	5467	96.8	10.5	65	128
78-87-5	1,2-Dichloropropane	4729	95.7	8.9	69	123
108-67-8	1,3,5-Trimethylbenzene	4679	98.3	10.4	67	130
106-99-0	1,3-Butadiene	3167	99.8	11.4	66	134
541-73-1	1,3-Dichlorobenzene	4737	97.1	10.9	65	130
142-28-9	1,3-Dichloropropane	165	105.2	14.4	62	148
542-75-6	1,3-Dichloropropene	560	100.7	8.1	77	125
106-46-7	1,4-Dichlorobenzene	4719	95.8	11.8	60	131
123-91-1	1,4-Dioxane	2656	96.5	8.6	71	122
540-84-1	2,2,4-Trimethylpentane [Isooctane]	3008	94.3	8.8	68	121
78-93-3	2-Butanone [MEK]	4635	98.4	10.4	67	130
95-49-8	2-Chlorotoluene	1092	101.9	9.2	74	130
591-78-6	2-Hexanone	4600	95.4	11	62	128
67-63-0	2-Propanol [Isopropyl alcohol]	3069	88.4	12.3	52	125
622-96-8	4-Ethyltoluene	4673	97.9	10.3	67	129
108-10-1	4-Methyl-2-pentanone [MIBK]	4646	98.5	10.5	67	130
67-64-1	Acetone	4600	92.7	11.6	58	128
75-05-8	Acetonitrile	1999	97.3	11.6	63	132
107-02-8	Acrolein [Propenal]	2469	93.8	10.6	62	126
107-13-1	Acrylonitrile	2105	103.7	10.9	71	137
107-05-1	Allyl chloride	2980	101.1	10.1	71	131
98-83-9	alpha-Methylstyrene	1976	97.3	10.2	67	128
71-43-2	Benzene	5436	93.8	8.4	69	119
100-44-7	Benzyl chloride	4419	98.7	16.2	50	147
75-27-4	Bromodichloromethane	4682	99.9	9.3	72	128
75-25-2	Bromoform	4638	102.3	12.1	66	139

Table C-43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
74-83-9	Bromomethane	2657	98.6	11.8	63	134
106-97-8	Butane	587	96.2	10.9	64	129
75-15-0	Carbon disulfide	4756	95.6	12.7	57	134
56-23-5	Carbon tetrachloride	4202	99.6	10.7	68	132
108-90-7	Chlorobenzene	4652	94.5	8	70	119
124-48-1	Chlorodibromomethane	4628	99.9	10	70	130
75-45-6	Chlorodifluoromethane	559	102.1	14.3	59	145
75-00-3	Chloroethane	5370	94.7	10.6	63	127
67-66-3	Chloroform	5481	95.3	9.3	68	123
74-87-3	Chloromethane	4540	95.2	12.2	59	132
156-59-2	cis-1,2-Dichloroethene	5320	95.6	8.4	70	121
10061-01-5	cis-1,3-Dichloropropene	4691	98.8	9.7	70	128
110-82-7	Cyclohexane	3178	93.5	7.7	70	117
124-18-5	Decane	1982	93.8	7.9	70	118
75-71-8	Dichlorodifluoromethane[Freon-12]	5307	93.6	11.5	59	128
108-20-3	Diisopropyl ether	2309	93.5	8	70	117
64-17-5	Ethanol	2981	91.8	11.1	59	125
141-78-6	Ethyl acetate	2835	96.4	10.5	65	128
100-41-4	Ethylbenzene	5420	96.8	9	70	124
142-82-5	Heptane	3163	95.7	8.9	69	123
87-68-3	Hexachlorobutadiene	4551	96.7	13.7	56	138
110-54-3	Hexane	3150	91.6	9.5	63	120
98-82-8	Isopropylbenzene	3022	95.6	9.3	68	124
179601-23-1	m/p-Xylene [3/4-Xylene]	5019	97.3	12.3	61	134
80-62-6	Methyl methacrylate	3037	98.9	9.7	70	128
1634-04-4	Methyl tert-butyl ether [MTBE]	4681	95.5	10	66	126
75-09-2	Methylene chloride	5314	88.8	8.9	62	115
71-36-3	n-Butyl alcohol	1981	97.5	11.7	62	133
104-51-8	n-Butylbenzene	2656	97.7	10.6	66	130
112-40-3	n-DoDecane	1932	104.4	14.1	62	147
103-65-1	n-Propylbenzene	2570	95.7	9	69	123

Table C-43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
91-20-3	Naphthalene	2439	97.5	13.4	57	138
111-84-2	Nonane	2617	95.4	10.8	63	128
95-47-6	o-Xylene	5334	96.3	9.7	67	125
111-65-9	Octane	2514	95	8.7	69	121
99-87-6	p-Isopropyltoluene [p-Cymene]	2694	98.1	10.5	67	130
109-66-0	Pentane	712	96.7	11.3	63	131
115-07-1	Propene	3193	96.6	13.3	57	136
135-98-8	sec-Butylbenzene	2665	96.4	9.6	68	125
100-42-5	Styrene	4735	100.1	9	73	127
75-65-0	tert-Butyl alcohol	2997	86.8	20.9	24	150
98-06-6	tert-Butylbenzene	2710	94.3	9.8	65	124
127-18-4	Tetrachloroethene	5432	95.2	9.7	66	124
109-99-9	Tetrahydrofuran	3192	93.7	9.8	64	123
108-88-3	Toluene	5406	92.7	8.8	66	119
156-60-5	trans-1,2-Dichloroethene	5411	95.5	9.5	67	124
10061-02-6	trans-1,3-Dichloropropene	4621	104	9.6	75	133
79-01-6	Trichloroethene	5478	96.7	8.7	71	123
75-69-4	Trichlorofluoromethane [Freon-11]	5376	93.7	10.6	62	126
1120-21-4	Undecane	1976	96.1	9	69	123
108-05-4	Vinyl acetate	4599	97.4	13.7	56	139
593-60-2	Vinyl bromide	1054	98.4	9.2	71	126
75-01-4	Vinyl chloride	5445	95.1	10.4	64	127

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Appendix D

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Appendix E

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