Department of Defense (DoD)

Department of Energy (DOE)

Consolidated

Quality Systems Manual (QSM) for

Environmental Laboratories

Based on ISO/IEC 17025:2005(E)

ISO/IEC 17025:2017(E)

and

The NELAC Institute (TNI) Standards, Volume 1, (September 2009)

DoD Quality Systems Manual Version 5.3
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Department of Defense (DoD)

Department of Energy (DOE)

Consolidated Quality Systems Manual (QSM) for Environmental Laboratories

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Note: This version of the DoD/DOE QSM supersedes previous versions of the Department of Energy Quality Systems for Analytical Services.

Preface

The Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) Data Quality Workgroup (DOE-DQW) participants developed this manual called the *DoD/DOE Quality Systems Manual (QSM) for Environmental Laboratories*. The DoD/DOE QSM provides baseline requirements for the establishment and management of quality systems for laboratories performing analytical testing services for the DoD and the DOE.

This manual is based on Volume 1 of The NELAC Institute (TNI) Standards (September 2009), which incorporates International Organization for Standards (ISO)/International Electrotechnical Commission (IEC) 17025:2005, *General requirements for the competence of testing and calibration laboratories* and ISO/IEC17025:2017. Conformance to the requirements contained in this manual is mandatory for any laboratory that is 1) seeking or maintaining accreditation in accordance with the DoD Environmental Laboratory Accreditation Program (ELAP) or 2) seeking or maintaining accreditation in accordance with the DOE Consolidated Audit Program Accreditation Program (DOECAP-AP). Laboratories that comply with the requirements of this manual must also comply with the TNI Standards (September 2009), ISO/IEC 17025:2005, and ISO/IEC 17025:2017 (where noted in grey boxes), unless specific provisions in those standards are superseded by this document. Laboratories that are accredited to ISO/IEC 17025:2017 will meet the ISO 17025:2017 grey box reference requirements of DoD/DOE QSM Version 5.3.

To alleviate issues of copyright and provide a manual that is freely available to all, this manual is presented in a format which must be used in conjunction with the TNI Standards (September 2009), ISO/IEC 17025:2005, and ISO/IEC 17025:2017 standards. DoD/DOE specific language is presented as text and appendices in the order in which topics are addressed in the TNI Standards. DoD/DOE text contains additional requirements, clarifications, and guidance to supplement the TNI Standards (September 2009), ISO/IEC 17025:2005, and ISO/IEC 17025:2017 language. New or more stringent requirements from the ISO/IEC 17025:2017 standard are referenced by grey boxes. Information that may be beneficial to a laboratory, but is not required, is marked as guidance. To the extent possible, DoD and DOE requirements have been consolidated. Text or appendices that are unique to either DoD or DOE are marked as such.

The DoD/DOE QSM is international in scope and applies to all laboratories 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. DoD ELAP Accreditation Bodies (AB) and the DOECAP-AP ABs will accredit laboratories to this version of the standard during their normal accreditation cycles. Laboratories will be permitted to continue work on existing contracts under the project required version of the DoD/DOE QSM. Laboratories must be assessed to this version of the DOD/DOE QSM during their routine accreditation cycle.

The DoD and DOE reserve the right to periodically, and as necessary, issue new requirements or clarification to current requirements. These addenda will be published as an official memo and posted on DoD Environment, Safety, and Occupational Health Network and Information Exchange (DENIX) and the DOE Sharepoint site.

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 that provided insight and guidance into the resolution of complex scientific issues that are now a part of this document. Moving forward, the goal of continued data quality improvement will always be at the forefront of both the DoD EDQW and DOE-DQW team.

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Acronym List

AA	Atomic Absorption
AB	Accreditation Body(ies)
AFFF	Aqueous Film Forming Foam
AIHA	American Industrial Hygiene Association
AK	Acceptable Knowledge
ANSI	American National Standards Institute
ASPM	Analytical Services Program Manager
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BS	Blank Spike
BSC	Background Subtraction Count
CAP	Corrective Action Plan
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CCVRF	Continuing Calibration Verification Response Factor
CFR	Code of Federal Regulations
CHP	Chemical Hygiene Plan
COC	Chain of Custody
CPM	Counts per Minute
CRM	Certified Reference Material
CS	Calibration Source
CSU	Combined Standard Uncertainty
CWA	Clean Water Act
DENIX	DoD Environment, Safety, and Occupational Health Network and Information Exchange
DER	Duplicate Error Ratio
DFTPP	Decafluorotriphenylphoshpine
DL	Detection Limit
DMC	Deuterated Monitoring Compound
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
ECHO	EPA's Public Domain Enforcement and History Online

EDQW	Environmental Data Quality Workgroup
EICP	Extracted Ion Current Profile
EIS	Extracted Internal Standard
ELAP	Environmental Laboratory Accreditation Program
EM	Electron Multiplier
EMPC	Estimated Maximum Possible Concentration
EPA	Environmental Protection Agency
EQL	Estimated Quantitation Limit
FAQ	Frequently Asked Questions
FLAA	Flame Atomic Absorption
FTS	Fluorotelomersulfonate
FWHM	Full Width at Half Maximum
g	gram
GC	Gas Chromatography
GC/MS	Gas Chromatography/ Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption
HASQARD	Hanford Analytical Services Quality Assurance Requirements Documents
HAZWOPER	Hazardous Waste Operator and Emergency Response
HPLC	High Performance Liquid Chromatography
HRCC	High-Resolution Concentration Calibration
ICAL	Initial Calibration
ICALCT	Initial Calibration - Cross-Talk Factors
ICALE	Initial Calibration - Efficiency
ICALSA	Initial Calibration - Self-Absorption Curve
ICB	Initial Calibration Blank
ICC	Instrument Contamination Check
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Solution
ICV	Initial Calibration Verification
ID	Identification
IDOC	Initial Demonstration of Capability
IEC	International Electrotechnical Commission
IECV	Efficiency Calibration Verification
IEEE	Institute of Electrical and Electronics Engineers
ILAC	International Laboratory Accreditation Cooperation
IS	Internal Standard
ISC	Instrument Sensitivity Check
ISM	Incremental Sampling Method
ISO	International Organization for Standardization
IT	Information Technology

keV	Kilo electron-volts (one thousand (10^3) electron-volts)
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
LDR	Linear Dynamic Range
LIMS	Laboratory Information Management System
LLCCV	Low-Level Calibration Check Standard
LLD	Lower Limit of Detection
LLQ	Lower Limit of Quantitation
LOD	Limit of Detection
LOQ	Limit of Quantitation
LRB	Laboratory Reagent Blank
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols
MB	Method Blank
MBM	Method Background Measurement
MCA	Multi-Channel Analyzer
MD	Matrix Duplicate
MDA	Minimum Detectable Activity
MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MeV	Mega electron-volts (one million (10^6) electron-volts)
MPC	Measurement Performance Criteria
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
NBL	New Brunswick Laboratory
NCSL	Refers to National Conference of Standards Laboratories (now called NCSL International or NCSLI)
NDA	Non-Destructive Assay
NELAC	National Environmental Laboratory Accreditation Conference (now referred to only as The NELAC Institute, or TNI)
NELAP	National Environmental Laboratory Accreditation Program
NEtFOSAA	N-ethyl Perfouorooctanesulfonamidoacetic Acid
NFPA	National Fire Protection Association
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NMeFOSAA	N-methyl Perfluorooctanesulfonamidoacetic Acid
NMI	National Metrological Institute
NOV	Notice of Violation
NR	Not reported
NRC	Nuclear Regulatory Commission

PAH	Polyaromatic Hydrocarbon
PCB	Polychlorinated byphenyl
PCDD	Polychlorinated dibenzodioxins
PCDF	Polychlorinated dibenzofurans
PDS	Post Digestion Spike
PE	Performance Evaluation
PFAS	Per- and Poly-fluoroalklyl Substances
PFBA	Perfluorobutyric Acid
PFBS	Perfluorobutanesulfonic Acid
PFHxS	Perfluorohexanesulfonic Acid
PFOA	Perfluorooctanoic Acid
PFOS	Perfluorooctanesulfonic Acid
PFPeA	Perfluoropentanoic Acid
POTW	Publicly Owned Treatment Works
PPE	Personal Protective Equipment
PPM	Parts Per Million
PT	Proficiency Testing
PTOB/PTPA	Proficiency Testing Oversight Body/ Proficiency Testing Provider Accreditor
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCAL	Method Calibration - Quench Curve
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RL	Reporting Limit
RML	Radioactive Materials License
ROI	Regions of Interest
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
RT	Retention Time
S/N	Signal to Noise Ratio
SAP	Sampling and Analysis Plan
SD	Standard deviation
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SI	Système International d’unitès (International System of Units)
SICP	Selected Ion Current Profile
SIM	Selected Ion Monitoring

SNM	Special Nuclear Material
SOP	Standard Operating Procedure
SPE	Solid Phase Extraction
SQA	Software Quality Assurance
STS	Sample Test Source
TCE	Trichloroethylene
TEQ	Toxicity Equivalents
TIC	Tentatively Identified Compound
TMU	Total Measurement Uncertainty
TNI	The NELAC Institute
TPU	Total Propagated Uncertainty
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
V&V	Verification and Validation
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compounds
WET	Whole Effluent Toxicity
WP	Water Pollution
WRM	Working Reference Material
WS	Water Supply

Volume 1, Module 1: Proficiency Testing (PT)

1.0 INTRODUCTION

This module provides baseline requirements for proficiency testing for laboratories performing analytical testing services for the Department of Defense (DoD) and the Department of Energy (DOE). **This module supersedes the entirety of Volume 1, Module 1 of The NELAC Institute (TNI) standards (September 2009), which incorporates ISO/IEC 17025:2005(E).**

2.0 REQUIREMENTS FOR ACCREDITATION (Section 2: DoD Only)

2.1 Initial Accreditation

2.1.1 Initial Accreditation for DoD ELAP

To obtain initial accreditation for the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP), the laboratory shall analyze at least two Proficiency Testing (PT) samples for each combination of analyte-matrix-method (e.g., Trichloroethylene (TCE)-water-Method 624, TCE-water-Method 8260, TCE-soil-Method 8260, lead-soil-Method 6010, or lead-soil-Method 6020) that corresponds to its scope of accreditation. Laboratories that combine multiple methods into one Standard Operating Procedure (SOP) (e.g., SOP that combines Method 624 volatiles & Method 8260 volatiles) can report those methods with a single PT sample. All other analyte-matrix-method combinations require unique PT samples.

2.1.2 PT Samples for Initial Accreditation

The PT samples used for initial accreditation shall be obtained from PT providers that are accredited under International Organization for Standardization/International Electrotechnical Commission (ISO/IEC) 17043:2010(E) (General Requirements for Proficiency Testing) from an International Laboratory Accreditation Cooperation (ILAC) approved signatory Accreditation Body (AB).

2.1.3 PT Samples not from ISO/IEC 17043:2010(E) Accredited PT Provider

When PT samples cannot be obtained from an ISO/IEC 17043:2010(E) accredited PT provider, the laboratory shall obtain permission to use non- ISO/IEC 17043:2010(E) PT providers from their AB prior to analyzing the PT sample. The requirements and criteria from the PT provider must be met by the laboratory for the PT sample to be considered successful.

2.1.4 PT Samples for Analyte-matrix-method not from PT Provider

When PT samples for an analyte-matrix-method combination cannot be obtained from any PT provider, and the analyte-matrix-method combination is required for a scope of accreditation, the laboratory shall submit this fact in writing to their DoD ELAP AB. Other measures (e.g., precision, bias, and selectivity) as outlined in the 2009 TNI Standard Test Modules 3-7 must be performed to satisfy the PT requirement until those PT samples are available.

2.1.5 Analysis Date of PT Samples

The PT samples analyzed by the laboratory for initial DoD ELAP accreditation shall be no more than 12 months old. The analysis date between PT samples shall be at least 15 calendar days apart if two or more successive PT samples are performed.

2.1.6 PT Study Determination

The success or failure of any analyte-matrix-method combination for a PT study shall be determined by the PT provider under the requirements of the governing regulatory or ISO/IEC 17043:2010(E) statistically derived program.

2.1.7 PT Samples Same as Regular Environmental Samples

PT samples must be analyzed and evaluated in the same manner as regular environmental samples. A laboratory shall employ the same quality control, sequence of preparation and analytical steps, and replicates as used when analyzing routine samples.

2.2 Continuing Accreditation

2.2.1 Maintaining Accreditation

To maintain DoD ELAP accreditation, the laboratory shall successfully analyze at least two PT samples per calendar year for each analyte-matrix-method combination on their scope of accreditation. These PT samples shall be analyzed not less than 4 months and not to exceed 8 months apart. A PT sample for Whole Effluent Toxicity (WET) testing is required at least once per year.

2.2.2 Laboratory PT History

The laboratory shall maintain a history of at least two (2) successful PT rounds out of the most recent three (3) attempts for each analyte-matrix-method combination on their scope of accreditation. If PT samples are required for corrective action to reestablish history of successful PT rounds, the analysis dates of successive corrective action PT samples shall be at least 15 calendar days apart.

2.2.3 Failure to Meet Criteria

Analyte-matrix-method combinations that do not meet the above criteria must be removed from the DoD ELAP scope of accreditation.

2.2.4 PT Samples Same as Regular Environmental Samples

PT samples must be analyzed and evaluated in the same manner as regular environmental samples. A laboratory shall employ the same quality control, sequence of preparation and analytical steps, and replicates as used when analyzing routine samples.

3.0 REQUIREMENTS FOR PARTICIPATION (Section 3: DOE Only)

3.1 Initial Accreditation

3.1.1 Initial Accreditation for DOECAP-Accreditation Program

To obtain initial accreditation for the Department of Energy Consolidated Audit Program-Accreditation Program (DOECAP-AP), the laboratory shall analyze at least two Proficiency Testing (PT) samples for each combination of analyte-matrix-method that corresponds to its scope of accreditation. Laboratories that combine multiple methods into one Standard Operating Procedure (SOP) (e.g., SOP that combines Method 624 volatiles and Method 8260 volatiles) can report those methods with a single PT sample provided the strictest Quality Control (QC) criteria are applied. All other analyte-matrix-method combinations may require other applicable PT samples as discussed under Section 3.1.2 below.

The laboratory shall demonstrate successful participation for a minimum of one year in an ISO/IEC 17043:2010(E) accredited PT program. The single blind studies must be related to regulatory or environmental programs, analytes, matrix types, and methods for each of the analytical disciplines (i.e., inorganic, organic, radiochemistry, biological, etc.) that each laboratory will perform in support of DOE sites. A laboratory is required to analyze PT samples in matrices containing analytes listed on the accreditation scope using methodology applicable to data they report under DOE contracts, where available. PT samples must be tested and evaluated in the same manner as regular environmental samples.

3.1.2 PT Samples for Initial Inclusion

PT providers will be required to maintain proficiency in an ISO/IEC 17043:2010(E) accredited PT program for all analytes, matrices, and methods that are included in the laboratory's scope of work as defined in the subcontracts issued by DOE sites. A laboratory must possess a radioactive materials license from the Nuclear Regulatory Commission, an Agreement State, or a DOE exemption authorized by the contract holder to receive PT samples that contain radiological materials.

Other programs (such as Drinking Water) require program-specific PT samples.

3.1.3 PT Samples not from an ISO/IEC 17043:2010(E) Accredited PT Provider

When PT samples cannot be obtained from an ISO/IEC 17043:2010(E) accredited PT provider, the laboratory shall obtain permission to use non- ISO/IEC 17043:2010(E) PT providers from their AB prior to analyzing the PT sample. The requirements and criteria from the AB and the PT provider must be met by the laboratory for the PT sample to be considered successful.

3.1.4 PT Samples for Analyte-Matrix-Method not from a PT Provider

When PT samples for an analyte-matrix-method combination cannot be obtained from any PT provider, and the analyte-matrix-method combination is required for a scope of accreditation, the laboratory shall submit this fact in writing to their DOECAP-AP Accreditation Body (AB). Other measures (e.g., precision, bias, and selectivity) as outlined in the appropriate 2009 TNI Standard Test Modules must be performed to satisfy the PT requirement until those PT samples are available.

3.1.5 Analysis Date of PT Samples

The PT sample analysis dates by the laboratory for initial DOECAP-AP accreditation shall be no more than 12 months old. The analysis date between PT samples shall be at least 7 calendar days apart if two or more successive PT samples are performed.

3.1.6 PT Study Determination

The success or failure of any analyte-matrix-method combination for a PT study shall be determined by the PT provider under the requirements of the governing regulatory or ISO/IEC 17043:2010(E) statistically derived program.

3.1.7 PT Samples Same as Regular Environmental Samples

PT samples must be tested and evaluated in the same manner as regular environmental samples. A laboratory shall employ the same quality control, sequence of preparation and analytical steps, and replicates as used when analyzing routine samples from DOE sites for the purposes of achieving accreditation.

3.2 Continued Participation

3.2.1 Maintaining Participation

The laboratory shall demonstrate continued proficiency throughout the term of the contract award. In addition, the client reserves the right to submit blind PT samples. Each laboratory shall continue to participate in all applicable rounds of external PT programs.

To maintain DOECAP-AP accreditation, the laboratory shall successfully analyze at least two PT samples, where available, per calendar year for each analyte-matrix-method combination on their scope of accreditation. Each PT sample shall be analyzed approximately six months apart (any time frame from four to eight months apart is considered acceptable) if two PT samples are analyzed.

3.2.2 Laboratory PT History

The laboratory shall maintain a history of at least two (2) successful PT rounds out of the most recent three (3) attempts for each analyte-matrix-method combination on their scope of accreditation.

3.2.3 Reporting Requirements to DOE Sites

The results of all PT programs will be utilized in the reports produced for DOE and the sites that have contracts with the laboratory. PT results from commercial PT studies will be provided to the Laboratory's applicable DOE contract holder sites no later than 10 days of receipt of results from the PT provider from the most recent PT study.

3.2.4 Failure to Meet Criteria

Any applicable analyte for which individual laboratory results are entered as NR or “not reported” will not be considered an acceptable result unless it is a remedial PT study. The success or failure of any analyte-matrix-method combination for a PT study shall be determined by the PT provider under the requirements of the governing regulatory or ISO/IEC 17043:2010(E) statistically derived program.

If the laboratory fails two consecutive evaluations or two (2) out of three (3) attempts, the laboratory should not receive samples from DOE sites for analysis by the failed analyte-matrix-method combination until acceptable PT performance has been achieved. However, the decision to withhold sample shipments will be at the discretion of the individual DOE contract holder. Analyte-matrix-method combinations that do not meet the above criteria must be removed from the DOECAP-AP scope of accreditation by the Accrediting Body.

The laboratory can demonstrate proficiency in remedial PT studies by acceptable performance in an unscheduled evaluation by the same PT program or by participation in the next regularly scheduled study. The use of quick turnaround and remedial samples will be acceptable, but the PT samples are included in the number of possible attempts at maintaining proficiency (i.e., receiving acceptable performance in two out of the last three rounds by analyte-matrix-method of proficiency testing reported). PT samples are required for corrective action to reestablish history of successful PT performance. The analysis dates of successive corrective action PT samples shall be at least 15 calendar days apart.

3.2.5 PT Samples Same as Regular Environmental Samples

PT samples must be analyzed and evaluated in the same manner as regular environmental samples. A laboratory shall employ the same quality control, sequence of preparation and analytical steps, and replicates as used when analyzing routine samples.

Volume 1, Module 2: Quality Systems General Requirements

1.0 INTRODUCTION, SCOPE, AND APPLICABILITY

1.1 Introduction

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.2 Scope DoD/DOE (Clarification)

*The following is a **clarification** of TNI section 1.2:*

The Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) participants developed the DoD/DOE Quality Systems Manual (QSM). This manual provides baseline requirements for the establishment and management of quality systems for laboratories performing analytical testing services for the DoD and the DOE. This manual is based on Volume 1 of The NELAC Institute (TNI) Standards (September 2009), which incorporates ISO/IEC 17025:2005(E), *General requirements for the competence of testing and calibration laboratories*. Conformance to the requirements contained in this manual is mandatory for any laboratory that is 1) seeking or maintaining accreditation in accordance with the DoD Environmental Laboratory Accreditation Program (ELAP) or 2) seeking or maintaining accreditation in accordance with the DOE Consolidated Audit Program Accreditation Program (DOECAP-AP) requirements and all applicable qualifications in accordance with the DOECAP and DOE related contract awards for analytical services and industrial hygiene testing. Laboratories that comply with the requirements of this manual must also comply with the TNI standards (September 2009) and ISO/IEC 17025:2005(E) unless superseded by this document. All references to the term "accreditation" in this manual refer to the DoD ELAP and the DOECAP-AP.

This manual is presented in a format which is designed for use in conjunction with the TNI (September 2009) and ISO/IEC 17025:2005(E) standards. Updated requirements from the ISO/IEC 17025:2017 standard are referenced by grey boxes. DoD/DOE specific language is presented as text and appendices in the order in which topics are addressed in the TNI standards. DoD/DOE text contains additional requirements, clarifications, and guidance to supplement the TNI and ISO/IEC language. Information that may be beneficial to a laboratory, but is not required, is marked as guidance. To the extent possible, DoD and DOE requirements

have been consolidated. Text or appendices that are unique to either DoD or DOE are marked as such.

The DoD/DOE QSM is international in scope and applies to all laboratories 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.

To ensure that laboratories are capable of generating data that will meet project-specific requirements, the EDQW and the DOECAP Data Quality Workgroup (DOE-DQW) strongly encourages the involvement of project chemists and laboratories during project-planning activities.

DOE Only (Clarification): Laboratories must follow the requirements of DOE Order 414.1B, C, or D as part of the laboratory contract. The DOECAP-AP will be an acceptable replacement to all references to the DOECAP program. DOECAP participation includes active participation in the DOECAP-AP.

Box 1
The following shall be implemented in addition to TNI section 1.2:
ISO/IEC 17025:2017(E) section 1

2.0 NORMATIVE REFERENCES (ISO/IEC 17025:2005(E), Clause 2)

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

Box 2
The following shall be implemented in addition to TNI section 2.0:
ISO/IEC 17025:2017(E) section 2

3.0 TERMS AND DEFINITIONS

3.1 Additional Terms and Definitions

*The following are DoD/DOE **clarifications and additions** to TNI section 3.1:*

Accreditation: Refers to accreditation in accordance with the DoD ELAP and the DOECAP-AP as applicable.

Accreditation Body (AB): 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 must 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.

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

Analytical Method: A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.

Analysis: A combination of sample preparation and instrument determination.

Analyte: 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.

Assessment (Clarification): Assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.

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

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.

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 prior to 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 in order to prevent recurrence. A root cause analysis may not be necessary in all cases.

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

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 (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).

Detection Limit (DL): 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.

Deuterated Monitoring Compounds (DMCs): SIM specific surrogates as specified for GC/MS SIM analysis.

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.

Documents: Written components of the laboratory management system (e.g., policies, procedures, and instructions).

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

False Negative: A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.

False Positive: A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.

Finding (Clarification): An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or

neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).

Holding Times (Clarification): The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.

Improper Actions: Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (i.e., DoD or DOE).

Incremental Sampling Method (ISM): Structured composite sampling and processing protocol having specific elements designed to reduce data variability and increase sample representativeness for a specified volume of soil under investigation.

Initial Calibration Verification (ICV): Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.

Injection Internal Standard Analyte: Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.

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

Laboratory Information Management Systems (LIMS): The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.

Limits of Detection (LOD) (Clarification): The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.

Limits of Quantitation (LOQ) (Clarification): The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.

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

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

Measurement Uncertainty: An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.

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

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

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

Quality System Matrix (Clarification): The matrix definitions in the TNI standard shall be used for purposes of 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.

Records: The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).

Reporting Limit: A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.

Selected Ion Monitoring (SIM): Using GC-MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.

Signal to Noise Ratio (S/N): 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 on a project-specific basis.

Test Method: A definitive procedure that determines one or more characteristics of a given substance or product.

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.).

Unethical Actions: Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.

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

Box 3

The following shall be implemented in addition to TNI section 3.1:
ISO/IEC 17025:2017(E) section 3

3.2 Sources

No additions or clarifications were made to this section. **TNI 2009 and ISO/IEC 17025:2005(E) standards shall be followed.**

3.3 Exclusions and Exceptions

No additions or clarifications were made to this section. **TNI 2009 and ISO/IEC 17025:2005(E) standards shall be followed.**

4.0 MANAGEMENT REQUIREMENTS

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

Box 4
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.1: ISO/IEC 17025:2017(E) sections 4.1, 4.2, 5.3, and 5.4

4.1.5 DoD/DOE Requirement

Box 5
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.1.5: ISO/IEC 17025:2017(E) sections 6.2.3, 6.2.4, and 6.2.5.d)

*The following shall be implemented **in addition to** ISO Clause 4.1.5 j):*

j) At a minimum, the following laboratory management staff (however named) shall be considered key managerial personnel:

i) Management (e.g., President, Chief Executive Officer, Chief Operating Officer, Laboratory Director);
ii) Technical Managers (e.g., Technical Director, Section Supervisors);
iii) Quality Managers;
iv) Support Systems and Administrative Managers (e.g., Laboratory Information Management System (LIMS) manager, purchasing manager, project managers); and
v) Customer Services Managers.

4.1.7.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 4.1.7.1 a) through h):*

i) implement, maintain, and improve the management system by using available tools such as audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews in efforts to monitor trends.

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

4.2.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 4.2.1:*

Copies of all management system documentation provided to DoD ELAP ABs, DOECAP-AP, or to personnel on behalf of DoD/DOE shall be in English.

4.2.2 DoD/DOE Requirement

Box 6
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.2.2: ISO/IEC 17025:2017(E) section 8.2.2

4.2.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 4.2.3:*

Top management (including 4.1.5 j) i) through iii)) shall be responsible for:

a) defining the minimum qualifications, experience, and skills necessary for all positions in the laboratory;

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

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

   i) each employee training file must contain a certification that the employee has read, understands, and is using the latest version of the management system records relating to his/her job responsibilities;

   ii) training courses or workshops on specific equipment, analytical techniques, or laboratory procedures shall all be recorded; and

   iii) review of analyst work by relevant technical managers on an on-going basis is recorded or another annual demonstration of capability is performed by one of the following:

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

b. at least four consecutive laboratory control samples with acceptable levels of precision and bias. The laboratory must determine the acceptable levels of precision and bias prior to analysis; or

c. if the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

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

e) ensuring adequate supervision of all personnel employed by the laboratory;

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

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

4.2.8.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 4.2.8.1 a) and b):*

c) The laboratory shall have a documented program to detect and deter improper or unethical actions. Data shall be produced according to the project-specific requirements as specified in the final, approved project-planning documents, such as the approved Quality Assurance Project Plan (QAPP), when these documents are provided to the laboratory. Following are the minimum elements of an acceptable program for detecting and deterring improper or unethical actions:

i) an ethics policy must be read and signed by all personnel;

ii) initial and annual ethics training must be conducted as described in Section 5.2.7;

iii) analysts must record an explanation and sign off on all manual changes to data; and

iv) where available in the instrument software, all electronic tracking and audit functions must be enabled.

4.2.8.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 4.2.8.2:*

The quality manager shall review (or oversee the review of) the quality manual at least annually and update it if needed.

4.2.8.4 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 4.2.8.4 a) through r):*

s) procedures for procurement of standards;

t) procedures for data management including validation, verification, and purging of electronic data and data systems;

u) procedures for manual entry of raw data from analytical measurements that are not interfaced to LIMS and the verification and records of the accuracy of manually entered data;

v) procedures for making changes to electronic data (including establishing the requirements for a hardcopy or electronic log to record all changes to electronic data that affect data quality);

w) procedures for how electronic data are processed, maintained, and reported;

x) procedures for ensuring that data review includes all quality-related steps in the analytical process, including sample preparation, dilution calculations, chromatography evaluation, and spectral interpretations. The SOP shall require that records of data review be maintained and available for external review;

y) a list of all current certifications and accreditations that the laboratory holds and the scope of certification or accreditation (with expiration date) for each;

z) health and safety (e.g., Chemical Hygiene Plan) **(DOE Only Requirement);** and

aa) materials (Waste) management; **(DOE Only Requirement).**

4.2.8.4 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 4.2.8.4 p):*

p) The procedures for audits and data reviews shall specify which records must be included in the review. Internal data reviews shall consist of a tiered or sequential system of verification, consisting of 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.

The analyst and verification review must include at least the following procedures:

i) determination of whether the results meet the laboratory-specific quality control criteria;

ii) checks to determine consistency with project-specific measurement performance criteria (MPCs) if available;

iii) checks to ensure that the appropriate sample preparatory and analytical SOPs and methods were followed and that chain of custody and holding time requirements were met.

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

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 12AM 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 12AM on October 2nd, or an exceedance will be considered to have occurred);

iv) checks to ensure that all calibration and quality control requirements were met; and

v) checks for complete and accurate explanations of anomalous results, corrections, and the use of data qualifiers in the Case Narrative.

The final administrative review shall verify that previous reviews were recorded properly and that the data package is complete.

In addition, the quality manager or designee shall review a minimum of 10% of all data packages for technical completeness and accuracy on a quarterly basis. This review is considered a part of overall data review and does not need to be completed before the data package is issued to the customer. If data quality issues are discovered during the review, the client shall be notified within 15 business days of the discovery of the issue.

If electronic audit trail functions are available, they must be in use at all times, and associated data must be accessible. If the instrument does not have an audit trail, the laboratory must have procedures to record the integrity of the data.

4.2.8.5 DoD/DOE Requirement

*The following shall be implemented **in addition to TNI** sections 4.2.8.5 a) through f):*

g) All technical SOPs (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.

h) The laboratory shall develop, maintain, and implement procedures, however named, for Chemical Hygiene, Waste Management, and Radiation Protection (as applicable) **(DOE Only Requirement).**

DoD/DOE (Guidance)

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

4.2.8.5 DoD/DOE Requirement

*The following shall be implemented **in addition t**o TNI sections 4.2.8.5 f) i) through xxiii):*

Each method shall include or reference the following topics where applicable:

xxiv) equipment/instrument maintenance;

xxv) computer hardware and software; and

xxvi) troubleshooting.

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

4.3.2.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 4.3.2.2 a) through d):*

e) affected personnel are notified of changes to management systems documents and supporting procedures, including technical documents;

f) reviews (internal or external) of management system documentation shall be maintained and made available for assessment; and

g) 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.

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

Box 7
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.4: ISO/IEC 17025:2017(E) section 7.1.3

*The following shall be implemented **in addition to** ISO Clause 4.4.4:*

4.4.4.1 DoD/DOE Requirement

Waivers from DoD/DOE QSM requirements must be requested in writing from the appropriate DoD or DOE Chemist or Contractor Project Chemist (however named) on a project-specific basis and shall include technical justification relating to the specific project for the waiver. Documentation of approval for the waiver must be maintained by the laboratory and readily available for review.

Box 8
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.4.4: ISO/IEC 17025:2017(E) section 7.1.4

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

Box 9
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.5: ISO/IEC 17025:2017(E) sections 6.6 and 7.1.1.c

*The following shall be implemented **in addition to** ISO clauses 4.5.1 through 4.5.4 and TNI section 4.5.5:*

4.5.6 DoD/DOE Requirement

Laboratories must ensure and document that subcontracted (sub-tier) laboratories meet the requirements of this standard.

4.5.7 DoD/DOE Requirement

Subcontracted laboratories performing analytical services must be accredited in accordance with the project.

4.5.8 DoD/DOE Requirement

Subcontracted laboratories must receive project-specific approval from the DoD or DOE customer before any samples are analyzed.

4.5.9 DoD/DOE Requirement

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.

4.5.10 DoD/DOE Requirement

All subcontracted or outsourced management system elements (such as data review, data processing, project management, and IT support) or outsourced personnel must comply with the laboratory's overall management system, must comply with the requirements of this standard, and must receive prior written approval and authorization from the DoD/DOE customer.

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

Box 10
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.6: ISO/IEC 17025:2017(E) sections 6.6 and 7.1.1.c

4.6.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 4.6.1:*

Records for services and supplies that may affect the quality of environmental tests must include the following, where applicable:

a) date of receipt;

b) expiration date;

c) source;

d) lot or serial number;

e) calibration and verification records;

f) accreditation or certification scopes/certificates; and

g) date opened **(DOE Only Requirement)**.

4.6.1 DoD/DOE (Guidance)

Examples of services and supplies that may affect the quality of environmental tests include but are not limited to: balance or pipette calibration, solvents, standards, reagents, and sample containers.

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

4.7.1 DoD/DOE (Clarification)

*The following is a **clarification** of ISO Clause 4.7.1:*

Examples of situations for which immediate clarification or feedback shall be sought from the customer include the following:

a) the customer has specified incorrect, obsolete, or improper methods;

b) methods require modifications to ensure achievement of project-specific objectives contained in planning documents (e.g., difficult matrix, poor performing analyte);

c) project planning documents (e.g., QAPP or Sampling and Analysis Plan (SAP)) are missing or requirements (e.g., action levels, detection and quantification capabilities) in the documents require clarification; or

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

4.8 Complaints (ISO/IEC 17025/2005(E), Clause 4.8)

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

Box 11

The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.8: ISO/IEC 17025:2017(E) sections 7.9.2 through 7.9.7

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

Box 12
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.9.1: ISO/IEC 17025:2017(E) sections 7.10.1 and 7.10.2

*The following shall be implemented **in addition to** ISO Clauses 4.9.1 and 4.9.2:*

4.9.3 DoD/DOE Requirement

The laboratory shall upon discovery, notify all affected customers of potential data quality issues resulting from nonconforming work within 15 business days. Notification shall be performed according to a written procedure. Records of corrections taken or proposed corrective actions to resolve the nonconformance shall be submitted to the customer(s) within 30 business days of discovery.

4.9.4 DoD/DOE Requirement

All DoD ELAP laboratories, and all DOECAP-AP laboratories, must report any instances of inappropriate and prohibited laboratory practices, as detailed in Section 5.2.7 of the DoD/DOE QSM, to their AB within 15 business days of discovery. Discovery includes findings of such inappropriate practices by laboratory staff or customer stakeholders. DoD ELAP laboratories, and all DOECAP-AP laboratories, must submit records of associated corrections taken or proposed corrective actions to their AB within 30 business days of discovery. The respective AB will then have the responsibility of informing the EDQW (for DoD) and the DOE Analytical Services Program Manager (ASPM) of the laboratory's deviation from the requirements of the DoD/DOE QSM. If the AB is not notified within 15 business days the AB will immediately suspend the laboratory's DoD ELAP and/or their DOECAP-AP accreditation, as applicable. The respective ABs, DOE, and the EDQW deem these infractions as quite serious and appreciate the cooperation from all involved parties.

4.10 Improvement (ISO/IEC 17025/2005(E), Clause 4.10)

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

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

Box 13
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.11:
ISO/IEC 17025:2017(E) sections 8.7.1.a, 8.7.1.b, 8.7.1.e, 8.7.1.f

*The following shall be implemented **in addition to** ISO Clauses 4.11.1 through 4.11.5 and TNI sections 4.11.6 through 4.11.7:*

4.11.8 DoD/DOE Requirement

The laboratory shall have and use a record system for tracking corrective actions to completion and for analyzing trends to prevent the recurrence of the nonconformance.

Corrective actions developed to address findings during DoD ELAP or DOECAP-AP assessments must be implemented. Any changes to reviewed corrective action plans must be reviewed by the DoD ELAP AB or the DOECAP-AP AB, as appropriate.

Box 14
The following shall be implemented in addition to DoD/DOE QSM Clause 4.11.8:
ISO/IEC 17025:2017(E) section 8.7.3

4.11.8 DoD/DOE (Guidance)

Willful avoidance of approved corrective action implementation may result in loss of DoD ELAP accreditation, DOECAP-AP accreditation, or result in DOECAP Priority I findings, if no accreditation exists. As a result, work may be discontinued until implementation is verified by the DoD ELAP AB or DOECAP-AP AB as appropriate.

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

4.12.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 4.12.1:*

Records of preventive actions shall be maintained for review.

Box 15
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.12:
ISO/IEC 17025:2017(E) section 8.5

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

4.13.1.2 DoD/DOE (Clarification)

*The following is a **clarification** of ISO Clause 4.13.1.2:*

Dual storage of records at separate locations is considered an acceptable option for the purpose of protecting records against fire, theft, or loss.

Box 16
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.13.2: ISO/IEC 17025:2017(E) section 7.5.2

4.13.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 4.13.3 g) i) and ii):*

iii) Records for changes made to data (either hardcopy or electronic) shall include the identification of the person who made the change and the date of change.

4.13.4 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 4.13.1, ISO Clause 4.13.2, and TNI section 4.13.3:*

Permanent, bound laboratory notebooks (logbooks) or notebooks with measures in place to prevent the removal or addition of pages are required if utilized. Electronic logbooks are acceptable. For permanent, bound logbooks the following applies:

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;

b) all notebook pages must 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; and

c) each laboratory notebook shall have a unique serial number clearly displayed.

4.13.5 DoD/DOE Requirement

The laboratory shall have procedures for the independent review of technical and quality records to ensure they are legible, accurate, and complete.

4.13.6 DoD/DOE Requirement

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

4.13.7 DoD/DOE Requirement

If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.13.8 DoD/DOE Requirement

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

4.13.9 DoD/DOE Requirement

All SOPs shall be archived for historical reference per regulatory or customer requirements. The laboratory must have a procedure for permanent laboratory closure and disposal of any remaining records associated with DoD/DOE analytical data.

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

Box 17

The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.14: ISO/IEC 17025:2017(E) section 8.8.2

4.14.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 4.14.2:*

Laboratories shall notify DoD/DOE clients within 15 business days of discovery of any investigation that casts doubt upon the validity of test results.

*The following shall be implemented **in addition to** TNI section 4.14.5:*

4.14.6 DoD/DOE Requirement

The audit schedule shall ensure that all areas of the laboratory are reviewed over the course of one year. The review shall include both technical and quality systems areas. The review shall also include raw electronic data files derived from test reports.

4.14.7 DoD/DOE Requirement

Audit personnel shall be trained and qualified in the specific management system element or technical area under review. Laboratories shall determine the training and qualification requirements for audit personnel, including quality managers, and shall establish procedures to ensure that audit personnel are trained and qualified (i.e., have the necessary education or experience required for their assigned positions). These requirements and procedures shall be recorded.

4.14.8 DoD/DOE Requirement

Management shall ensure that sufficient resources are available so that all internal audits shall be conducted by personnel independent of the activity to be audited. Personnel conducting independent assessments shall have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the results of such assessments to laboratory management.

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

Box 18

The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.15: ISO/IEC 17025:2017(E) sections 8.9.2 and 8.9.3

4.15.1 DoD/DOE (Clarification)

*The following is a **clarification** of ISO Clause 4.15.1:*

Management reviews and internal audits are separate activities. The management review shall not be performed in lieu of an internal audit. It is an independent, executive review of the laboratory's management system.

4.15.1 DOE Only Requirement

*The following shall be implemented **in addition to** ISO Clause 4.15.1:*

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).

4.16 Data Integrity Investigations (TNI Section 4.16)

DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 4.16:*

All DoD ELAP and DOECAP-AP laboratories must report any instances of inappropriate and prohibited laboratory practices, as detailed in Section 5.2.7 of the DoD/DOE QSM, to their AB within 15 business days of discovery. Discovery includes findings of such inappropriate practices by laboratory staff or customer stakeholders. DoD ELAP laboratories, and DOECAP-AP laboratories, must submit records of associated corrections taken or proposed corrective actions to their AB within 30 business days of discovery. The respective AB will then have the responsibility of informing the EDQW (for DoD-ELAP) and/or ASPM (for DOECAP-AP) of the laboratory's deviation from the requirements of the DoD/DOE QSM. The respective ABs, DOE, and the EDQW deem these infractions as quite serious and appreciate the cooperation from all involved parties.

5.0 TECHNICAL REQUIREMENTS

5.1 General (ISO/IEC 17025/2005(E), Clause 5.1)

No additions or clarifications were made to this section. **TNI 2009 and ISO/IEC 17025:2005(E) standards shall be followed.**

Box 19
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.1: ISO/IEC 17025:2017(E) section 6.1

5.2 Personnel (ISO/IEC 17025/2005(E), Clause 5.2)

Box 20
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.2: ISO/IEC 17025:2017(E) section 6.2

5.2.3 DoD/DOE (Clarification)

*The following is a **clarification** of ISO Clause 5.2.3:*

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

5.2.4 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.2.4:*

The job description elements itemized in the note following *ISO Clause 5.2.4* are minimum requirements.

5.2.7.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 5.2.7:*

Top management acknowledges its support for data integrity by implementing the specific requirements of the laboratory's data integrity program.

The following practices are prohibited:

a) fabrication, falsification, or misrepresentation of data;

   i) creating data for an analysis that was not performed (dry lab),

   ii) creating information for a sample that was not collected (dry lab),

   iii) using external analysts, equipment, and/or laboratories to perform analyses when not allowed by contract

b) improper clock setting (time traveling) or improper date/time recording;

   i) resetting the internal clock on an instrument to make it appear that a sample was analyzed within holding time when in fact it was not,

   ii) changing the actual time or recording a false time to make it appear that holding times were met, or changing the times for sample collection, extractions or other steps to make it appear that holding times were met

c) unwarranted manipulation of samples, software, or analytical conditions;

   i) unjustified dilution of samples,

   ii) manipulating GC/MS tuning data to produce an ion abundance result that appears to meet specific QC criteria,

   iii) changing the instrument conditions for sample analysis from the conditions used for standard analysis (e.g., changing EM voltage),

   iv) unwarranted manipulation of computer software (e.g., forcing calibration or QC data to meet criteria, removing computer operational codes such as the “M” flag, inappropriately subtracting background, or improperly manipulating the chromatographic or spectrophotometric baseline),

   v) turning off, or otherwise disabling, electronic instrument audit/tracking functions

d) misrepresenting or misreporting field or QC samples;

   i) representing spiked samples as being digested or extracted when this has not been done,

ii) substituting previously generated runs for a non-compliant calibration or QC run to make it appear that an acceptable run was performed,

iii) failing to prepare or analyze Method Blanks and the laboratory control sample (LCS) in the same manner that samples were prepared or analyzed,

iv) tampering with QC samples and results, including over spiking and adding surrogates after sample extraction,

v) performing multiple calibrations or QC runs (including CCVs, LCSs, spikes, duplicates, and blanks) until one meets criteria, rather than taking needed corrective action, and not documenting or retaining data for the other unacceptable data,

vi) deleting or failing to record non-compliant QC data to conceal the fact that calibration or other QC analyses were non-compliant

e) improper calibrations;

i) discarding points in the initial calibration to force the calibration to be acceptable,

ii) discarding points from an MDL study to force the calculated MDL to be higher or lower than the actual value,

iii) 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,

iv) performing improper manual integrations, including peak shaving, peak enhancing, or baseline manipulation to meet QC criteria or to avoid corrective actions

f) concealing a known analytical or sample problem;

g) concealing a known improper or unethical behavior or action;

h) failing to report the occurrence of a prohibited practice or known improper or unethical act to the appropriate laboratory or contract representative or to an appropriate government official.

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

Box 21
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.3: ISO/IEC 17025:2017(E) section 6.3.5

5.3.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.3.3:*

a) When cross-contamination is a possibility, samples suspected of containing high concentrations of analytes shall be isolated from other samples.

b) A Storage Blank must 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. Laboratories shall have written procedures and 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. The data from the analysis of the Storage Blanks shall be available for review.

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

5.4 Environmental Methods and Method Validation (ISO/IEC 17025/2005(E), Clause 5.4)

Box 22
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.4.2: ISO/IEC 17025:2017(E) section 7.2.1.5

Box 23
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.4.3: ISO/IEC 17025:2017(E) section 7.2.1.6

*The following shall be implemented **in lieu of** TNI sections 5.4.4 and 5.4.5:*

General ISO/IEC 17025:2005(E), Sections 5.4.4 and 5.4.5 are applicable to this standard.

Box 24
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.4:
ISO/IEC 17025:2017(E) sections 7.2.2.2 and 7.2.2.4

5.4.6 DoD/DOE Requirement

Box 25
The following shall be implemented in addition to TNI Section 5.4.6:
ISO/IEC 17025:2017(E) section 7.6.1

*The following shall be implemented **in addition to** TNI section 5.4.6:*

a) The exact nature of some test methods may preclude rigorous, statistically valid estimation of analytical uncertainty. In these cases the laboratory shall attempt to identify all components of analytical uncertainty and make a reasonable estimation and shall ensure that the form of data reporting does not give a wrong impression of the uncertainty. A reasonable estimation shall be based on knowledge of method performance and previous experience. When estimating the analytical uncertainty, all uncertainty components which are of importance in the given situation shall be taken into account.

b) In those cases where a well-recognized test method specifies limits to the values of the major source of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied the requirements on analytical uncertainty by following the test method and reporting instructions.

c) The laboratory is only responsible for estimating the portion of measurement uncertainty that is under its control. As stated in Section 5.10.3.1.c, test reports shall include a statement of the estimated analytical uncertainty only when required by the customer. If a project requires analytical uncertainty to be reported, the laboratory shall report the estimated uncertainty based on project-specific procedures or, if not available, any other scientifically valid procedures. The estimated analytical uncertainty can be expressed as a range (±) around the reported analytical results at a specified confidence level. A laboratory may report the in-house, statistically-derived LCS control limits based on historical LCS recovery data as an estimate of the minimum laboratory contribution to analytical uncertainty at a 99% confidence level. For testing laboratories, the laboratory shall ensure that the equipment used can provide the analytical portion of measurement uncertainty needed by the customer.

5.4.7 DoD/DOE Requirement

Box 26
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.4.7: ISO/IEC 17025:2017(E) sections 7.11.1 through 7.11.5

5.4.7.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.4.7.1:*

The laboratory shall establish SOPs:

a) to ensure that the reported data are free from transcription and calculation errors;

b) to ensure that all quality control measures are reviewed and evaluated before data are reported;

c) to address manual calculations; and

d) to address manual integrations.

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., the chromatograms obtained before and after the manual integration must be retained to permit reconstruction of the results). This requirement applies to all analytical runs including calibration standards and QC samples. The person performing the manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration (electronic signature is acceptable). Records for manual integrations may be maintained electronically as long as all requirements, including signature requirements, are met and the results can be historically reconstructed.

5.4.7.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clauses 5.4.7.2 a) through c):*

d) the laboratory shall have a procedure to ensure individual user names and passwords are required for all LIMS users. LIMS passwords shall be changed on a regular basis, at a minimum of once per year;

e) 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 and available for review;

f) periodic inspections (at least annually) of the LIMS shall be performed by the Quality Manager or designee to ensure the integrity of electronic data. The Quality Manager or designee shall maintain records of inspections and submit reports to laboratory management noting any problems identified with electronic data processing and stating the corrective actions taken;

g) the laboratory shall have a procedure to notify the customer prior to changes in LIMS software or hardware configuration that will adversely affect customer electronic data;

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

i) the laboratory shall have SOPs for:

   i) software development methodologies that are based on the size and nature of the software being developed;

   ii) testing and QC methods to ensure that all software accurately performs its intended functions, including:

      a. acceptance criteria,

      b. tests to be used,

      c. personnel responsible for conducting the tests,

      d. records of test results,

      e. frequency of continuing verification of the software, and

      f. test review and approvals.

   iii) software change control methods that include instructions for requesting, authorizing, testing (to include quality control), approving, implementing and establishing the priority of software changes. The requirements to be met by each software change must be documented;

   iv) software version control methods that record the software version currently used. Data sets are recorded with the date and time of generation and/or the software version used to generate the data set;

v) maintaining a historical file of software, software operating procedures, software changes, and software version numbers;

vi) defining the acceptance criteria, testing, records, and approval required for changes to LIMS hardware and communication equipment.

j) records available in the laboratory to demonstrate the validity of laboratory-generated software include:

i) software description and functional requirements;

ii) listing of algorithms and formulas;

iii) testing and QA records; and

iv) installation, operation and maintenance records.

k) Electronic Data Security measures must ensure:

i) individual user names and passwords have been implemented;

ii) operating system privileges and file access safeguards are implemented to restrict the user of the LIMS data to users with authorized access;

iii) all LIMS users are trained in computer awareness security on an annual basis;

iv) system events, such as log-on failures or break-in attempts, are monitored;

v) the electronic data management system is protected from the introduction of computer viruses;

vi) system backups occur on a regular and published schedule and can be performed by more than one person within an organization;

vii) testing of the system backups must be performed and recorded to demonstrate that the backup systems contain all required data; and

viii) 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.

5.5 Calibration Requirements (ISO/IEC17025:2005(E) Clause 5.5)

5.5.1 DoD/DOE Requirement

Box 27
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.5.1: ISO/IEC 17025:2017(E) section 6.4.1

5.5.2 DoD/DOE Requirement

Box 28
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.5.2: ISO/IEC 17025:2017(E) section 6.4.6

5.5.5 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.5.5 a) through h):*

i) date placed in service;

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

k) operational status; and

l) instrument configuration and settings.

Box 29
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.5.5: ISO/IEC 17025:2017(E) section 6.4.13.a and 6.4.13.f

5.5.8 DoD/DOE Requirement

Box 30
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.5.8: ISO/IEC 17025:2017(E) section 6.4.8

5.5.11 DoD/DOE Requirement

Box 31
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.5.11: ISO/IEC 17025:2017(E) section 6.4.11

5.5.13.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 5.5.13.1 a):*

a) The laboratory shall have procedures for recording catastrophic failure of support equipment (e.g., refrigerators, freezers) that address identification of affected samples and customer notification.

5.5.13.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 5.5.13.1 d):*

d) These checks must be performed in the expected use range using reference standards that are obtained, where available, from an accredited third party or a NMI (e.g., NIST) traceable to the International System of Units (SI).

5.5.13.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 5.5.13.1 a) through e):*

f) The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used, or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 5-1. Minimum requirements to follow in the absence of method-specific requirements.

Performance Check	Frequency	Acceptance Criteria
Balance verification check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ± 2% or ± 0.02g, whichever is greater Analytical balance: ± 0.1% or ± 0.5 mg, whichever is greater

Performance Check	Frequency	Acceptance Criteria
Balance Calibration	Annual	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Calibration of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e., 7 days per week) [Use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily. If a notification has been sent to laboratory personnel for out of control conditions, the laboratory is expected to respond with corrective actions within 24 hours of sent notification or the client of the affected samples will be notified.]	Refrigerators: 0˚C to 6˚C Freezers: ≤ -10˚C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, verify at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly Traceable thermometers shall be verified as required and correction factors used when appropriate	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ± 2% of nominal volume Precision: RSD ≤ 1% of nominal volume (based on 10 replicate measurements)

Performance Check	Frequency	Acceptance Criteria
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use and upon evidence of deterioration	Bias: Mean within ± 3% of nominal volume Precision: RSD ≤ 3% of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Daily before use	Bias: Mean within ± 2% of nominal volume Precision: RSD ≤ 1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, verify at the volume of use or using two volumes that bracket the range of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within ± 5% of set temperature
Water purification system	Daily prior to use	Per Laboratory SOP
Radiological Survey Equipment	Daily prior to use [The battery is checked; the physical integrity of the unit and high voltage is checked; a background reading is taken; and verified with a radiological source]	Per Laboratory SOP
Timer	Timer traceable to NIST where time is critical to the performance of a test	Per Laboratory SOP

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

5.6 Measurement Traceability (ISO/IEC 17025:2005(E), Clause 5.6)

5.6.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 5.6.1:*

General ISO/IEC 17025:2005(E), Clause 5.6.1 is applicable to this standard.

5.6.2 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 5.6.2:*

Specific Requirements ISO/IEC 17025:2005(E), Clause 5.6.2 is applicable to this standard.

5.6.4.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 5.6.4.2 a):*

a) Records for standards, reagents, and reference materials shall include lot numbers. Documentation for reagents and solvents shall be checked to ensure that the stated purity will meet the intended use, and the supporting records of the checks shall be filed in a manner that is retrievable.

5.6.4.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 5.6.4.2 d):*

d) The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

5.6.4.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 5.6.4.2 f):*

f) If a standard exceeds its expiration date and is not re-certified, the laboratory shall remove the standard or clearly designate it as acceptable for qualitative purposes only.

g) Standards and reference materials shall be stored separately from samples, extracts, and digestates and protected in an appropriate cabinet or refrigerator.

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

5.7.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.7.1:*

Sample handling procedures shall address laboratory practices for recording the presence of extraneous materials (e.g., rocks, twigs, vegetation) present in samples in the case of heterogeneous materials. To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01 g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example. The handling of multiphase samples shall be addressed in specific sampling procedures, as appropriate. The laboratory's sampling procedures shall comply with recognized consensus standards (for example, ASTM standards or EPA's *Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples* (EPA/600/R-03/027)) where available.

Box 32

The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.7.1: ISO/IEC 17025:2017(E) section 7.3.2

Note: for the purposes of this standard, a sampling method and a sampling procedure are equivalent.

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

5.8.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.8.1:*

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

5.8.3 DoD/DOE Requirement

*The following shall be implemented **in addition t**o ISO Clause 5.8.3:*

The laboratory shall have a procedure addressing instances when it receives samples that require non-routine or additional sample preparation steps.

Box 33
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.8.3: ISO/IEC 17025:2017(E) section 7.4.3

5.8.4 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.8.4:*

a) The laboratory shall have SOP(s) in place to address the use of ventilation hoods or suitable containment for opening shipping containers, radiation screening of samples, laboratory notification, and labeling requirements for radioactive samples.

b) The laboratory shall have a procedure and records to verify ventilation hood contamination control on a semiannual basis, such as a smoke test or flow meter measurements. Materials submitted for industrial hygiene or asbestos analysis must be opened in an established manner to prevent worker exposure. Therefore, receiving practices must be developed and implemented for the receipt of beryllium, beryllium oxide, and asbestos **(DOE Only Requirement)**.

c) Shipping containers and packages shall be opened inside a ventilation hood or other designated area that provides adequate ventilation for personnel. All shipping containers from known radiological areas must be surveyed for radiological contamination on all external surfaces. The laboratory must develop and implement administrative policies for the receipt of radiological shipping containers and samples. Radiological surveys of sample shipping containers shall be performed as soon as possible from the time of receipt by the laboratory.

d) Instrumentation and equipment used for monitoring shall be:

   i) maintained and calibrated on an established frequency;

   ii) appropriate for the type(s), levels, and energies of the radiation encountered;

   iii) appropriate for existing environmental conditions; and

iv) routinely tested for operability (10 CFR 835.401(b)).

5.8.6 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 5.8.6 a) through g):*

h) a clear outline of the circumstances under which samples shall be accepted or rejected.

5.8.7.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 5.8.7.1:*

a) 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 procedures may be used.

b) Chemical preservation is matrix specific. The laboratory shall refer to the chain of custody (COC) for the matrix definition. In the case where the matrix is not identified on the COC, the laboratory shall contact the customer prior to proceeding.

c) Chemical preservation must 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 samples). If any of the following conditions exist, chemical preservation must be rechecked in the laboratory:
   i) continued preservation of the sample is in question (e.g., the sample may not be compatible with the preservation); or

   ii) deterioration of the preservation is suspected.

d) The laboratory shall have procedures in place that ensure that the appropriate laboratory personnel are notified when samples are received with a quick turn-around time request, short hold times, or a short amount of hold time remaining.

DOE Only Requirements

e) Laboratories shall develop and maintain procedures for sample receiving and login that minimizes changes in thermal preservation.

f) The laboratory shall document if thermal preservation is not maintained in accordance with the laboratory's procedure during sample receiving and login. The client will be notified in writing if thermal preservation is not maintained. This requirement is for

environmental samples and does not apply to industrial hygiene samples (unless the IH method requires thermal preservation).

g) Subcontract laboratories performing analytical services (i.e., testing, data review, data processing, project management, IT support, etc.) for DOE shall be approved in writing by the appropriate DOE or subcontractor client prior to the commencement of work.

5.8.8 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI 5.8.8:*

Legal/Evidentiary Custody: When the legal chain of custody (COC) protocols are not provided by a state or federal program and legal custody is required to be maintained for a given project, the following protocols shall be incorporated.

a) Basic Requirements:

The legal COC protocol records shall 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, collectively referred to below as “samples”. The COC records shall account for all time periods associated with the samples. For ease of discussion, the above-mentioned items shall be referred to as samples.

i) A sample is in someone’s custody if:

a. it is in one’s actual physical possession;

b. it is in one’s view, after being in one’s physical possession;

c. it has been in one’s physical possession and then locked or sealed so that no one can tamper with it; and/or

d. it is kept in a secure area, restricted to authorized personnel only.

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

DoD/DOE (Guidance)

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

It is recommended that a designated sample custodian be appointed to be responsible for receiving, storing, and distributing samples.

The COC records are not limited to a single form or document; however, organizations should attempt to limit the number of records that would be required to establish COC.

Legal COC should begin at the point established by the federal or state oversight program. This may begin at the point that cleaned sample containers are provided by the laboratory or the time sample collection occurs.

The COC forms should remain with the samples during transport or shipment.

If shipping containers and/or individual sample containers are submitted with sample custody seals and any seals are not intact, the custodian should note this on the COC.

Mailed packages should be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent COC records.

Once received by the laboratory, laboratory personnel are responsible for the care and custody of the sample and must be able to testify that the sample was in their possession and within view or secured in the laboratory at all times. This includes from the moment it was received from the custodian until the time that the analyses are completed until the time that the sample is disposed.

b) Required Information in Custody Records:

Tracking records shall be maintained until final disposition or return of samples to the customer. Tracking records shall include, by direct entry or linkage to other records:

i) time of day and calendar date of each transfer or handling;

ii) signatures of all personnel who physically handled the samples and parent organization and physical address;

iii) all information necessary to produce unequivocal, accurate reports that record the laboratory activities associated with sample receipt, preparation, analysis, and reporting; and

iv) Common Carrier records.

5.8.9 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI 5.8.9 c):*

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

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

iii) 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 ***(DOE Only).***

Note: Further instructions on waste management and disposal are contained in Section 6.4 of this manual.

5.8.9 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI 5.8.9 a) through c):*

d) Access to all evidentiary samples and subsamples shall be controlled and recorded for all samples associated with legal chain of custody.

i) A clean, dry, isolated room, building, and/or refrigerated space that can be securely locked from the outside must be designated as a custody room.

ii) Where possible, distribution of samples to the analyst performing the analysis must be made by the custodian(s).

iii) The laboratory area must be maintained as a secured area, restricted to authorized personnel only.

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

e) Transfer of samples, subsamples, digestates or extracts to another party are subject to all of the requirements for legal COC for all samples associated with legal chain of custody.

5.9 Quality Assurance (QA) of Environmental Testing (ISO/IEC 17025:2005(E) Clause 5.9)

5.9.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.9.1:*

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

Box 34
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.9.1: ISO/IEC 17025:2017(E) sections 7.7.1 and 7.7.2

5.10 Reporting the Results (ISO/IEC 17025:2005(E) Clause 5.10)

*The following shall be implemented **in addition to** ISO Clause 5.10.1:*

The requirements of Appendix A in this standard shall be used for reporting results for DoD/DOE unless client specified reporting requirements are invoked.

Laboratories must have a written procedure for communicating with the customer for the purpose of establishing project-specific data reporting requirements, including 1) conventions for reporting results below the LOQ and 2) specification for the use of data qualifiers. The basis for the use of all data qualifiers must be adequately explained in the test report.

Box 35
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.10.1: ISO/IEC 17025:2017(E) sections 7.8.1.1 and 7.8.1.2

5.10.2 DoD/DOE Requirement

*The following shall be implemented **in addition t**o ISO Clause 5.10.2 b):*

b) in addition, the name of a contact person and their phone number shall also be included in the laboratory information;

Box 36
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.10.2: ISO/IEC 17025:2017(E) section 7.8.2.1.e, 7.8.2.1.l and 7.8.2.2

5.10.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.10.2 a) through k):*

l) any failures identified;

m) for Whole Effluent Toxicity, identification of the statistical method used to provide data;

n) the date of issuance; and

o) for solid samples, a statement of whether the results are based on a dry weight or wet weight basis.

5.10.3.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.10.3.1 a) through e):*

f) information on any non-standard conditions that may have affected the quality of the results, including the use and definitions of data qualifiers, any manual integrations; and

g) where management system requirements are met, a statement of compliance/noncompliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet sample acceptance requirements such as improper container, holding time, or temperature.

Box 37

The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.10.3.1: ISO/IEC 17025:2017(E) section 7.8.3.1.c and 7.8.3.1.e

5.10.3.1.1 DoD/DOE Requirement

In the absence of project-specific requirements, the minimum standard data qualifiers to be used by laboratories are:

U – Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J – The reported result is an estimated value (e.g., matrix interference was observed, or the analyte was detected at a concentration outside the calibration range).

B – Blank contamination. The recorded result is associated with a contaminated blank.

N – Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q – One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

The laboratory may use additional data qualifiers or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., this document, the contract, and project-planning documents).

Note: These data qualifiers are for laboratory use only. Data usability must be determined by the project team.

5.10.3.1.1 DoD Only (Guidance)

Example:

Detection Limit (DL) = 2, Limit of Detection (LOD) = 4, Limit of Quantitation (LOQ) = 20, and Reporting Limit (RL) = 30 for the project, with the precision and bias of the LOQ meeting project RL. All samples are undiluted.

Sample #1: Analytical Result: Non-detect; Reported result: 4U

Sample #2: Analytical Result: 2; Reported result: 2J

Sample #3: Analytical Result: 10; Reported result: 10J

Sample #4: Analytical Result: 20; Reported result: 20

Sample #5: Analytical Result: 30; Reported result: 30

5.10.3.2 DoD/DOE Requirement

Box 38

The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.10.3.2: ISO/IEC 17025:2017(E) sections 7.8.5.d and 7.8.5.f

Box 39

The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.10.3: ISO/IEC 17025:2017(E) section 7.8.6

5.10.5 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.10.5:*

When included, opinions and interpretations shall be contained in the Case Narrative.

Box 40
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.10.5: ISO/IEC 17025:2017(E) section 7.8.7

5.10.6 DoD/DOE Requirement

*The following shall be implemented **in addition to** ISO Clause 5.10.6:*

The laboratory shall make a copy of the subcontractor's report available to the customer when requested by the customer.

Box 41
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.10.6: ISO/IEC 17025:2017(E) section 7.8.2.1.p

5.10.9 DoD/DOE Requirement

Box 42
The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 5.10.9: ISO/IEC 17025:2017(E) section 7.8.8.1

5.10.11 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 5.10.11 a):*

a) The date and time of sample collection, preparation, and analysis are required to be included as part of the laboratory report, regardless of the length of holding time. If the time of the sample collection is not provided, the laboratory must assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

5.10.11 DoD/DOE (Guidance)

<u>A practical approach for determining start time</u>: The start time/date for "Sampling" is the moment that the sample is separated from its natural environment; for "Extraction" it is the

moment that extraction solvent touches the sample; for “Analysis” it is the moment that the extract is introduced into the instrument.

5.10.11 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI 5.10.11 a) through d):*

e) Qualification of numerical results with values outside the calibration range.

6.0 HAZARDOUS AND RADIOACTIVE MATERIALS MANAGEMENT AND HEALTH AND SAFETY PRACTICES (Section 6: DOE Only)

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

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

The laboratory shall comply with all applicable federal and state regulations governing laboratory operations by developing, training and implementing Standard Operating plans/procedures (SOPs). Plans are to be developed as they apply specifically to the laboratory’s facility, staff, and DOE contractual requirements, deliverables, and obligations. The laboratory will be assessed to these plans and their implementation on site. Failure to include in these plans/procedures specific requirements of federal, state, licensure or permit requirements

will be identified as a finding in need of corrective action. The following sections discuss requirements for plans and procedures to support and achieve DOE's objectives:

6.1 Radiological Material Management Plan

6.2 Waste Management Plan

6.3 Chemical Hygiene Plan

6.4 Sample Receiving and Control

6.1 Radioactive Materials Management Plan

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

6.1.1 Radioactive Materials Management

6.1.2 Radioactive Materials License (RML) Requirements

6.1.3 Identification of Radiation Safety Personnel

6.1.4 Radiation Safety Training

6.1.5 Radiation Survey Plan and Equipment

6.1.6 Radioactive Material Receipt and Control

6.1.1 Radioactive Materials Management

For laboratories accepting, receiving, and handling radioactive samples, or potential radioactive samples, the laboratory will be required to develop and implement a radioactive materials management plan or radiation safety plan. This plan, however named, shall comply, identify, and address all applicable site specific related federal and state regulations governing radioactive materials control and radiological protection.

6.1.1.2 The laboratory shall review, at least annually, the radiation protection program content and implementation. Records of audits, reviews, and inspections for the last five years shall be maintained and readily available for review.

6.1.1.3 The laboratory shall develop and implement an effective program of radiological controls and procedures for radioactive material handling, emergency actions, and use of instrumentation.

6.1.1.4 Airborne releases of radioactivity to the environment shall be monitored, evaluated, and controlled. Records shall be maintained for five years and be readily available for review.

6.1.2 Radioactive Materials License Requirements

6.1.2.1 A description is required of how the laboratory will address, implement, and manage the requirements of their site specific radioactive materials license. The laboratory must operate within the parameters of their 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.

6.1.3 Radiation Safety Personnel

6.1.3.1 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 in the event that the RSO is not available.

6.1.3.2 Initial and refresher training of the RSO and the alternate RSO will be identified and completed on an established frequency.

6.1.4 Radiation Safety Training

6.1.4.1 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.

6.1.4.2 All individuals entering any portion of a restricted area shall be instructed in the potential health effects associated with exposure to radioactive materials or radiation, precautions/procedures to minimize exposure, and the purpose and functions of protective devices employed.

6.1.5 Radiation Survey Plan and Equipment

6.1.5.1 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.

6.1.5.2 Radiological survey equipment must be calibrated according to the manufacturer's recommendation or per more frequent procedures as documented by the laboratory. Prior to performing radiological surveys, the radiological survey instrumentation shall be checked for operational performance using a radiological source, a battery check, and a measurement of the nominal background is measured. All performance checks shall be documented and readily available for review. Calibrated backup survey equipment must be available. Calibration is required by an approved supplier or licensed calibration company and must be accredited to ISO 17025:2005 and ANSI/NCSL Z540-1-1994.

6.1.5.3 Instrument and equipment calibration records showing the results of daily calibration checks and daily checks of the operability of radiological monitoring instruments shall be maintained and retained for five years.

6.1.6 Radioactive Material Receipt and Control

Laboratories must ensure:

6.1.6.1 Personnel dealing with radioactive waste management and materials shall be trained in general awareness, security and safety.

6.1.6.2 Active use of a radioactive materials inventory program developed and implemented which is 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 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.

6.1.6.3 Radiological surveys of sample shipping containers shall be conducted as soon as possible from the time of receipt by the laboratory. Shipping containers from radiological areas shall be surveyed on all external surfaces.

6.1.6.4 A radiological control program that addresses analytical radiological control shall be implemented by the laboratory.

6.1.6.5 Low level and high-level samples will be identified, segregated, and processed in order to prevent sample cross-contamination.

6.1.6.6 At sample receiving, samples from potentially radioactive sites shall be screened to ensure that:

- Customer identification of radioactivity (or lack of radioactivity) is correct;
- The sample is properly categorized (per the laboratory's definition of radioactivity) for sample handling in the laboratory;
- Data input is obtained for the radioactive materials license tracking system in the absence of customer supplied information; and
- the shipping container does not exhibit loose contamination or unacceptable external radiation readings.

6.1.6.7 Prior to performing radiological surveys, the radiological survey instrumentation shall be checked for operational performance using a radiological source, a battery check, is performed, and the nominal background is measured. All performance checks shall be documented and readily available for review. Instrument and equipment calibration records showing the results of daily calibration checks and daily checks of the operability of radiological monitoring instruments shall be maintained and retained for five years. Records shall be readily available for review.

6.1.6.8 The laboratory shall ensure that licensed material is secure from unauthorized access or removal.

6.2 Waste Management Plan

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

6.2.1 Waste Management Plan Requirements

6.2.2 Waste Disposal

6.2.3 Waste Storage Area

6.2.4 Toxic Substances Control Act

6.2.1 Waste Management Plan Requirements

The laboratory shall develop and implement a waste management plan identifying how they will comply with all federal and state regulations governing waste management and disposal. The plan shall:

6.2.1.1 Identify all waste streams generated by the laboratory including universal wastes such as batteries, thermostats, etc.

6.2.1.2 Identify the process for management and disposal of the various waste streams.

6.2.1.3 Track the disposition of waste samples by Sample Delivery Group (SDG).

6.2.1.4 Demonstrate compliance through administrative programs to demonstrate compliance for managing effluent discharges as required by regulatory agencies and applicable DOE Orders.

6.2.1.5 Provide training procedures, required frequency, and management of training records in the areas of waste management, shipping, waste handling, and radioactive materials control.

6.2.1.6 Communicate radioactive volumetric and surface release policies.

6.2.1.7 Detail permits and licenses to handle hazardous and radioactive waste.

6.2.1.8 Give policy or direction on how to conduct waste brokering and TSDF evaluation to ensure proper disposition of waste. This includes waste packaging, control and tracking, labeling, classification identification, and preparing/forwarding manifests.

6.2.1.9 Tracking of individual sample container from receipt to final disposition.

6.2.1.10 Address waste minimization and pollution prevention program requirements or plans which include substitution (when permitted), segregation, and recycling.

6.2.1.11 Identify how radioactive and mixed wastes shall be segregated from non-radioactive waste.

6.2.1.12 Develop and implement a radioactive materials inventory program capable of tracking radioactive waste. The radioactive material inventory shall be updated according to the schedule established by the laboratory RML. If no schedule is established by the license, the laboratory shall update the inventory within seven days of receipt of radioactive materials.

6.2.2 Waste Disposal

In managing waste disposition resulting from the receipt, analysis, and shipping of DOE samples, laboratories shall develop and implement policies and practices to identify the following:

6.2.2.1 Waste brokering and TSDF evaluation shall be based upon the results of a site visit to the waste facility or a desktop review that includes information from audits of the facilities conducted by state or federal agencies. The evaluation shall include liability coverage, financial stability, any Notices of Violations (NOVs) from the last three years, relevant permits and licenses to accept the waste, and other relevant information.

6.2.2.2 Waste shipments shall only be transferred to qualified facility/person specifically licensed to receive the waste.

6.2.2.3 TSDF reviews of waste brokering and TSDF evaluations shall be performed every three years, unless there are changes in the facility operations that require the reviews to be conducted on a more frequent basis (e.g., NOVs, change of ownership, notices of fines, and penalties). The laboratory shall develop criteria for the evaluation of waste brokers and TSDFs. Documentation of the evaluations and a list of the facilities that are approved shall be maintained for at least 5 years and records made readily available. Reference to the Environmental Protection Agency (EPA) public domain Enforcement and History Online (ECHO) and “ENVIROFACTS” websites for information on TSDFs. DOECAP TSDF audits can be used in place of onsite visit requirements provided all other requirements not included in these audits are addressed (i.e. financial stability, liability insurance, etc.).

6.2.2.4 Certificate(s) of disposal or destruction shall be obtained for all samples sent to a TSDF, retained for five years, and made readily available.

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

6.2.2.6 Analytical process waste shall be segregated and removed to a designated storage area to minimize the potential for cross-contamination.

6.2.2.7 Laboratory analysis for derived waste characterization shall be repeated at a frequency adequate to account for all known variation in the waste streams.

6.2.2.8 Samples that are consumed during analysis must be included in the sample accountability tracking.

6.2.2.9 The laboratory shall have provisions for the disposition of excess samples. Unless directed otherwise by contract, the laboratory shall receive written permission from the DOE client prior to disposition of any excess samples.

6.2.2.10 Management of excess samples whether they are bulked, special samples, or drain disposed.

6.2.2.11 The laboratory must address how they will manage the requirements for the pre-treatment requirements if disposal includes a Publicly Owned Treatment Works (POTW) or wastewater treatment system. The program must address how the laboratory will be able to demonstrate compliance with these requirements.

6.2.2.12 Records of waste disposal, destruction, and characterization, including analytical test results and process knowledge determinations, shall be kept for at least five years. Records shall be readily available for review.

6.2.2.13 Laboratories shall accumulate no 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 label must indicate the applicable hazard (accepted labels include completed Department of Transportation (DOT) shipping label, National Fire Protection Association (NFPA) label, or RCRA waste characterization code).

6.2.2.14 Radioactive and mixed wastes generated during laboratory sample processing shall be labeled as Radioactive.

6.2.2.15 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 client), and the name of the individual who performed the task.

6.2.3 Waste Storage Areas

6.2.3.1 Laboratories will identify their affiliation and requirements as to their RCRA status as a very small, small, or large quantity generator and will identify the locations, storage limitations, and container sizes for each accumulation and storage area identified. Laboratories must reference the requirements per the applicable CFR.

6.2.3.2 Hazardous waste storage containers shall be:

- Labeled with the words “Hazardous Waste”;
- Labeled with the start date upon which each period of accumulation begins; and

- Clearly marked with the accumulation start date and visible for inspection.

6.2.3.3 Waste storage areas, and containers of waste shall be monitored weekly by an operator or someone knowledgeable in waste operations specific to this facility.

6.2.3.4 The user(s) or operator(s) of the satellite accumulation areas understand container/waste compatibility and have been trained with respect to container selection, waste identification, documentation, and management.

6.2.3.5 Ignitable and reactive waste shall be stored at least 50 feet from the property line.

6.2.3.6 Incompatible waste shall not be stored near other containers or separated by a dike, berm, wall, or other device.

6.2.3.7 The waste storage area shall provide secondary containment of sufficient capacity for the waste expected to be stored in the areas.

6.2.3.8 Accumulation containers shall be:

- In good condition;
- Compatible with the waste; and
- Kept closed at all times when not in immediate use.

6.2.4 Toxic Substances Control Act (TSCA) Material

6.2.4.1 The laboratory shall develop and implement a plan or program stating how laboratory operations will comply with all federal regulations governing TSCA materials control and protection.

6.2.4.2 The laboratory shall segregate all radioactive TSCA materials from all other analytical samples and associated derived wastes.

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

6.2.4.4 The TSCA Polychlorinated biphenyl (PCB) waste shall be stored for less than one year from the date the material was first placed in storage.

6.2.4.5 The TSCA PCB waste containers shall be labeled with the accumulation start date.

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

6.2.4.7 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.).

6.2.4.8 Laboratory-generated TSCA PCB wastes shall be stored in a Temporary Storage Area more than 30 days from the time of generation without being placed in an area that meets one year storage facility requirements.

6.2.4.9 TSCA PCB waste containers and sample storage areas shall be marked with the required TSCA PCB labeling.

6.3 Chemical Hygiene Plan (CHP)

6.3.1 A CHP shall be developed and implemented in the laboratory and readily available to all employees. SOPs relating to safety and health considerations shall be developed and implemented.

6.3.2 The laboratory shall have a written contingency plan and ensure a copy is available at the facility

6.3.3 The following information shall be included in the plan and posted next to the phone in the vicinity of the accumulation area:

- Name and number of the emergency coordinator;
- Location of fire extinguishers and spill control material; and
- Fire department number or a direct alarm.

6.3.4. Required equipment shall be available at the accumulation/storage area. Equipment includes, but is not limited to:

- Internal communication or alarm system;
- Telephone or hand-held two-way radio;
- Portable fire extinguishers/fire control equipment; and
- Spill control equipment, and water at adequate volume and pressure (e.g., 15 minutes of continuous pressure).

6.3.5 An emergency eye wash will be located within the immediate work area, unobstructed, and shall be readily available to all personnel. Location requirements and ease of access, frequency for testing, refilling or restocking as needed, and an emergency shower will be addressed in the plan. All tests and inspections will be clearly marked by a tag on each device. Records will be maintained by the personnel responsible for the implementation of the chemical hygiene plan.

6.3.6 The employer shall provide, mounted, located, identified, and inspected portable fire extinguishers so that they are readily available to all employees without subjecting the employees to possible injury. The location requirements and the frequency for inspection will be established in the Chemical Hygiene Plan, or equivalent plan. All tests and inspections will be clearly marked by a tag on each device. Records will be maintained by the personnel

responsible for the implementation of the chemical hygiene plan. Records will be made readily available for review.

6.3.7 The employer shall have a spill control policy developed. All personnel will be provided documented training on the use and location of each spill kit.

6.3.8 The facility shall be equipped with an alarm system that is capable of being detected and recognized by the employee in case of emergency.

6.3.9 Initial and periodic exposure monitoring for hazardous chemicals shall be conducted. Exposure limits are to be identified or referenced and actions taken should an exceedance occur.

6.3.10 Safety Data Sheets shall be on file for all hazardous chemical substances maintained by the laboratory and readily accessible to all employees.

6.3.11 Measures shall be in place to ensure the performance and maintenance of ventilation hoods and protective equipment. This will include frequency and tested flow rates for each hood. The laboratory shall have a procedure and records to verify contamination control on a semiannual basis such as a smoke test or flow meter measurements. Documentation is required on the process for ventilation hood contamination control.

6.3.12 Laboratory analytical employees shall have readily available records for training associated on:

- Contents of the employer's CHP;
- Physical and health hazards of chemicals in the work area; and
- Methods and observations used to detect the presence or release of a hazardous substance (e.g., monitoring conducted by the employer, continuous monitoring devices, visual appearance or odor of hazardous substances being released, etc).

6.3.13 If respirators are used during sample or waste handling/processing, the laboratory shall have an appropriate written respiratory protection program, including:

- SOPs governing the fit-testing of personnel using respirators, selection and use of respirators; and an annual evaluation to ensure effectiveness.

6.3.14 Chemical hazard labeling on chemical containers shall be in accordance with the laboratory's approved CHP.

6.3.15 A laboratory safety inspection program shall be developed and implemented that includes routine inspections of laboratory areas for health and safety related concerns.

6.3.16 Annual safety requirements will be identified for training and briefings that will be required of all visitors, maintenance personnel, and auditors shall receive a safety orientation prior to entering the laboratory. All training and briefings will be documented and records will be readily available.

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

6.3.18 The laboratory shall develop an emergency response plan to include re-entry procedures once the laboratory is safe to return.

6.3.19 Laboratories shall require clear posting of signs on doors, work stations, and/or safety devices to indicate use of:

- Safety glasses required
- Laboratory coats or protective clothing
- Appropriate footwear;
- Safety showers;
- Eyewash stations;
- Other safety and first aid equipment;
- Exits; and
- Areas where food and beverage consumption and storage are permitted.

6.3.20 Areas containing biological hazards shall be appropriately posted.

6.3.21 All hazardous or toxic chemical cabinets shall be appropriately labeled with the following:

- Identity of the hazardous chemical(s);
- Appropriate hazard warnings; and
- Name and address of the chemical manufacturer, importer, or other responsible party.

6.3.22 All exits shall be properly identified and unobstructed.

6.3.23 Locations and procedures for personal protective equipment (PPE), (to include laboratory coats, safety glasses, shoes, etc.) will be established. These procedures will identify when, what, and where PPE is required and allowed.

6.4 Sample Receiving

6.4.1 The laboratory shall have a documented system for uniquely identifying the items (samples) to be tested.

6.4.2 The laboratory shall have SOPs in place to address the following:

- Containers are opened in a manner to prevent worker exposure
- Checking sample preservation (pH);
- Proper containers;
- Preserving samples when required;

- Documenting and notifying clients of shipping or sample anomalies;
- Checking holding times and notifying laboratory personnel of short holding times;
- Use of fume hoods for opening samples and shipping containers;
- Chain of Custody (COC) is not broken during times when laboratory staff are not present;
- Access to all samples and subsamples is controlled and documented;
- COC forms remain with the samples during transport or shipment; and
- Record 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.

6.4.3 Materials submitted to the laboratory for industrial hygiene or asbestos analyses shall be opened in an established manner to prevent worker exposure and sample receiving practices shall be developed and implemented for the receipt of beryllium, beryllium oxide, and asbestos materials.

6.4.4 The sample receipt personnel shall document anomalies encountered in the sample receiving process.

6.4.5 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 person making the entry.

6.4.6 When the laboratory receives samples, there shall be an internal COC procedure in place. Internal custody shall be maintained until final disposition or return of the sample to the client.

Volume 1, Module 3: Quality Systems for Asbestos Testing

1.0 ASBESTOS TESTING

1.1 Introduction

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.2 Scope

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.3 Terms and Definitions

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.4 Method Selection

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.5 Method Validation

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.6 Demonstration of Capability

1.6.2.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.6.2.2:*

Option *1.6.2.2 e) i)* is not allowed. Only Option *1.6.2.2 e) ii*) shall be performed.

1.7 Technical Requirements

1.7.1.1.1 DoD/DOE (Clarification)

*The following is a **clarification** of TNI 1.7.1.1.1:*

Frequencies shall be increased following non-routine maintenance or unacceptable calibration performance.

1.7.1.1.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.1.1.1 a):*

A logbook or electronic record shall be maintained with the calibrated magnification, the date of calibration, and the analyst's signature or initials recorded.

1.7.1.1.1 DoD/DOE (Clarification)

The following is a **clarification** to *TNI section 1.7.1.1.1 b)*:

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

1.7.1.2.2 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.1.2.2:*

The phase-shift detection limit of the microscope shall be checked daily and after modification.

1.7.1.3.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.1.3.1:*

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.

b) All alignments and function checks shall be documented in the proper log book or electronic record.

Volume 1, Module 4: Quality Systems for Chemical Testing

1.0 CHEMICAL TESTING

1.1 Introduction

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.2 Scope

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.3 Terms and Definitions

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.4 Method Selection

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.5 Method Validation

1.5.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.5.1 a) and b):*

c) The laboratory must evaluate modified reference methods and non-standard methods (including laboratory-developed methods) using quality control (QC) procedures and

acceptance criteria that are consistent with those of similar standard methods or technologies, and the evaluation must include the following:

i) scope;

ii) calibration/calibration verification;

iii) interferences/cross-contamination;

iv) analyte identification;

v) analyte quantitation;

vi) selectivity;

vii) sensitivity;

viii) precision; and

ix) bias.

d) The use of any modified reference method or non-standard method must be approved by DoD/DOE project personnel.

e) Methods must be validated when substantive modifications are made to reference methods (i.e., stoichiometry, technology, mass tuning acceptance criteria, quantitation ions, compressing digestion or extraction timeframes, reducing reagent or solvent volumes, changing solvents, or compressing instrument runtimes).

1.5.1 DoD/DOE (Guidance)

DoD/DOE allows method modifications as described in the November 20, 2007 USEPA Memorandum on method flexibility.

Methods that are 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, are considered non-standard methods.

1.5.2.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.5.2.1 b):*

b) A laboratory shall establish a detection limit (DL) using accepted, published methodologies from recognized entities such as USEPA, USDOE, ASTM, or NIOSH for each suite of analyte-matrix-method, including surrogates. The DL may be established based on historical data. The DL shall be used to determine the LOD for each analyte and matrix as well as for all preparatory and cleanup methods routinely used on samples.

1.5.2.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.5.2.1 f):*

f) Each preparation method listed on the scope of accreditation must have quarterly LOD verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD verifications. If LOD verifications are not performed on all combinations, the laboratory must base the LOD verifications on the worst case basis (preparation method with all applicable cleanup steps).

After each DL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the DL (i.e., 2x DL ≤ LOD Spike ≤ 4x DL). This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

i) The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean Method Blank concentration. This is initially estimated based on a minimum of four Method Blank analyses and later established with a minimum of 20 Method Blank results.

ii) If the LOD verification fails, then the laboratory must repeat the DL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.

iii) The laboratory shall maintain documentation for all DL determinations and LOD verifications.

iv) The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.

g) In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis, prior to sample analysis, in lieu of quarterly verification. All verification data shall be in compliance, reported, and available for review.

1.5.2.2 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.5.2.2 c):*

c) The laboratory procedure for establishing the LOQ must empirically demonstrate precision and bias at the LOQ for each suite of analyte-matrix-method, including surrogates. The LOQ and associated precision and bias must meet client requirements and must be reported. If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. For DoD/DOE projects, the LOQ must be set within the calibration range, including the lowest calibration level.

NOTE: Precision and bias at the limit of quantitation (LOQ) can be demonstrated by analyzing multiple LOQ-verification samples over multiple days. A minimum of 4 samples should be analyzed to establish the initial precision and bias. If specific methods recommend a greater number of LOQ-verification replicates, then more replicates should be initially analyzed (e.g., SW846 6010D and 6020B recommend 7 replicates). If multiple instruments are used for analysis, then LOQ verifications should be performed on each instrument and all of the results should be used for calculation of precision and bias. The precision and bias should be updated at least annually using any additional LOQ verification sample results. LOQ verification samples should meet laboratory-established or Method acceptance criteria.

Precision should be determined by calculating the standard deviation for each set of analyte-matrix-method results. The standard deviation (precision) should be reported as a percent of the spiked value. Bias should be determined by calculating the difference of the average result from the spiked value. The bias should also be reported as a percentage of the spiked value. Bias will be negative, positive, or zero.

When reporting precision and bias, 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. This table (or a similar presentation of precision and bias) must

be provided to the client during project planning, and on request. Precision and bias information should also be provided in laboratory data packages.

1.5.2.2 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.5.2.2 e):*

e) For DoD/DOE, at a minimum, the LOQ shall be verified quarterly. However, not all possible combinations of preparation and cleanup techniques are required to have LOQ verifications. If LOQ verifications are not performed on all combinations, the laboratory must base the LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps). In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis in lieu of quarterly verification, prior to sample analysis.

1.6 Demonstration of Capability (DOC)

1.6.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.6.2:*

a) The laboratory shall have a documented procedure for performing the initial demonstration of capability (IDOC) for methods used.

b) Changes in any condition that could potentially affect the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, method revision, or other components of the sample analytical system) must result in a new initial DOC.

1.7 Technical Requirements

1.7.1.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.1.1 d):*

d) all initial instrument calibrations shall be verified with a standard obtained from a second manufacturer prior to analyzing any samples.

The use of a standard 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 shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

1.7.1.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.1.1 g):*

g) the LOQ and the highest calibration standard of a multi-level calibration curve establish the calibration range. For metals analysis with a single-point calibration, the LOQ and the calibration standard establish the calibration range, which must lie within the linear dynamic range.

When sample responses exceed the calibration range, the laboratory shall dilute and reanalyze the sample (when sufficient sample volume and holding time permit) to bring results within the calibration range. For metals 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 within the same calibration). Results outside the calibration range shall be reported as estimated values and qualified using appropriate data qualifiers that are explained in the Case Narrative.

1.7.1.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.1.1 j):*

j) the initial calibration range shall consist of a minimum of five calibration points for organic analytes and three calibration points for inorganic analytes and Industrial Hygiene samples (except metals by ICP-AES or ICP-MS with a single-point calibration or otherwise stated in the method). All reported analytes and surrogates (if applicable) shall be included in the initial calibration. Reported results for all analytes and surrogates shall be quantified using a multipoint calibration curve (except as noted above). Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

1.7.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.2 c) i) through iii):*

iv) the concentration of the continuing calibration verification (CCV) standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

1.7.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.2 d):*

d) All CCVs analyzed must be evaluated and reported. If a bracketing CCV fails, reanalysis or corrective actions must be taken.

1.7.2 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.2 e):*

e) If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for analytes to be reported without reanalysis of samples, while ignoring spurious failures. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) require that all samples since the last acceptable CCV be reanalyzed.

i) Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.

ii) If either of these two CCVs fail, the associated samples cannot be reported and must be reanalyzed.

iii) If the laboratory cannot immediately analyze two CCVs, then perform corrective action(s) and repeat the CCV and all associated samples since the last successful CCV.

iv) Recalibration must occur if the above scenarios fail. All affected samples since the last acceptable CCV must be reanalyzed.

v) Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

1.7.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.3:*

Method specific Quality Control (QC) requirements are located in Appendix B of this standard. All method QC parameters and samples shall follow Appendix B requirements, as appropriate. Appendix B requirements are considered the minimum technology based requirements for DoD accreditation or DOE acceptance regardless of method version.

1.7.3.2.3 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.3.2.3 b):*

b) all reported analytes must 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.

   The concentration of the spiked compounds shall be at or below the midpoint of the calibration if project specific concentrations are not specified.

c) a laboratory shall establish LCS in-house limits that:

   i) are statistically-derived based on in-house historical data, using scientifically valid and documented procedures;

   ii) meet the limits specified by the project or as stated in the method if available;

   iii) are updated on at least an annual basis or as stated in the method, whichever is more frequent, and re-established after major changes in the analytical process (e.g., new instrumentation);

   iv) are based on at least 30 data points generated under the same analytical process;

   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); and

vi) control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.

vii) The LCS shall consist of a quality system matrix that is similar to the associated samples.

d) 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 on an on-going basis (at least quarterly) for shifts in mean recovery, changes in standard deviation, and development of trends. Laboratories may choose representative compounds for control charts for the purpose of trend analysis.

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

f) a laboratory must use its in-house statistically established LCS control limits for the purpose of trend analysis and may use in-house control limits as a component in estimating measurement uncertainty.

g) in the absence of client specified LCS acceptance criteria, the LCS control limits outlined in the DoD/DOE QSM Appendix C tables shall be used when reporting data for DoD/DOE projects. Laboratories must develop processes or procedures to incorporate these limits.

h) the LCS limits specified in the DoD/DOE QSM Appendix C tables shall be used for batch control unless project specific criteria exist. Sporadic marginal exceedances are allowed for those analytes outside the 3 standard deviation control limits but still within 4 standard deviations. Marginal exceedances are not allowed for those analytes determined by a project to be target analytes (i.e. “risk drivers”) without project specific approval.

i) for analytes that are not listed in the DoD/DOE QSM Appendix C control limits tables, a laboratory shall use their in-house control limits for batch control and data reporting.

j) for DoD ELAP accreditation, a laboratory must develop in-house control limits for all analytes on their scope of accreditation. In-house control limits shall be used for trend analysis, and batch control for those analytes not listed in the DoD/DOE QSM Appendix C LCS tables **(DoD Only Requirement)**.

1.7.3.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.3.3:*

The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD/DOE Appendix C LCS limits or project limits, if specified. If the specific analyte(s) are not available in the DoD/DOE QSM Appendix C tables, the laboratory shall use their LCS in-house limits as a means of evaluating MS/MSDs.

1.7.3.3.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.3.3.1 b):*

b) Each preparation batch of samples must contain an associated MS and MSD (or matrix duplicate (MD)) using the same matrix collected for the specific project. The requirements for MS/MSD are not applicable to all methods (e.g., certain radiochemical samples, air-testing samples, classic chemistry, and industrial hygiene samples). If adequate sample material is not available, then the lack of MS/MSDs (or MD) shall be noted in the Case Narrative, and a LCS Duplicate (LCSD) shall be used to determine precision. Additional MS/MSDs may be required on a project-specific basis.

1.7.3.3.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.3.3.1 c):*

c) The MS and MSD must be spiked with all reported analytes (with the exception of Aroclor analysis, which is spiked per the method).

1.7.3.3.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.3.3.3 a) through c):*

d) Surrogate spike results shall be compared with DoD/DOE QSM Appendix C LCS limits or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria.

Note: DoD/DOE QSM Appendix C tables do not include all possible surrogates for all methods.

1.7.3.5 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.3.5 a) through c):*

d) The quality (e.g., purity) specifications for all standards and reagents (including water) shall be documented or referenced in SOPs.

1.7.3.6 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.3.6:*

a) Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation is necessary for all results greater than the DL and when the composition of samples is not well characterized. Confirmation techniques include further analysis using a second column with dissimilar stationary phase, GC/MS (full scan or SIM) or HPLC/MS (if concentration permits), GC or HPLC with two different types of detectors, or by other recognized confirmation techniques. HPLC UV-Diode Array detectors are not considered confirmation for a UV detector.

b) When reporting data for methods that require analyte confirmation using a secondary column or detector, project-specific reporting requirements shall be followed. If project-specific requirements have not been specified, follow the reporting requirements in the method. If the method does not include reporting requirements, then report the results from the primary column or detector, unless there is a scientifically valid and documented reason for not doing so and is concurred with by the client.

c) The DoD/DOE specific client shall be notified of any results that are unconfirmed (e.g., confirmation was not performed or confirmation was obscured by interference). Unconfirmed results shall also be identified in the test report, using appropriate data qualifier flags, and explained in the Case Narrative. Analyte presence is indicated only if both original and confirmation signals are positive or if confirmation signal cannot be discerned from interference.

1.7.4.1 DoD/DOE Requirement

*The following shall be implemented **in lieu of** TNI section 1.7.4.1 a):*

a) the Method Blank shall be considered to be contaminated if:

   i) the concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ or is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater;

ii) the concentration of any common laboratory contaminant in the blank exceeds the LOQ;

b) if a Method Blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

1.7.4.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.4.2 b):*

c) Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval.

d) DoD/DOE considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

1.7.4.2 DoD/DOE (Guidance)

Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

Volume 1, Module 5: Quality Systems for Microbiological Testing

No additions or clarifications were made to Volume 1, Module 5. **The TNI Standard shall be followed.**

VOLUME 1, MODULE 6: Quality Systems for Radiochemical Testing

1.0 RADIOCHEMICAL TESTING

1.1 Introduction

No additions or clarifications were made to this section. **The TNI Standard shall be followed.**

1.2 Scope

No additions or clarifications were made to this section. **The TNI Standard shall be followed.**

1.3 Terms and Definitions (DoD/DOE Clarification)

*The following is a **clarification** of TNI section 1.3:*

This DoD/DOE module references the radiochemical terms, definitions, and requirements contained in the 2009 TNI Standard *Module 6 Quality Systems for Radiochemical Testing*. However, it does not preclude the use of other terms, definitions, and requirements from the consensus document *Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual*, July 2004.

The following equations are defined in section 18 of MARLAP and are used for QC evaluations identified in this module of the DoD/DOE QSM.

Section 1.7.2.1 c) ii)

$$Z_{\text{Blank}} = \frac{x}{u_c(x)}$$

where x denotes the measured blank activity and $u_c(x)$ denotes its combined standard uncertainty.

Section 1.7.2.1 b) xi)

$$Z_{\text{Dup}} = \frac{x_1 - x_2}{\sqrt{u_c^2(x_1) + u_c^2(x_2)}}$$

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.

Section 1.7.2.2 l)

$$Z_{\mathrm{LCS}} = \frac{x - d}{\sqrt{u_c^2(x) + u_c^2(d)}}$$

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.

Section 1.7.2.3 a) xi)

$$Z_{\mathrm{MS}} = \frac{x - x_0 - d}{\sqrt{u_c^2(x) + u_c^2(x_0) + u_c^2(d)}}$$

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.

1.4 Method Selection

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.5 Method Validation

1.5.2.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.5.2.1 a) through d):*

e) SOPs that incorporate equations to calculate the decision level and the minimum detectable concentration (or activity) must be documented and consistent with the mandated method or regulation.

1.5.2.1.1 DoD/DOE Requirement

a) Minimum Detectable Activity (MDA):

The MDA is the smallest amount of an analyte in a sample that will be detected with a probability β of non-detection (Type II error), while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). Confidence levels may be dictated by the project. For the purposes of this module and the equations below, the α and β probabilities are assumed to be 0.05. MARLAP utilizes the Minimum Detectable Concentration (MDC) term instead of MDA.

b) 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 detection reporting limit (RL). 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 RLs. If RLs are not achieved, then the cause shall be addressed comprehensively in the Case Narrative.

c) 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 the MARLAP Manual Chapter 20. The following general equations derived from the work of L. A. Currie can be used to calculate the MDA.

i) With a Blank Population:

$$MDA = \frac{3.29 * s_b}{KT_S} + \frac{3}{KT_S}$$

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 Appendix B population is in net blank counts in count time T_S

Use of blank populations for calculation of MDAs requires the selection of an implementation method, which includes but is not limited to:

a. Identification of blanks to be used in the population;

b. The number of blanks to use in the population;

c. How the blank population changes; and

d. Limitations on the deletion of blanks.

The method of implementation shall not introduce any statistical bias.

The appropriate blank subtraction shall be the mean blank value of the blank population.

The implementation of blank populations for calculation of MDAs shall be described in detail in a SOP.

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 3 is more appropriate (*Multi Agency Radiation Survey & 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 DoD/DOE client and document the use of 2.71 in the Case Narrative or in procedures available to the client.

ii) *Without a Blank Population:*

MDA for samples without a blank population can be determined if based on appropriate Currie or MARLAP calculations, such as:

$$MDA = \frac{3.29 * \sqrt{\frac{b}{T_S} + \frac{b}{T_B}}}{K} + \frac{3}{K * T_S}$$

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

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.

General:

- The above equation for MDA has the units of dpm/sample. Any other units will require appropriate conversion.
- Site specific requirements may be provided for other MDA formulations.
- MDAs for samples without a blank population can be determined if based on appropriate L. A. Currie or MARLAP calculations.

d) MDA Optimization:

The laboratory shall optimize analysis parameters in order to achieve analyte MDAs less than or equal to the required detection threshold. Samples with elevated activities shall be handled according to the following requirements:

i) The appropriate aliquot size shall be determined based on the activity level in the sample. The aliquant shall be large enough to generate data, which meet the following criteria:

a. the measurement uncertainty shall not be greater than 10% (1 sigma) of the sample activity;

b. the MDA for the analysis shall be a maximum of 10% of the sample activity.

e) Sample-specific MDAs are routinely calculated and reported as standard practice. If MDAs are reported as a nominal detection capability of the measurement process, that shall be clearly stated in the data package.

f) The definition of the MDA presupposes that an appropriate detection threshold (i.e., the decision level) has already been defined. The α probabilities assumed for the decision level shall also be used for the calculation of the MDA.

1.5.2.1.2 DoD/DOE Requirement

a) Decision Level:

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

b) Decision Level Factors and Conditions:

Decision levels are determined *a posteriori* based on sample-specific sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency.

c) 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. The following general equation below can be used to calculate the decision level.

The decision level can either be based on the Combined Standard Uncertainty (CSU) of the blank (preparation or method) or the standard deviation determined from a set of appropriate blanks.

i) With 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 blank 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.

Decision levels are used as the default detection threshold. Alternatively, the client may use/specify detection thresholds that meet project/site-specific requirements.

ii) *<u>Without a Blank Population:</u>*

Decision levelss for samples without a blank population can be determined if based on appropriate L. A. Currie or MARLAP calculations using a CSU. (Refer to Section 1.4.5.4 for the definition of CSU).

1.5.4 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.5.4:*

Measurement Uncertainties (for Radiochemistry Analysis):

Each result shall be reported with the associated measurement uncertainty as a combined standard uncertainty. The SOP for determining the measurement uncertainty must be consistent with mandated method and regulation.

Combined Standard Uncertainty (CSU):

All measurement uncertainties shall be propagated and reported with each result. The formula for calculating the CSU of a result shall be documented in the appropriate SOP. The CSU shall include both systematic and random error. CSU is always 1 sigma. Results should be reported at the 95% confidence level, which is 1.96-sigma (often abbreviated as 2-sigma).

The uncertainty of a count may be estimated as the square root of counts except when there are zero (0) counts. In the case of zero (0) counts, the uncertainty of the count is assumed to be the square root of one count. For counting methodologies where very low counts are possible, the MARLAP 19.57 equation may be used with acceptance by the client.

Systematic Error shall include but is not necessarily limited to:

a) the errors from all measurement devices, such as, but not limited to, pipettes and balances;

b) the uncertainty of known values of tracer solutions, calibration uncertainties, etc.

Random Error shall include, but is not necessarily limited to, the total random counting error associated with each sample and appropriately propagated when more than one variable is used to determine the result.

1.6 Demonstration of Capability (DOC)

No additions or clarifications were made to this section. **TNI and ISO/IEC 17025:2005(E) standards shall be followed.**

1.7 Technical Requirements

1.7.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.1 a) i) through vii):*

viii) Detection efficiency shall be determined with sources that are traceable to NIST or accepted international standards, or with sources prepared from NIST/international traceable standards, when available. When sources used for determinations for detection efficiency are not prepared from NIST/international traceable standards, they shall be "working reference materials" defined as follows: a reference material with one or more properties sufficiently well established to be used for calibration or assessment of a measurement method. Working reference materials may be prepared by the laboratory for their own use. (See ASTM C 1128).

ix) For alpha spectrometry, a material balance check shall be done on each source to clearly demonstrate accountability of all activity by mass balance. The material balance check shall be done on the fraction remaining from the neodymium fluoride precipitation or the electro-deposition plus all rinses from an adequate cleaning of any vessel used in the process. The estimated error in preparing the source shall be propagated into the error of the efficiency determination.

1.7.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.1 b):*

When results for instrument performance checks exceed predetermined acceptance criteria (i.e., limit of a statistical or tolerance chart or other QC parameters), the cause of the problem shall be investigated.

If a performance check fails, the laboratory can immediately analyze two additional consecutive performance checks (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed performance check and the two additional performance checks). This approach allows for analytes to be reported without reanalysis of samples, while ignoring spurious failures. Both of these performance checks must meet acceptance criteria in order for the samples to be reported without reanalysis.

If either of these two performance checks fail, or if the laboratory cannot/does not immediately analyze two performance checks, perform corrective action(s) and repeat the performance check and all associated samples since the last successful performance check. Any corrective actions that change the dynamics of the system requires that all samples since the last acceptable performance check be reanalyzed.

If the problem is not corrected and indicates an intrinsic change in instrument response, then the instrument shall be recalibrated. All affected samples since the last acceptable performance check must be reanalyzed.

*The following shall be implemented **in addition to** TNI section 1.7.1 b) i):*

i) For systems using sample changers and/or long count times that run more than a day, the energy calibration shall be checked before each analytical batch.

*The following shall be implemented **in addition to** TNI section 1.7.1 b) ii):*

ii) Detector response (counting efficiency) determinations shall be performed when the check source count is outside the acceptable limits of the control chart (reference ANSI N42.23, Annex A5).

*The following shall be implemented **in addition to** TNI section 1.7.1 b) iv):*

iv) For radon scintillation detectors, efficiency shall be verified at least monthly, when the system is in use.

1.7.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.1 c) i) through iv):*

v) Background Subtraction Count (BSC) measurements shall be conducted after calibration and monthly thereafter and monitored for trends to ensure that a laboratory maintains its capability to meet required project objectives.

vi) Successive long background measurements may be evaluated in lieu of shorter background check measurement.

vii) Low levels of contamination not detected in a shorter background counting time may bias the results of sample analyses. The duration of the background check measurement shall be of sufficient duration (i.e., at least as long as the sample count time) to quantify contamination that may impact routine sample measurements.

viii) The background check frequency may be extended to accommodate long sample count times.

ix) 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. An Instrument Contamination Check (ICC) for alpha spectroscopy can be a shorter measurement that can be performed on a weekly basis, in which case reporting sample results is not contingent on bracketing ICC checks.

x) A background check shall also be collected before and after any counting chamber changes are made (i.e., cleaning, liner replacement, or instrument modification).

1.7.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.2:*

QC Sample Preparation:

All samples and QC samples in each prep batch shall be prepared concurrently and in the same manner.

QC Sample Counting:

All QC samples shall be counted and analyzed in the same manner as the samples in the prep batch, in the same time frame, and using the same instrument calibration parameters, instrument analysis algorithms, etc.

Method specific Quality Control requirements are located in Appendix B of this standard. All method QC samples shall follow Appendix B requirements, as appropriate.

Note: The "same time frame" implies that where multiple detectors are used and are sufficient to count the entire batch, counting shall be at the same time, with the same count time duration. If the number of detectors is not sufficient to count the entire batch at the same time, then samples shall be counted consecutively on the available detector(s).

Note: The "same instrument calibration parameters, instrument analysis algorithms, etc." implies that these parameters for a given instrument shall not be changed for the samples in that batch. It is understood that for multiple detectors, the parameters may not be identical.

1.7.2.1 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.2.1 a) through c):*

d) 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.

e) The batch blank matrix shall be the same as the samples, as can be reasonably achieved, and shall be documented in the Case Narrative.

f) Blank Acceptance Criteria:

i) A Method Blank shall be one per preparatory batch. (MARLAP 18.4.1)

ii) The blank acceptance criteria shall be: $|Z_{Blank}| \leq 3$ (MARLAP 18.4.1) or a laboratory shall use Method Blank in-house control limits of ± 3σ of the mean.

iii) The Batch Blank MDA shall be less than the Reporting Limit.

If these criteria are not met, corrective actions shall be taken (e.g., recount, interferent cleanup, as appropriate) unless all sample results are greater than five times the blank activity. If the criteria are still not met, then the samples shall be reanalyzed.

g) The following batch blank matrices shall be used for all radiochemistry analyses:

i) Distilled or deionized water, analyte free, as demonstrated in Method Blanks;

ii) Characterized solid material representative of the sample matrix; and

iii) Filters, physically and chemically identical filter media, analyte free (if supplied to the laboratory by customer).

1.7.2.2 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.2.2 a) through i):*

j) The LCS shall be counted for a sufficient time to quantify the activity level of the LCS.

k) The LCS matrix shall be the same as the samples, or as close as can be reasonably achieved, and the matrix shall be documented in the Case Narrative.

l) LCS Acceptance Criteria:

$|Z_{LCS}| \leq 3$ (MARLAP 18.4.3) or use in-house control limits of LCS ± 3σ of the mean. In-house control limits may not fall more than 25% from the known LCS value.

m) LCS Selection and Level:

The LCS shall contain at least one analyte reported for samples by that analytical method (separation chemistry and decay mechanism) and **should** be at least five times, but not greater than 20 times, the RL with the following exceptions:

i) Some programs may require, following TNI, at least 10 times the MDA and at a level compatible with routine samples.

ii) For RLs of low activity, the analyte shall be at a level where the random counting error does not exceed 10% in the counting time required to attain the RL.

iii) Analytes for gamma spectroscopy need not be the same as the sample analyte but should fall in the approximate energy region of the spectrum (i.e., low, mid-range, and high energy) of the reported analytes.

iv) For gross alpha and/or gross beta analysis, the analytes in the LCS shall be the same analytes used for the calibration curve.

v) If a laboratory standard containing the reported analyte is not available, an LCS analyte having similar separation chemistry, energy and decay mechanisms shall be used unless otherwise agreed to by the client.

n) LCS shall be traceable to the NIST or accepted international standard, or shall be a working reference material as described in 1.7.1 a) viii) and may be used repeatedly for different analytical batches as long as it is appropriate for the matrix and geometry of the batch.

1.7.2.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.2.3 a) i) through vii):*

a) Matrix Spike:

viii) Matrix spikes shall be added as early in the sample preparation steps as practicable.

ix) Matrix spikes are not required for radiochemical analyses if an isotopic tracer or chemical carrier is used in the analysis to determine chemical recovery (yield) for the chemical separation and sample mounting procedures. Matrix spikes are not required for gross alpha, gross beta, gamma, or non-aqueous tritium analysis.

x) Matrix spikes shall be run on a separate sample aliquot using the same analyte as that being analyzed whenever possible.

xi) Acceptance Criteria:

Matrix spike recoveries shall be evaluated using the following criteria:

If the activity of the sample is less than 5 times the spiking level, matrix spike recoveries shall be within the control limits of 60 - 140%, or as specified by the client.

If the activity of the sample is greater than 5 times the spiking level, $|Z_{MS}| \leq 3$ shall be used (MARLAP 18.4.3).

xii) Matrix Spike Selection and Level:

The matrix spike shall be added at a concentration of at least five, but not greater than 20 times the RL. For samples having known significant activity of the targeted radionuclides, more than 20 times the RL may be added to minimize the effect of the sample activity on determination of spike recoveries. Some programs may require, following the TNI standard, at least 5 times the MDA.

xiii) Counting:

The matrix spike shall be counted for a sufficient time to quantify the activity level of the spiking.

Where the original (unspiked) sample contains significantly elevated activity, the matrix spike shall be counted for a duration equal to that of the associated original sample.

1.7.2.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.2.3 b) i) through iv):*

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

v) The purpose of the Duplicate sample analysis is to assess laboratory precision by providing information on the laboratory's reproducibility and the homogeneity of the sample.

vi) The Duplicate activity shall not be averaged with the corresponding sample activity when reporting results.

vii) Samples identified as Field Blanks shall not be used for Duplicate sample analysis.

viii) At least one Duplicate sample shall be prepared and analyzed with every Analytical Batch of samples.

ix) The Duplicate shall be counted for the same duration to meet the required detection limit.

x) When the sample does not contain significantly elevated activity, QC samples shall be counted for a duration equal to that of the associated original sample.

xi) Evaluation Criteria:

Duplicates are evaluated using three possible criteria:

a. $|Z_{Dup}| \leq 3$ (MARLAP 18.4.1) if using MARLAP; or

b. the Duplicate Error Ratio (DER) between the sample and the Duplicate is < 3; or

c. the relative percent difference (RPD) is < 25%.

When the MARLAP, DER or the RPD criteria pass, then the Duplicate is acceptable.

Duplicates that do not meet the above requirements due to difficulty in sub-sampling shall be described in the Case Narrative.

1.7.2.3 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.2.3 c):*

c) Tracer:

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

i) Requirements for indirect yield measurements:

(e.g., radiometric results are corrected for chemical yield using 'indirect' yield measurement techniques such as a second radiometric measurement of added tracer.)

The chemical yield for each sample determined using an indirect yield measurement method shall fall within the range 30% - 110% or as specified by the client. The

technique used for the indirect yield measurement should be sufficient to maintain relative uncertainties associated with the yield correction below 10% at the 2-sigma level.

ii) Sample results with yields below 30% are quantitative and considered acceptable if:

a. The relative uncertainty associated with the yield correction is less than 10% (2-sigma);

b. Spectral resolution requirements are met and there are no indications of spectral interferences; and

c. Detection limit requirements are met.

iii) Reporting yield measurement uncertainties:

The uncertainty associated with chemical yield corrections shall be incorporated into the CSU of the associated sample results.

iv) Tracer yield requirements for isotope direct yield methods: (usually alpha spectroscopy).

The chemical yield for isotope dilution methods shall fall within the range 30% - 110% or as specified by the client. Tracer activity and sample count duration shall be adequate to achieve relative uncertainties for the tracer measurement of less than 10% at the 2-sigma level.

1.7.2.3 DoD/DOE (Requirement)

*The following shall be implemented **in addition to** TNI section 1.7.2.3 d):*

d) Carrier:

Carriers chemically mimic but do not interfere with the target analyte through radiochemical separations. Carriers are typically nonradioactive (e.g., natural strontium). They are added to samples to determine the overall chemical yield for the analytical preparation steps. When carriers are used, each sample (including any batch associated QC samples) shall also be spiked with the same materials and individual sample yields determined. The carrier shall be added to the sample at the very beginning of the sample preparation. For solid samples, the carrier shall be added after grinding, sieving, etc., but prior to any muffling or dissolution of the sample.

i) Requirements for indirect yield measurements:

(e.g., radiometric results are corrected for chemical yield using 'indirect' yield measurement techniques such as gravimetric measurement of added carriers.)

The chemical yield for each sample determined using an indirect yield measurement method shall fall within the range 30% - 110% or as specified by the client. The technique used for the indirect yield measurement should be sufficient to maintain relative uncertainties associated with the yield correction below 10% at the 2-sigma level.

ii) Sample results with yields below 30% are quantitative and considered acceptable if:

a. The relative uncertainty associated with the yield correction is less than 10% (2-sigma);

b. Resolution requirements are met, and there are no indications of spectral interferences; and

c. Detection limit requirements are met.

iii) Reporting yield measurement uncertainties:

The uncertainty associated with chemical yield corrections shall be incorporated into the CSU of the associated sample results.

1.7.2.4 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.2.4 a) through c):*

d) Negative Numbers:

All negative activities shall be reported as such. If the sum of the activity and the measurement uncertainty at ± 3 sigma is a negative number, the cause shall be investigated and evaluated to determine if it is systematic or random error. If the cause is systematic, it shall be corrected. If the cause is random, it shall be documented in the Case Narrative. Recurrent problems with significant negative results suggest that the background subtraction and/or blank subtraction, if applicable, are in error or that the estimate of error is low. Investigation of such problems and documentation of the resolution is required and shall be discussed in the Case Narrative.

References:

- DOE / EH - 0173T "Environmental Regulatory Guide for Radiological Effluent Monitoring and Environmental Surveillance," January 1991.

- "Multi-Agency Radiological Laboratory Analytical Protocols Manual NRC NUREG-1576," EPA 402-B-04-001C, NTIS PB2004-105421 July 2004 Section 18.6.5.

1.7.2.5 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI section 1.7.2.5 a) through c):*

d) Water purity shall be at least distilled or deionized water.

e) Standards shall be verified prior to initial use.

   i) Preparations of standards solutions used for a period of time exceeding one year shall be verified annually, at a minimum, and documented in a logbook.

   ii) At least three verification measurements of a standard shall be used to determine the mean value and standard deviation of the verification results.

   iii) The mean value shall be within 5% of the decay corrected certified value.

   iv) The 2-sigma value used for the 95% confidence interval of the mean shall not exceed 10% of the mean value of the three verification measurements.

   v) If all criteria are met, the certified value shall be used.

f) Corrections for radioactive decay and/or ingrowth of progeny shall be performed for radionuclide standards.

1.7.2.7 DoD/DOE Requirement

*The following shall be implemented **in addition to** TNI sections 1.7.2.7 a) through c):*

d) Background contamination monitoring samples shall be analyzed at a sufficiently low level of detection to confirm that no impacts to client samples have occurred due to cross-contamination. Samples shall be segregated by activity levels in sample receipt, processing areas, and storage areas.

1.8 Method Specific Directions DoD/DOE Requirements

*The following shall be implemented **in addition to** TNI sections 1.1 through 1.7:*

1.8.1 Isotopic Determinations by Alpha Spectrometry

a) Tracer:

Shall be used for isotope specific analysis by alpha spectrometry. Initial sample preparation shall include treatment to ensure that tracer and analyte will undergo similar reactions during processing. All tracers shall be of the same element or of an element with the same chemistry for the separations. All tracers used for alpha spectrometry 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 site accepted prior to use.

b) Background Correction:

The gross counts in each target analyte and tracer ROI shall be corrected for the particular detector's background contribution in those same ROIs.

c) Blank Correction:

Shall not be performed, except where required by client and fully documented in the Case Narrative.

d) Conditions Requiring Reanalysis:

i) Sample- and Analyte-Specific Conditions:

Any one of the following are additional conditions that require reanalysis for a particular sample and analyte if sufficient sample quantity remains:

a. If the tracer recovery for the sample does not fall within 30% - 110%, reanalysis is required, beginning with preparation (but see 1.7.2.3 c) i) through iii)).

b. If the FWHM for the tracer peak exceeds 100 keV and/or the peak energy does not fall within ± 50 keV of the known peak energy, reanalysis is required.

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 resolution of tracer and analyte peaks is accomplished.

d. If the sample analyte spectrum contains significant interferences with the analyte and/or tracer ROIs, reanalysis is required.

ii) Analytical Batch Conditions:

If the tracer chemical recovery for the Batch Blank does not fall within 30% - 110%, reanalysis of the entire Analytical Batch, beginning with the preparation, is required if sufficient sample is available.

e) Instrument Calibration:

Calibration of each alpha spectrometry detector used to produce data shall include channel vs. energy calibration, detector response, efficiency determination and background determination for each ROI.

f) Alpha Spectrum.

Alpha spectrum regions of interest shall be selected with consistency from analyte to analyte.

g) Energy Calibration:

i) The energy calibration for each detector shall be performed. A curve shall be fit for Energy (Y-axis) versus Channel (X-axis) and the equation with the slope and Y-intercept for the fit shall be documented.

ii) The slope of the equation shall be < 15 keV/channel.

iii) The energy calibration shall be performed using at least three isotopes within the energy range of 3 to 6 MeV.

iv) The final peak energy positions of all observed isotopes shall be within ± 40 keV of the expected peak energy.

h) Background Requirements:

i) The background total counts (or counts per unit time) for each target analyte and tracer isotope ROI shall be analyzed on each detector and documented.

ii) The background for each ROI shall be sufficiently low to ensure that required detection limits are met.

iii) The limits of acceptability for each background ROI shall be documented. These shall be set such that RLs can be obtained for backgrounds at the limit of acceptability.

iv) Background count times shall be equal to or longer than sample count times.

i) Detector Response Determination Requirements:

Typically, when a tracer is used for the analysis, detector response (detector efficiency) is not used directly in calculation of final results. Detector response is used to calculate estimated yields for evaluation of method performance.

i) The response (efficiency) counts for the ROI shall be background corrected using the same ROI for the background unless the background is less than 0.5% of the total counts in the ROI.

ii) The response (efficiency) shall be determined on at least 3,000 net counts in the ROI (after background correction).

iii) Check source counts to verify detector response (efficiency) shall be determined on at least 2,000 counts.

iv) The detector response and detector response error shall be documented.

v) The detector response check as determined by the check source and/or pulsar count and the associated error and limits of acceptability for the check source result shall be documented.

j) Spectrum Assessment:

i) ROIs shall be clearly indicated either graphically or in tabular form on alpha printouts. Spectra with ROIs shall be saved and made available for review upon request.

ii) The full width at half maximum (FWHM) resolution for each sample and QC sample tracer peak shall be ≤ 100 keV.

iii) The tracer peak energy for each sample and QC sample shall be within ± 50 keV of the expected energy.

iv) 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.

v) Any manual integration or adjustment of ROIs shall be fully discussed in the Case Narrative.

1.8.2 Radon Scintillation (Lucas Cell)

a) SOPs for sample analyses by Lucas Cell shall incorporate and adhere to ASTM D3454 (current version), *Standard Test Method for Radium-226 in Water*. Performance shall be in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. Reference is to the current version of the method. When references are updated, an implementation schedule shall be determined by the laboratory.

b) The operating voltage plateau for the detector shall not exceed a slope of 2%/100V.

c) A new Lucas Cell shall be calibrated every month for the first six months of use and then annually after the initial six months of use.

d) Background measurements for quantitation in each cell shall be carried out prior to each sample measurement using the same cell/detector combination used for establishing the calibration factors.

e) The bubbler used for radium-226 standardization shall not be used for sample analysis.

1.8.3 Liquid Scintillation Counting

a) <u>Tritium in Water:</u>

Water samples for tritium analysis and all associated QC samples shall be distilled prior to analysis unless specified otherwise by the client. The applicable preparation SOP shall specify the fraction to be collected. The same fraction shall be collected for samples and all associated QC samples.

b) <u>Counting Vial Preparation:</u>

Samples shall be counted in vials equivalent to or superior to low potassium glass vials or high density polyethylene vials. Samples in polyethylene vials shall be counted within a time period not to exceed the manufacturer's specification for the cocktail used in the analysis. Analysis documentation shall contain sufficient information for this to be verified. Vials shall be prepared according to manufacturer's specification for the cocktail. The vials shall be "dark adapted" for a minimum of 30 minutes or according to

the cocktail manufacturer's specifications before counting. The prepared vials shall be inspected to verify that the sample loaded properly in the cocktail.

c) Laboratory SOPs for methods using liquid scintillation counting shall incorporate and adhere to ANSI N42.15 (current version), American National Standard Check Sources for and Verification of Liquid Scintillation Systems. Performance shall be in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. References are for the current version. When references are updated, an implementation schedule shall be determined by the lab.

d) Instrument Background:

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. The instrument background vial shall be prepared with the same water to cocktail ratio as the samples are prepared. The type of water used to prepare the instrument background vial shall be explicitly noted on the preparation and counting documentation. The instrument background shall be run with each sample batch. Unless calculated from a running average of background counts or a background quench curve, the most recent background count shall be used to calculate sample activities and MDAs. This is not a performance check, rather a background subtraction sample in a configuration equivalent to that of associated samples in the batch. It is used to generate the background subtraction data for the batch (using the results associated directly with that batch, results of a rolling mean, or background quench curve). The effect of quench on background shall be evaluated and corrected using a background quench curve if it is significant.

e) For analysis methods using quench curves to determine individual sample detection efficiency or background, the quench curves shall be generated at a frequency defined by the laboratory.

f) If the calibration method is constant quench, the detection efficiency shall be checked at least weekly when in use or with each counting batch.

g) Sample-Specific Conditions:

The following are conditions that require reanalysis for a particular sample and analyte, beginning with the preparation or recounting, as appropriate.

i) If the constant quench method of calibration is used, the quench of each sample analyzed shall fall within +/-5% relative to the average efficiency at that quench level. If this condition is not met, the sample must be reanalyzed beginning with vial preparation.

ii) If the sample quench does not fall within the range of the quench curve, the samples shall be reanalyzed such that the sample quench is in the range of a quench curve.

h) Spectrum Assessment:

For analytes requiring separations other than distillation:

i) Sample spectra shall be retained (electronic or hardcopy) for each sample and QC sample including identification of ROIs.

ii) 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.

1.8.4 Gas Flow Proportional Counting

a) Planchets:

Planchets shall be thoroughly cleaned before use to ensure that there are no interfering residues or contamination. All Planchets shall be prepared not to exceed sample weights in excess of the calibrated ranges of established self-absorption curves. Sample weights shall be documented and stable prior to counting. Planchets exhibiting physical characteristics notably different from the self-absorption standards (e.g., evidence of corrosion) shall not be counted unless remediation efforts such as additional sample preparation and remounting or flaming prove unsuccessful. Any non-routine counting situations shall be documented in the Case Narrative.

b) Laboratory SOPs for sample analysis by gas flow proportional counting shall incorporate and adhere to ANSI N42.25 (current version), Calibration and Usage of Alpha/Beta Proportional Counters. Performance shall be performed in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. References are for the current version. When references change, an implementation schedule shall be determined.

c) Calibration Sources and Standards:

The standard reference material used to prepare sources for determining detector efficiencies and self-absorption curves shall be traceable to NIST or accepted international standards. The calibration sources shall provide adequate counting statistics over the period for which the source is to be counted.

i) However, the source shall not be so radioactive as to cause pulse pileups or dead time that is significantly different from that to be expected from routine analyses.

ii) The geometry of the calibration sources used for efficiency and self-absorption/cross-talk curves shall be the same as that of the prepared sample and QC sample planchets. The depth and shape (flat, flanged, ringed, etc.), in addition to the diameter, are factors that shall be the same for calibration sources as for samples.

iii) The sources used for the determination of self-absorption and cross-talk should be of similar isotope content to that of the analytical samples. Am-241; Po-210; or Th-230 shall be used for alpha and Cs-137 or Sr-90/Y-90 for beta.

d) Self-Absorption and Cross-talk Curves:

i) Self-absorption curves are required for both alpha and beta counting.

ii) A cross-talk curve shall be established for alpha to beta cross-talk versus residue weight.

iii) 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.

iv) The data used to generate self-absorption and cross-talk curves shall consist of at least seven points, well distributed throughout the mass range.

v) Each alpha and beta calibration standard shall be counted to an accumulation of at least 10,000 counts minimum for the initial calibration and 5,000 counts minimum for the calibration verification.

vi) A new cross-talk curve must be measured prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1).

e) Check Source Requirements:

i) The alpha and beta response and corresponding cross-talk of each detector used to count analytical samples or QC samples shall be checked daily with separate alpha and beta emitting sources. The only exception to this requirement is when performing analyses with extended count times. In this case, check source measurements may be performed between sample sets.

ii) Following gas bottle changes, check sources and backgrounds shall be analyzed before samples are counted.

iii) Check source data shall be documented and retained.

1.8.5 Gamma Spectrometry

a) Sample Counting Requirements:

i) Laboratory SOPs for sample analysis by gamma spectrometry shall incorporate and adhere to ANSI N42.14 (current version), Calibration and Use of Germanium Spectrometers for the Measurement of Gamma Ray Emission Rate of Radionuclides, and/or ANSI N42.12 (current version), Calibration and Usage of Thallium-Activated Sodium Iodide Detector Systems for Assay of Radionuclides. Performance shall be in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. References are for the current version. When references change, an implementation schedule will be determined.

ii) The gamma detector system shall consist of any detector suitable for measuring the gamma isotopes of interest in the typical energy range of approximately 0.059 to 2 MeV with the capacity to attain specified RLs 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.

iii) Detectors shall be calibrated for the specific geometry and matrix considerations used in the sample analysis. The laboratory shall have the capability to seal soil (or other solid matrix) samples in airtight cans or equivalent in order to allow ingrowth of radon for accurate analysis of Ra-226 or its progeny by gamma spectroscopy when requested.

iv) Spectral Data Reference:

Identification of the reference used for the half-life, abundance, and peak energy of all nuclides shall be documented. The laboratory shall document, review, and provide configuration control for gamma spectrometry libraries. Assumptions made for libraries (i.e., half-lives based on supported/unsupported assumptions, inferential determinations (e.g., Th-234 = U-238 because supported)) shall be documented and narrated.

b) Efficiency Calibration Requirements:

i) Each gamma spectrometry system shall be efficiency calibrated for the sample geometry and matrix with NIST traceable or accepted international standards or prepared from NIST/international traceable sources.

Germanium Detectors: Refer to ANSI N42.14 for guidance on isotope specific efficiency and efficiency as a function of energy calibrations. The efficiency

calibration approach selected for broad spectrum gamma analysis must cover the energy range of the gamma ray peaks used for nuclide quantification. 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.

At least 10,000 net counts (total counts minus the Compton continuum and ambient background) shall be accumulated in each full-energy gamma-ray peak of interest used for the efficiency equation (ASTM D 3649-98a).

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. Acceptable approaches include:

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 LCSs to contain the isotope to be quantified (or other isotope with lower emission energies). Acceptable recovery must be demonstrated for every detector used for that isotope analysis.

2) 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.

In all cases, the laboratory must be able to demonstrate that sample matrix effects (including potential attenuation from sample containers) on low energy emissions have been accounted for.

<u>Sodium Iodide Detectors:</u> Refer to ANSI N42.12.

Efficiencies shall be determined when there is a change in resolution, geometry, or system configuration (ASTM D 3649-98a).

ii) Current software that does not require a physical calibration standard to obtain efficiencies for various matrices and geometries may be used to count samples where a standard calibration source of known matrix and geometry cannot be specified. This type of calibration technique is preferred for matrices such as waste or debris. When such software is used, the laboratory shall supply detailed information and documentation regarding the selection of parameters used to specify the efficiency calibration and sample models. Each sample selected for analysis using this type of calibration shall have a unique set of model parameters associated

with it. When such models are used, the closest model to the actual sample shall be selected. The model selected for each sample shall be presented in the Case Narrative and shall include a discussion of actual and predicted peak ratios for isotopes with multiple gamma energies present in the sample.

c) Energy Calibration Requirements:

Each gamma spectrometry system shall be energy calibrated with NIST/international traceable standards or prepared from NIST/international traceable sources.

Germanium Detectors: Refer to ANSI N42.14, Section 5.1 for guidance on calibrating gamma-ray energy as a function of channel number at a fixed gain. The energy calibration measurements shall be made using at least six peaks which cover the energy range from 0.059 to approximately 2 MeV. Additional peaks shall be used as deemed appropriate by the laboratory.

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).

Energy calibration shall be linear and accurate to 0.5 keV.

Sodium Iodide Detectors: Refer to ANSI N42.12, Section 4.3.2.

d) Performance Evaluation:

Germanium Detectors: Refer to ANSI N42.14, Section 7.

Sodium Iodide Detectors: Refer to ANSI N42.12, Section 4.3.5.

e) Spectrum Assessment:

Each sample and QC sample spectrum shall be assessed for acceptability of key peak width and shape and interference due to superimposed peaks or other sources. Any major contributor to the spectrum that is an unidentified peak shall be discussed in the Case Narrative.

Volume 1, Module 7: Quality Systems for Toxicity Testing

No additions or clarifications were made to Volume 1, Module 7. **The TNI Standard shall be followed.**

Appendix A: Reporting Requirements

In the absence of client specified reporting criteria, the reporting requirements outlined in this appendix shall be used for hard-copy data reports or electronic versions of hard-copy data (such as pdf). They include mandatory requirements for all printed data reports and requirements for data reports requiring third party data review or validation. Optional reporting requirements are those that may be required by a specific project depending upon their needs. The following elements are required: 1) cover sheet, 2) table of contents, 3) Case Narrative, 4) analytical results, 5) sample management records, and 6) Quality Control (QC) information. Information for third-party review may be required depending on project-specific requirements or the method being used.

1.0 COVER SHEET

The cover sheet shall specify the following information:

a) title of report (i.e., test report, test certificate);

b) name and location of laboratory (to include a point of contact, phone and facsimile numbers, and e-mail address);

c) name and location of any subcontractor laboratories and appropriate test method performed (information can also be located in the Case Narrative as an alternative);

d) unique identification of the report (such as serial number);

e) client name and address;

f) project name and site location;

g) statement of data authenticity and official signature and title of person authorizing report release, date of issuance;

h) amendments to previously released reports that clearly identify the serial number for the previous report and state the reason(s) for reissuance of the report; and

i) total number of pages.

2.0 TABLE OF CONTENTS

Laboratory data packages shall be organized in a format that allows for easy identification and retrieval of information. An index or table of contents shall be included for this purpose.

3.0 CASE NARRATIVE

A Case Narrative shall be included in each report. The purpose of the Case Narrative is to:

1) Describe any abnormalities, deviations, and failures that may affect the analytical results;

2) Summarize any issues in the data package that need to be highlighted for the data user to help them assess the usability of the data; and

3) Provide a summary of samples included in the report with the methods employed in order to assist the user in interpretation.

The Case Narrative shall provide:

a) a table(s) summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical, preparation, and clean-up methods were performed. If multiple laboratories performed analyses, the name and location of each laboratory shall be associated with each sample;

b) a list of samples that were received but not analyzed;

c) date of samples received;

d) sample preservation and condition at receipt;

e) a description of extractions or analyses that were performed out of holding times;

f) a definition of all data qualifiers or flags used;

g) identification of deviations of any calibration standards or QC sample results from appropriate acceptance limits and a discussion of the associated corrective actions taken by the laboratory;

h) identification of multiple sample runs with reason(s) identified (e.g., dilutions or multiple cleanups);

i) identification of instances where manual integration was performed including the technical justification; and

j) appropriate notation of any other factors that could affect the sample results (e.g., air bubbles in volatile organic compounds (VOC) sample vials, excess headspace in soil VOC containers, the presence of multiple phases, sample temperature or pH excursions, and container type or volume).

4.0 ANALYTICAL RESULTS

The results for each sample shall contain the following information at a minimum:

a) project name and site location;

b) field sample ID number as written on custody form;

c) laboratory sample ID number;

d) preparation batch number(s);

e) matrix (soil, water, oil, air, etc.);

f) date and time sample collected;

g) date and time sample prepared;

h) date and time sample analyzed;

i) method numbers for all preparation, cleanup, and analysis procedures employed;

j) analyte or parameter with the Chemical Abstracts Service (CAS) Registry Number if available;

k) sample aliquot analyzed;

l) final extract volume;

m) identification of analytes for which manual integration occurred, including the cause and justification;

n) analytical results with correct number of significant figures;

o) Detection Limit, Limit of Detection, and Limit of Quantitation associated with sample results and adjusted for sample-specific factors (e.g., aliquot size, dilution/concentration factors, and moisture content);

p) any data qualifiers assigned;

q) concentration units;

r) dilution factors;

s) all multiple sample run results shall be reported;

t) percent moisture or percent solids (all soils are to be reported on a dry weight basis); and

u) statements of the estimated uncertainty of test results (optional).

5.0 SAMPLE MANAGEMENT RECORDS

Sample Management records shall include the documentation accompanying the samples, such as:

a) chain of custody records;

b) shipping documents;

c) 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);

d) telephone conversation or e-mail records associated with actions taken or quality issues; and

e) records of sample compositing done by the laboratory.

6.0 QC INFORMATION

The minimum laboratory internal QC data package shall include:

a) Method Blank results;

b) percent recoveries for Laboratory Control Samples **(**LCS), Laboratory Control Sample Duplicates (LCSD), Matrix Spikes (MS), and Matrix Spike Duplicates (MSD);

c) MSD or Matrix Duplicate relative percent differences (RPD);

d) surrogate percent recoveries;

e) tracer recoveries;

f) spike concentrations for LCS, MS, surrogates;

g) QC acceptance criteria for LCS, MS, surrogates;

h) Post-Digestion Spike (PDS) recoveries;

i) serial dilutions percent difference; and

j) batch numbers (preparation, analysis, and cleanup).

7.0 DATA REPORTS FOR THIRD PARTY REVIEW OR VALIDATION

The data validation guidelines established in other DoD/DOE guidance or project-specific guidelines may have distinct reporting formats. The appropriate QAPP should be consulted to determine what type (stage) of data package is required.

Data validation guidelines define the minimum reporting requirements for each stage (formerly called “level”) of data package as outlined below:

A cover sheet, table of contents, and Case Narrative including all of the information specified in the previous sections are required for all stages of data reports.

Stage 1: Sample results forms, chain of custody, laboratory receipt checklist.

Stage 2A: Sample results forms, chain of custody, laboratory receipt checklist, method QC forms.

Stage 2B: Sample results forms, chain of custody, laboratory receipt checklist, method QC forms, instrument QC forms, instrument and preparation logs.

Stage 3: Sample results forms, chain of custody, laboratory receipt checklist, method QC forms, instrument QC forms, instrument and preparation logs, instrument quantitation forms (raw data), standards traceability.

Stage 4: Sample results forms, chain of custody, laboratory receipt checklist, sample related method QC forms, instrument QC forms, instrument and preparation logs, instrument quantitation forms (raw data), instrument chromatograms and spectra, standards traceability.

Appendix B: Quality Control Requirements

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be ≤ 15%.	Correct problem, then repeat breakdown checks.	Flagging is not appropriate.	No samples shall be run until degradation of DDT and Endrin is each ≤ 15%.
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD 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.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
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.	NA.	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticide multi-component analytes (i.e., Toxaphene, Chlordane and Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards (IS)	If employed, every field sample, standard, and QC sample.	Retention time within ± 0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-1. Organic Analysis by Gas Chromatography (GC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All results > the DL must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not a requirement).	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%.	NA.	Apply J-flag if RPD > 40%. Discuss in the Case Narrative.	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.

Table B-2. Organic Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD 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.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
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.	NA.	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.

Table B-2. Organic Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 15% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 15% true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.

Table B-2. Organic Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	If employed, every field sample, standard and QC sample.	Retention time within ± 30 seconds from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% 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. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-2. Organic Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for the failed reported analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-2. Organic Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-2. Organic Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

Table B-2. Organic Analysis by High-Performance Liquid Chromatography (HPLC) (Including Method 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation of positive results (second column)	All results > the DL must be confirmed.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column/detector RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the Case Narrative.	Spectral match confirmation of a UV detector with a UV diode array detector (or vice versa) is not considered an acceptable confirmation technique. A second column confirmation is required. Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise, report the result from the primary column.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Soil drying procedure	Each sample, LCS, and Method Blank. The appropriateness of the drying step is determined by each project.	Laboratory must have a procedure to determine when the sample is dry to constant mass. Entire sample must be air dried at room temperature.	NA.	Flagging is not appropriate.	Commercial PT samples must reflect the grinding, extraction, and analysis steps as a minimum. Record date, time, and ambient temperature on a daily basis while drying samples. If a laboratory utilizes a self-spiked LCS, the fortification must be performed prior to any preparation steps performed (drying, grinding, etc.) Drying may introduce a bias and is not recommended for certain compounds. Drying should be performed in the laboratory, not the field. LCS reference material is not required to be air dried if the vendor specifies that drying is not required. LCS and Blank matrix can be Ottawa sand, clean soil, or other vendor provided clean matrix.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Soil sieving procedure	Each sample, LCS, and Method Blank. The appropriateness of the sieving step is determined by each project.	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Collect and weigh any portion unable to pass through the sieve.	NA.	Flagging is not appropriate.	Do not include vegetation or debris in the portion of the sample that passes through the sieve unless that is a project specific requirement. Projects may require an alternate sieve size.
Soil grinding procedure	Initial demonstration at start up and any time major equipment is changed or when a reduction in the number or time of grinding cycles occurs. Each required sample, LCS, Blank, and Matrix Spike sample. The appropriateness of the grinding step is determined by each project.	**Initial demonstration of grinding equipment**: The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 µm by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA.	Flagging is not appropriate.	Grinding and sieving is an iterative process, so cycles and duration can be varied to reduce heat if all samples are treated the same. Grinding may introduce a bias and is not recommended for certain compounds. Each sample, LCS, and Method Blank must use the same grinding process (i.e., same time intervals and number of grinding cycles).

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Grinding Blanks	One per batch of samples. The Grinding Blank must be processed: after the LCS (if ground), or after a client identified sample with known contamination, or at the end of the batch.	No reported analytes must be detected > 1/2 LOQ.	Blank results must be reported and the affected samples must be flagged accordingly if blank criteria are not met. If required, reprep and reanalyze Method Blank and all QC samples and field samples processed with the contaminated blank.	If any individual Grinding blank is found to exceed the acceptance criteria, apply B-flag to the samples following that blank.	At least one Grinding Blank per batch must be analyzed. For batch preparation, the Grinding Blank and the Method Blank can be one in the same. A Grinding Blank using clean solid matrix (such as Ottawa sand) must be prepared (e.g., ground and subsampled) and analyzed in the same manner as the sample. If cross-contamination is a concern, then more than one Grinding Blank per batch may be necessary.
Soil subsampling process	Each sample, LCS, blank, and Matrix Spike sample. All sample types must be subsampled, including those that were not initially dried, ground, or sieved.	Entire sample is mixed and spread out evenly on a large flat surface (e.g., baking tray), and 30 or more randomly located increments are removed from the entire depth and breadth to obtain the appropriate subsample size.	NA.	Flagging is not appropriate.	The total subsample weight collected can vary based on the requirements of the extraction process.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Soil Sample Triplicate	At the subsampling step, performed on one sample per batch. Cannot be performed on any sample identified as a blank (e.g., Field Blank, Method Blank, Grinding Blank).	The RSD for results above the LOQ must not exceed 20%.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	If reported per the client, apply J-flag to all samples within that batch if acceptance criteria are not met and explain in the Case Narrative.	Sample triplicates are randomly selected unless the project specifies the sample to be used.
Aqueous sample preparation	Each sample and associated batch QC samples.	Solid phase extraction (SPE) using resin-based solid phase disks or cartridges are required.	NA.	Flagging is not appropriate.	The salting-out procedure is not permitted.
Ion Transitions (Parent-> Product)	Prior to method implementation.	The chemical derivation of the ion transitions must be documented.	NA.	Flagging is not appropriate.	NA.
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument setup and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; 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.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analyte(s) and surrogates within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within ± 20% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards (IS)	If employed, every field sample, standard and QC sample.	Retention time within ± 30 seconds from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect instrumentation for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze Method Blank and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. For batch preparation, the Grinding Blank and the Method Blank can be one in the same.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. Use LCS Tables 8330B for HPLC analysis. Use LCS Tables 8321 for LC/MS or LC/MS/MS analysis.	Correct problem. If required, reprep and reanalyze the LCS and all samples in the associated preparatory batch for the failed analytes, from the sub-sampling step on, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	A solid reference material containing all reported analytes must be prepared (e.g., ground and subsampled) and analyzed in exactly the same manner as a field sample. 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. If a laboratory utilizes a self-spiked LCS, the fortification must be performed prior to any preparation steps performed, such as drying, grinding, and sieving. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Analytes and surrogates are spiked into the MS and MSD after subsampling. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Analytes and surrogates are spiked into the MS and MSD after subsampling. For matrix evaluation only. If MSD results are outside the limits, the data shall be evaluated to determine the source(s) of difference. For Sample/MD: %Recovery and RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Confirmation of positive results (second column)	All results > the DL must be confirmed.	Calibration and QC criteria are the same for the confirmation analysis as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	Report from both columns.	Apply J-flag if RPD > 40%. Discuss in the Case Narrative.	Use of a UV detector with a UV diode array detector or vice versa is not considered a valid confirmation technique. Confirmation analysis is not needed if LC/MS or LC/MS/MS was used for the primary analysis. Secondary column – Must be capable of resolving (separating) all of the analytes of interest and must have a different retention time order relative to the primary column. Use project specific reporting requirements if available; otherwise, report from the primary column.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until performance check is within criteria. The DDT breakdown and Benzidine/ pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	Each analyte must meet one of the three options below: <u>Option 1:</u> RSD for each analyte ≤ 15%; <u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$; *(continued next page)*	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes (including surrogates) *(Continued)*		Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.			If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA.	After maintenance is performed which may affect retention times, RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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 ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal Standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 10 seconds from retention time 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. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL.	Verify mass calibration per method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Retention Time window defining mix	At method set-up and prior to analyzing calibration standards.	Verify descriptor switching times per method.	Correct problem, then repeat Retention Time window defining mix.	Flagging is not appropriate.	NA.
GC column performance check (for SP-2331 column or equivalent)	At the beginning and end of each 12-hr period during which samples or calibration solutions are analyzed.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers: Resolved with a valley of ≤ 25%. For calibration verification standard only: Peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD must be resolved with a valley of ≤ 50%, per method.	Correct problem, then repeat column performance checks.	Flagging is not appropriate.	Needed only if using a column other than DB-5 or equivalent.

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
GC Column performance check (for DB-5 column or equivalent)	At the beginning and end of each 12-hr period during which samples or calibration solutions are analyzed. Included with the ICAL standard (CC3) and the calibration verification standard.	Peak separation of standard CC3: Peak between the ^{13}C-2,3,7,8-TCDD and ^{13}C-1,2,3,4-TCDD must be resolved with a valley of ≤ 25%; For calibration verification standard only: Peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD must be resolved with a valley of ≤ 50%.	Correct problem, then repeat column performance checks.	Flagging is not appropriate.	No samples shall be analyzed until GC column performance check is within criteria.
Initial calibration (ICAL) for all analytes identified in method	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	Ion abundance ratios must be in accordance with the method. RSD of the RFs ≤ 15% for labeled IS and unlabeled PCDD/PCDF.	Correct problem then repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ICAL has passed.

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Ion abundance specified in the method must be met for all PCDD/PCDF peaks, including labeled internal and recovery standards. Sensitivity criteria of an S/N ratio > 2.5 for unlabeled PCDD/PCDF ions and > 10 for labeled internal and recovery standards. All reported analytes and IS within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Calibration Verification (CCV)	At the beginning of each 12-hr period of sample analysis, after successful GC and MS resolution checks.	Ion abundance specified in the method must be met for all PCDD/PCDF peaks, including labeled internal and recovery standards. Sensitivity criteria of an S/N ratio > 2.5 for unlabeled PCDD/PCDF ions and > 10 for labeled internal and recovery standards. All reported analytes and IS within ± 20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards (IS)	Every field sample, standard, and QC sample.	% Recovery for each IS in the original sample (prior to any dilutions) must be within 25-150% of the CCV.	Correct problem, then reprep and reanalyze the sample(s) with failed IS.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant Internal Standard Flagging is not appropriate for failed standards.	NA.

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Sensitivity Check	At the end of 12-hr sample analysis period or at the end of analysis (whichever comes first). Injection must be done within the 12-hr period.	See calibration verification for criteria on ion abundances, and S/N ratios. See Retention Time window defining mix for retention time criteria.	Correct problem, then repeat calibration and reanalyze samples indicating a presence of PCDD/PCDF less than LOQ or when maximum possible concentration is reported.	Flagging is not appropriate.	NA.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	Use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike (MS)	One per preparatory batch.	Use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	Use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, otherwise use in-house limits.	Correct problem, then re-prep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	NA.

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Sample PCDD/PCDF Identification	Identify all positive sample detections per method.	Verify that absolute RT at maximum height is within −1 to +3 seconds of that for corresponding labeled standard, or the RRT of analytes is within 0.05 RRT units of that for unlabeled standard in the calibration verification standard, or RT for non-2,3,7,8-substituted isomers within the RT window established by the window defining mix for the corresponding homologue per method. Absolute RTs of the recovery standards must be within ± 10 seconds of those in the calibration verification standard. All ions listed in Table 8 of the method must be present in the SICP, must maximize simultaneously (± 2 sec.), and must have not saturated the detector. *(continued next page)*	Correct problem, then re-prep and reanalyze the sample(s) with failed criteria for any of the internal, recovery, or cleanup standards. If PCDPE is detected or if sample peaks present do not meet all identification criteria, calculate the EMPC (estimated maximum possible concentration) according to the method.	Flagging is not appropriate.	Positive identification of 2,3,7,8-TCDF on the DB-5 or equivalent column must be reanalyzed on a column capable of isomer specificity (DB-225).

Table B-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Sample PCDD/PCDF Identification *(Continued)*		S/N ratio of Iss ≥ 10 times background noise. Remaining ions in Table 8 of the method must have an S/N ratio ≥ 2.5 times the background noise. Ion abundance in Table 9 of the method must be met for all analytes, internal, and recovery standards.			

Table B-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Resolving Power	Prior to 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.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check	Prior to ICAL or calibration verification. At the beginning of each 12-hr period during which samples or calibration solutions are analyzed.	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 eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram. Absolute retention times for switching from one homologous series to the next ≥ 10 sec. for all components of the mixture.	Correct problem then repeat column performance check.	Flagging is not appropriate.	Use GC column performance check solution If the laboratory operates during consecutive 12-hr periods. No samples shall be analyzed until performance check is within criteria.
Initial calibration (ICAL) for all analytes identified in method	At instrument setup and after ICV or CCV failure, prior to sample analysis, and when a new lot is used as standard source for HRCC-3, sample fortification (IS), or recovery solutions.	Ion abundance ratios in accordance with the method. S/N ratio ≥ 10 for all reported analyte ions. RSD ≤ 20% for the response factors (RF) for all 17 unlabeled standards. RSD ≤ 20% for the RFs for the 9 labeled IS.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed run until ICAL has passed. Calibration may not be forced through origin.

Table B-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Ion abundance specified in the method must be met. For unlabeled standards, RF within ± 20% D of RF established in ICAL; and For labeled standards, RF within ± 30%D of the mean of RF established in ICAL.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Calibration Verification (CCV)	At the beginning of each 12-hour period and at the end of each analytical sequence.	Ion abundance specified in the method must be met. For unlabeled standards, RF within ± 20% D of RF established in ICAL; and For labeled standards, RF within ± 30% D of RF established in ICAL.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV. *(continued next page)*	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Calibration Verification (CCV) *(Continued)*			End-of-run CCV: If the RF for unlabeled standards ≤ 25% RPD and the RF for labeled standards ≤ 35% RPD (relative to the RF established in the ICAL), the mean RF from the two daily CCVs must be used for quantitation of impacted samples instead of the ICAL mean RF value. If the starting and ending CCVRFs differ by more than 25% RPD for unlabeled compounds or 35% RPD for labeled compounds, the sample may be quantitated against a new initial calibration if it is analyzed within two hours. Otherwise analyze samples with positive detections, if necessary.		
Internal Standards (IS)	Every field sample, standard, and QC sample.	% Recovery for each IS in the original sample (prior to dilutions) must be within 40-135% of the ICAL average RF.	Correct problem, then re-prep and reanalyze the samples with failed IS.	Apply Q-flag to results of all affected samples and explain in the Case Narrative.	

Table B-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per preparatory batch, run after calibration standards and before samples.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table B-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Sample Estimated Maximum Possible Concentration (EMPC)	Every sample with a response S/N ≥ 2.5 for both quantitation ions.	Identification criteria per method must be met, and the S/N of response for both quantitation ions must be ≥ 2.5.	NA.	Flagging is not appropriate.	NA.
Sample 2,3,7,8-TCDD toxicity equivalents (TEQ) concentration	All positive detections.	Per method.	NA.	Flagging is not appropriate.	Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	$R^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a Calibration Blank. CVAA/Mercury: minimum 5 standards and a Calibration Blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-level Calibration Check Standard (LLCCV)	Daily.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid Low-Level Calibration Check Standard (LLCCV). LLCCV should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as the LLCCV prior to the analysis of any samples.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reprepped or reanalyzed. Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be re-analyzed without re-analysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without valid Calibration Blanks Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: %Recovery and RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution Test (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the Case Narrative.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a Calibration Blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-Level Calibration Check Standard (LLCCV)	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid Low-Level Calibration Check Standard (LLCCV). LLCCV should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as LLCCV prior to the analysis of any samples.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be re-analyzed without re-analysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without valid Calibration Blanks. Non-detects associated with positive blank infractions may be reported. Sample results >10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: 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.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to the source(s) of difference (i.e., matrix effect or analytical error).

Table B-8. Inorganic Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Criteria applies for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA.	NA.	NA.	Document use of MSA in the Case Narrative.

Table B-9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or High-Level Check Standard	At initial set-up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the calibration range without an established/passing high-level check standard.
Tuning	Prior to ICAL.	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a Calibration Blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Low-Level Calibration Check Standard (LLCCV)	Daily.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid Low-Level Calibration Check Standard (LLCCV). LLCCV should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as LLCCV. Otherwise, a separate standard must be analyzed as the LLCCV prior to the analysis of any samples.

Table B-9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem and rerun all associated failed field samples.	Flagging is not appropriate.	Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.

Table B-9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be re-analyzed without re-analysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without valid Calibration Blanks. Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: 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.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.

Table B-9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the Case Narrative.

Table B-10. Inorganic Analysis by Colorimetric Hexavalent Chromium

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL)	Daily ICAL prior to sample analysis.	$R^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum three standards and a Reagent Blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis, after every 15 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for hexavalent chromium in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-10. Inorganic Analysis by Colorimetric Hexavalent Chromium

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for hexavalent chromium in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for hexavalent chromium in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-10. Inorganic Analysis by Colorimetric Hexavalent Chromium

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	Once per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Dilute and reanalyze sample; persistent interference indicates the need to use the method of standard addition, alternative analytical conditions, or an alternative method.	Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error). Verification check ensures lack of reducing conditions or interference from matrix.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	Aqueous matrix: One per every 10 project samples. Solid matrix: One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Dilute and reanalyze sample; persistent interference indicates the need to use the method of standard addition, alternative analytical conditions, or an alternative method. Re-prep and reanalyze all samples in the prep batch.	Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. Results may not be reported without a valid pair. Flagging is only appropriate in cases where the samples cannot be reanalyzed. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-10. Inorganic Analysis by Colorimetric Hexavalent Chromium

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soluble and Insoluble Pre-Digestion Matrix Spikes (solid matrix samples only)	One soluble and insoluble pre-digestion MS analyzed per preparatory batch prior to analysis.	MS recoveries within 75-125%.	Correct problem and re-homogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results.	Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the Case Narrative.	NA.
Post-digestion Matrix Spike (solid matrix samples)	One per preparatory batch.	Recovery within 85 – 115%.	No specific CA unless required by the project.	Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the Case Narrative.	Criteria apply for samples with concentrations > 50 X LOQ prior to dilution.

Table B-11. Cyanide Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL)	Daily ICAL prior to sample analysis.	$R^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum three standards and a Reagent Blank. No samples shall be analyzed until ICAL has passed.
Distillation Verification	Once after each ICAL, with two distilled ICAL standards prior to sample analysis. Not required if all ICAL standards are distilled.	Within ± 10% of non-distilled standard value.	Correct problem, rerun distilled standards or repeat ICAL.	Flagging is not appropriate.	One high and one low distilled ICAL standard. No samples shall be analyzed until distillation technique has been verified.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified.

Table B-11. Cyanide Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	Within ± 10% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for cyanide in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-11. Cyanide Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all cyanide results in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-11. Cyanide Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be < ½ LOQ or < 1/10th the amount measured in any sample.	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCBs may not be re-analyzed without re-analysis of the associated samples and CCV(s).	Flagging is not appropriate.	Results may not be reported without valid Calibration Blanks Non-detects associated with positive blank infractions may be reported. Sample results > 10X the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-rep and reanalyze the LCS and all samples in the associated preparatory batch if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-11. Cyanide Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to the source of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) and Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the Case Narrative.

Table B-12. Common Anions Analysis by Ion Chromatography (IC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	ICAL prior to sample analysis.	$R^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Minimum 3 standards and a Calibration Blank. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per multipoint calibration.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte.
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 over a 24-hour period.	NA.	NA.	Calculated for each analyte.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Freshly prepared ICV. No samples shall be analyzed until calibration has been verified.

Table B-12. Common Anions Analysis by Ion Chromatography (IC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis; after every 10 field samples; and at the end of the analysis sequence.	All reported analytes within established retention time windows. All reported analytes within ± 10% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-12. Common Anions Analysis by Ion Chromatography (IC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for all reported analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Follow project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all reported analytes. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-12. Common Anions Analysis by Ion Chromatography (IC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 15% (between MS and MSD or sample and MD).	Follow project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all reported analytes. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-13. Perchlorate by Mass Spectrometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference Threshold 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).	Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the concentration of the common suppressors where perchlorate recovery falls outside an 80-120% window.	NA.	Flagging criteria are not appropriate.	This study and site history will determine the concentration at which the ICS suppressors should be set.
Mass Calibration	Instrument must have a valid mass calibration prior to any sample analysis. The mass calibration is updated on an as-needed basis (e.g., QC failures, ion masses show large deviations from known masses, major instrument maintenance is performed, or the instrument is moved).	Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for an analytical run, and the same mass calibration must be used for all data files in an analytical run. Mass calibration must be verified by acquiring a full scan continuum mass spectrum of a perchlorate stock standard.	If the mass calibration fails, recalibrate. If it still fails, consult manufacturer instructions on corrective maintenance.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be analyzed under a failing mass calibration. Perchlorate ions should be within ± 0.3 *m/z* of mass 99, 101, and 107 or their respective daughter ion masses (83, 85, and 89), depending on which ions are quantitated.

Table B-13. Perchlorate by Mass Spectrometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standards must span the mass range of the analytes of interest and meet acceptance criteria outlined in the laboratory SOP.	Retune instrument and verify. If the tune check will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Flagging is not appropriate.	No samples shall be analyzed without an acceptable tune check.
Initial Calibration (ICAL)	At instrument setup or after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the two options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Minimum of 6 calibration levels must be used. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL.	Perchlorate concentration must be within ± 15% of its true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	ICV shall be a second source standard with its concentration at the midpoint of the calibration. No samples shall be analyzed until calibration has been verified with a second source.

Table B-13. Perchlorate by Mass Spectrometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	On days an ICAL is performed, after every 10 field samples, and at the end of the analytical sequence. On days an ICAL is not performed, at the beginning of the sequence, after every 10 field samples, and at the end of the analytical sequence.	Perchlorate concentration must be within ± 15% of its true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-13. Perchlorate by Mass Spectrometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Isotope Ratio $^{35}Cl/^{37}Cl$	Every sample, batch QC sample, and standard.	Monitor for either the parent ion at masses 99/101 or the daughter ion at masses 83/85 depending on which ions are quantitated. Must fall within 2.3 to 3.8.	If criteria are not met, the sample must be rerun. If the sample was not pretreated, the sample must be re-extracted using cleanup procedures. If, after cleanup, the ratio still fails, use alternative techniques to confirm presence of perchlorate, e.g., a post spike sample or dilution to reduce any interference.	If reanalysis after cleanup fails to meet acceptance criteria, data must be qualified with a Q-flag and explained in the Case Narrative. The disposition of results of alternate techniques used to confirm presence of perchlorate must be discussed in the Case Narrative.	Decision to report data failing ratio check should be thoroughly documented in Case Narrative. The use of cleanup procedures, post spike samples, and dilutions must be identified in the Case Narrative.
Internal Standard (IS)	Addition of ^{18}O-labeled perchlorate to every sample, batch QC sample, standard, instrument blank, and Method Blank.	Measured ^{18}O IS area within ± 50% of the value from the average of the IS area counts of the ICAL. RRT of the perchlorate ion must be 1.0 ± 2% (0.98 – 1.02).	Rerun the sample at increasing dilutions until the ± 50% acceptance criteria are met. If criteria cannot be met with dilution, the interference is suspected and the sample must be re-prepped using additional pretreatment steps.	If reanalysis after pretreatment steps fails to meet acceptance criteria, data must be qualified with a Q-flag and explained in the Case Narrative.	If peak is not within retention time window, presence is not confirmed. Failing internal standard must be thoroughly documented in the Case Narrative.

Table B-13. Perchlorate by Mass Spectrometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference Check Sample (ICS)	One ICS is prepared with every batch of 20 samples and must undergo the same preparation and pretreatment steps as the samples in the batch. It verifies the method performance at the matrix conductivity threshold (MCT). At least one ICS must be analyzed daily. The ICS shall be prepared at the LOQ.	Perchlorate concentration must be within ± 20% of its true value.	Correct problem. Reanalyze all samples and QC samples in the batch. If poor recovery from the cleanup filters is suspected, a different lot of filters must be used to re-extract all samples in the batch. If column degradation is suspected, a new column must be calibrated before the samples can be reanalyzed.	Flagging criteria are not appropriate.	Analysis of a standard containing perchlorate at the LOQ and interfering anions at the concentration determined by the interference threshold study. No samples may be reported that are associated with a failing ICS.

Table B-13. Perchlorate by Mass Spectrometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Reagent Blank (LRB)	Prior to calibration and at the end of the analytical sequence.	No perchlorate detected > ½ LOQ.	Reanalyze Reagent Blank (until no carryover is observed) and all samples processed since the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated batch.	Problem must be corrected. Results may not be reported without valid Reagent Blanks. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Additional LRBs may be needed to ensure that there was no carryover from over range samples.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-13. Perchlorate by Mass Spectrometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem. Reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	LCS must be spiked at the LOQ. Problems must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. LCS must undergo the same preparation and pretreatment steps as the samples in the batch.

Table B-13. Perchlorate by Mass Spectrometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch per matrix.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The MS must be spiked at the LOQ. For matrix evaluation only. If MS results are outside the limits, the data must be evaluated to determine the source of the difference and to determine if there is a matrix effect or analytical error. MS must undergo the same preparation and pretreatment steps as the samples in the batch.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch per matrix.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 15% (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The MSD must be spiked at the LOQ. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-14. Chemical Warfare Agents by GC/MS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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 8270.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for all analytes and surrogates	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; 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.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If laboratory developed methodology requires additional evaluations (e.g., RFs or low calibration standard analysis and recovery criteria), these additional requirements must also be met.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes and surrogates within ± 25% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-14. Chemical Warfare Agents by GC/MS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
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.	NA.	NA.	Calculated for each analyte and surrogate.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes within established RT windows. All reported analytes and surrogates within ± 25% of true value. All reported analytes and surrogates within ± 50% for end of analysis sequence CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-14. Chemical Warfare Agents by GC/MS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, Standard and QC sample.	Retention time within ± 10 seconds from retention time 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. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	Use in-house LCS limits if project limits are not specified. Limits may be set at 50-150% until sufficient data has been generated to establish in-house control limits.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-14. Chemical Warfare Agents by GC/MS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	Use in-house LCS limits if project limits are not specified. Limits may be set at 50-150% until sufficient data has been generated to establish in-house control limits.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	Use in-house LCS limits if project limits are not specified. Limits may be set at 50-150% until sufficient data has been generated to establish in-house control limits. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-14. Chemical Warfare Agents by GC/MS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise limits may be set at 50-150% until sufficient data has been generated to establish in-house control limits.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Aqueous Sample Preparation	Each sample and associated batch QC samples.	Solid Phase Extraction (SPE) must be used unless samples are known to contain high PFAS concentrations (e.g., Aqueous Film Forming Foam (AFFF) formulations). Inline SPE is acceptable. Entire sample plus bottle rinsate must be extracted using SPE. Known high PFAS concentration samples require serial dilution be performed in duplicate. Documented project approval is needed for samples prepared by serial dilution as opposed to SPE.	NA.	NA.	Samples with > 1% solids may require centrifugation prior to SPE extraction. Pre-screening of separate aliquots of aqueous samples is recommended.
Solid Sample Preparation	Each sample and associated batch QC samples.	Entire sample received by the laboratory must be homogenized prior to subsampling.	NA.	NA.	NA.
Biota Sample Preparation	Each sample and associated batch QC samples.	Sample prepared as defined by the project (e.g., whole fish versus filleted fish).	NA.	NA.	NA.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
AFFF and AFFF Mixture Samples Preparation	Each sample and associated batch QC samples.	Each field sample must be prepared in duplicate (equivalent to matrix duplicate). Serial dilutions must be performed to achieve the lowest LOQ possible for each analyte.	NA.	NA.	Adsorption onto bottle is negligible compared to sample concentration so subsampling is allowed. Multiple dilutions will most likely have to be reported in order to achieve the lowest LOQ possible for each analyte.
Sample Cleanup Procedure	Each sample and associated batch QC samples. Not applicable to AFFF and AFFF Mixture Samples.	ENVI-Carb™ or equivalent must be used on each sample and batch QC sample.	NA.	Flagging is not appropriate.	Cleanup should reduce bias from matrix interferences.
Mass Calibration	Instrument must have a valid mass calibration prior to any sample analysis. Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL).	Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ±0.5 amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard.	If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance.	Flagging is not appropriate.	Problem must be corrected. No samples may be analyzed under a failing mass calibration. The mass calibration is updated on an as-needed basis (e.g., QC failures, ion masses fall outside of the ±0.5 amu of the true value, major instrument maintenance is performed, or the instrument is moved).

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Mass Spectral Acquisition Rate	Each analyte, Extracted Internal Standard (EIS) Analyte.	A minimum of 10 spectra scans are acquired across each chromatographic peak.	NA.	Flagging is not appropriate.	NA.
Calibration, Calibration Verification, and Spiking Standards	All analytes.	Standards containing both branched and linear isomers must be used when commercially available. PFAS method analytes may consist of both branched and linear isomers, but quantitative standards that contain the linear and branched isomers do not exist for all method analytes. For PFAS that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative standard.	NA.	Flagging is not appropriate.	Standards containing both branched and linear isomers are to be used during method validation and when reestablishing retention times, to ensure the total response is quantitated for that analyte. Technical grade standards cannot be used for quantitative analysis.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample PFAS Identification	All analytes detected in a sample.	The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFBA and PFPeA). Documentation of the primary and confirmation transitions and the ion ratio is required. In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50-150%. Signal to Noise Ratio (S/N) must be ≥ 10 for all ions used for quantification and must be ≥ 3 for all ions used for confirmation. Quant ion and confirmation ion must be present and must maximize simultaneously (±2 seconds).	NA.	PFAS identified with Ion ratios that fail acceptance criteria must be flagged. Any quantitation ion peak that does not meet the maximization criteria shall be included in the summed integration and the resulting data flagged as “estimated, biased high”.	For example: Ion Ratio = (quant ion abundance/ confirm ion abundance) Calculate the average ratio (A) and standard deviation (SD) using the ICAL standards. An acceptance range of ratio could be within A ±3SD for confirmation of detection.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Ion Transitions (Precursor-> Product)	Every field sample, standard, blank, and QC sample.	In order to avoid biasing results high due to known interferences for some transitions, the following transitions must be used for the quantification of the following analytes: PFOA: 413 → 369 PFOS: 499 → 80 PFHxS: 399 → 80 PFBS: 299 → 80 4:2 FTS: 327 → 307 6:2 FTS: 427 → 407 8:2 FTS: 527 → 507 NEtFOSAA: 584 → 419 NMeFOSAA: 570 → 419 If these transitions are not used, the reason must be technically justified and documented (e.g., alternate transition was used due to observed interferences).	NA.	Flagging is not appropriate	NA.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	The isotopically labeled analog of an analyte (Extracted Internal Standard Analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation). Commercial PFAS standards available as salts are acceptable providing the measured mass is corrected to the neutral acid concentration. Results shall be reported as the neutral acid with appropriate CAS number. If a labeled analog is not commercially available, the Extracted Internal Standard Analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (Internal Standard Quantitation) Analytes must be within 70-130% of their true value for each calibration standard. *(continued next page)*	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ICAL has passed. External Calibration is not allowed for any analyte. Calibration can be linear (minimum of 5 standards) or quadratic (minimum of 6 standards); weighting is allowed.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) *(Continued)*		ICAL must meet one of the two options below: Option 1: The RSD of the RFs for all analytes must be ≤ 20%. Option 2: Linear or non-linear calibrations must have r^2 ≥ 0.99 for each analyte.			
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and EIS.
Retention Time (RT) window width	Every field sample, standard, blank, and QC sample.	RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the daily calibration verification or, on days when ICAL is performed, from the midpoint standard of the ICAL. Analytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to analyte and labeled analog pairs.	Correct problem and reanalyze samples.	NA.	Calculated for each analyte and EIS.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument Sensitivity Check (ISC)	Prior to analysis and at least once every 12 hours.	Analyte concentrations must be at LOQ; concentrations must be within ±30% of their true values.	Correct problem, rerun ISC. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ISC has met acceptance criteria. ISC can serve as the initial daily CCV.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Analyte concentrations must be within ±30% of their true value.	Correct problem, rerun ICV. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified.
Continuing Calibration Verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence.	Concentration of analytes must range from the LOQ to the mid-level calibration concentration. Analyte concentrations must be within ±30% of their true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Instrument Sensitivity Check (ISC) can serve as a bracketing CCV.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument Blanks	Immediately following the highest standard analyzed and daily prior to sample analysis.	Concentration of each analyte must be ≤ ½ the LOQ. Instrument Blank must contain EIS to enable quantitation of contamination.	If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met. If sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria (>1/2 LOQ), they must be reanalyzed.	Flagging is only appropriate in cases when the sample cannot be reanalyzed and when there is no more sample left.	No samples shall be analyzed until instrument blank has met acceptance criteria. Note: Successful analysis following the highest standard analyzed determines the highest concentration that carryover does not occur. When the highest standard analyzed is not part of the calibration curve, it cannot be used to extend out the calibration range, it is used only to document a higher concentration at which carryover still does not occur.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Extracted Internal Standard (EIS) Analytes	Every field sample, standard, blank, and QC sample.	Added to solid sample prior to extraction. Added to aqueous samples, into the original container, prior to extraction. For aqueous samples prepared by serial dilution instead of SPE, added to final dilution of samples prior to analysis. Extracted Internal Standard Analyte recoveries must be within 50% to 150% of ICAL midpoint standard area or area measured in the initial CCV on days when an ICAL is not performed.	Correct problem. If required, re-extract and reanalyze associated field and QC samples. If recoveries are acceptable for QC samples, but not field samples, the field samples must be re-extracted and analyzed (greater dilution may be needed). Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.	Apply Q-flag and discuss in the Case Narrative only if reanalysis confirms failures in exactly the same manner.	Failing analytes shall be thoroughly documented in the Case Narrative. EIS should be 96% (or greater) purity. When the impurity consists of the unlabeled analyte, the EIS can result in a background artifact in every sample, standard and blank, if the EIS is fortified at excessive concentrations.
Method Blank (MB)	One per preparatory batch.	No analytes detected >½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, re-extract and reanalyze MB and all QC samples and field samples processed with the contaminated blank. Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	Blank spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-extract and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch. Not required for aqueous samples prepared by serial dilution instead of SPE.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch. For MD: Each aqueous sample prepared by serial dilution instead of SPE.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is ≥ LOQ. The MD is a second aliquot of the field sample that has been prepared by serial dilution.
Post Spike Sample	Only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of < LOQ for analyte(s).	Spike all analytes reported as < LOQ into the dilution that the result for that analyte is reported from. The spike must be at the LOQ concentration to be reported for this sample as < LOQ. When analyte concentrations are calculated as < LOQ, the post spike for that analyte must recover within 70-130% of its true value.	When analyte concentrations are calculated as < LOQ, and the spike recovery does not meet the acceptance criteria, the sample, sample duplicate, and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.	Flagging is not appropriate.	When analyte concentrations are calculated as < LOQ, results may not be reported without acceptable post spike recoveries.

Table B-16. Alpha Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) **(Energy, efficiency and FWHM peak resolution)**	Prior to initial use, following repair or loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.3)	Verify manufacturer's specifications for point source efficiency (MARLAP); and 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.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Use traceable calibration source (CS) that matches sample test source (STS) configuration (type, size and position relative to the detector). May use same count for initial efficiency calibration. No samples may be run until energy and FWHM calibration criteria are met.

Table B-16. Alpha Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	After initial calibration.	Determine peak location, resolution, and ROI/alpha peak efficiency (where counting efficiency is an analytical requirement) using at least two alpha peaks (MARLAP 18.5.6.3) or FWHM ≤ 100 keV and within ± 40 keV of corresponding calibration peaks in initial energy calibration.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate.	Flagging criteria are not appropriate.	Use a traceable second-source standard that matches STS configuration (type, size and position relative to the detector) or pulsar for energy check only. Bracketing peaks may also be used that are > 1000 keV apart. No samples may be run until calibration has been verified.
Continuing Calibration Verification (CCV) (Pulser check)	Pulser energy verification weekly, prior to analysis of samples. Use either Pulser check or Check source.	Energy response check shall have a tolerance limit set at ± 3% or control chart set at ± 3σ (MARLAP 18.5.6.3) or Observed peak centroid falls ≤ 20 keV from reference energy.	Check control chart for trends. Determine cause, correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	Pulser check can be used to verify energy calibration when using radiotracers during analysis. Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-16. Alpha Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV) (Check source)	Weekly source check verification prior to analysis of samples. Use either Pulser check or Check source.	Response checks shall have a tolerance limit or control chart set at ± 3% or 3σ (MARLAP 18.5.6.3) or FWHM ≤ 100 keV and within 40 keV of corresponding calibration peaks in initial energy calibration.	Check control chart for trends. Determine cause, correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	Source check can be used to verify energy, FWHM and efficiency. Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Background Subtraction Count (BSC) Measurement	Prior to initial use or after initial calibration and monthly. (MARLAP 18.5.6.3)	Use a statistical test to determine a change in the background count rate value (MARLAP 18.5.6.3) or 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 and reanalyze all impacted samples since last acceptable BSC.	If reanalysis cannot be performed, apply B-flag to all samples associated with the failed BSC.	BSC test source matches STS configuration (type, size, and position relative to the detector). Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-16. Alpha Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument Contamination Check (ICC)	Performed weekly, at minimum, and after counting high activity samples.	Use a statistical test to determine a change in the background count rate value or 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 and reanalyze all affected samples since last acceptable ICC.	If reanalysis cannot be performed, apply Q-flag to all results in all samples associated with the failed ICC.	Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-16. Alpha Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch. (MARLAP 18.4.1)	$\lvert Z_{Blank} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Blank} \rvert \geq 2$ (MARLAP 18.4.1) or In-house control limits of ± 3 σ of the mean.	Recount the blank to confirm results, unless all sample results are > 5 time the blank activity. Inspect MB control chart for indication of significant bias. If required, reprep and reanalyze Method Blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Unless required by method or program, Method Blanks are not used to correct sample activities, but only to monitor for contamination. (MARLAP 18.4.1) Blank matrices must be the same as the associated samples (i.e., radon-free distilled or deionized water, representative solid material, physically and chemically identical filter media).

Table B-16. Alpha Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	$\lvert Z_{LCS} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{LCS} \rvert \geq 2$ (MARLAP 18.4.3) or Use in-house control limits of LCS ± 3 σ of the mean. In-house control limits may not fall more than 25% from the known LCS value. Acceptance criteria for LCS recovery may be specified by the project.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. If required, reprep and reanalyze the LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	LCS matrices must be the same as the associated samples. LCS must be counted for a sufficient time to meet the required project minimum activity. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch. (*MS not required when chemical yield tracers or carriers are employed).*	If activity of the sample > 5 times the spiking level, then $\lvert Z_{MS} \rvert \leq 3$ (MARLAP 18.4.3) or Within 60-140% recovery.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-16. Alpha Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Duplicate	One per preparatory batch per matrix.	$\lvert Z_{Dup} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Dup} \rvert \geq 2$ (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is < 3; or the relative percent difference (RPD) is < 25%.	Check for lab error. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Tracers (if used)	Added to each sample as isotopic yield monitor.	Isotopic yield within 30-110%. FWHM < 100 keV and peak energy within ± 40 keV of known peak energy.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Carriers (if used)	Added to each sample as chemical yield monitor.	Chemical yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-17. Gamma Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) **(Energy, efficiency and FWHM peak resolution)**	Prior to initial use, following repair or loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.2)	Verify manufacturer's specifications for gamma peak resolution. (MARLAP 18.5.6.2) Efficiency vs. energy for each geometry/matrix. 95% confidence limit of the fitted function: ≤ 8% over energy range (MARLAP 18.5.6.2) or 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.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	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 peak in at least six calibration peaks that bracket the range of use. Low energy or thin-window gamma systems shall be calibrated to an energy range in accordance with manufacturer's specifications or data sheets. No samples may be run until all calibration criteria are met.

Table B-17. Gamma Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	After ICAL for energy/efficiency and prior to analysis of samples.	Observed peaks of second source standard fall within ± 10% of initial calibration value relative to energy, FWHM, and efficiency.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV as appropriate.	Flagging criteria are not appropriate.	Use a traceable second-source standard that matches STS configuration (type, size, geometry and position relative to the detector). Minimum of 10,000 net counts in each peak in at least six calibration verification peaks that bracket the range of use. No samples may be run until calibration has been verified.

Table B-17. Gamma Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV) (Daily Check)	Daily or prior to use. When working with long count times or batch sequences that run more than a day, CCV is performed at the beginning and end of each analytical batch as long as it is not longer than a week.	Response checks shall have a tolerance limit or control chart set at ± 3% or 3σ of the mean. (MARLAP 18.5.6.2) or <u>Peak Energy/Efficiency:</u> low, mid, and high energies within 10% of the initial calibration value; <u>FWHM:</u> low, mid, and high energies within 10% of initial FWHM value.	Check control chart for trends. Determine cause, correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-17. Gamma Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Background Subtraction Count Measurement (BSC) (Long count for subtracting background from blanks or test sources)	Immediately after ICAL and then performed on at least a monthly basis. (MARLAP 18.5.6.2)	Statistical test of successive counts and count rates for identified background peaks show no significant difference (MARLAP 18.5.6.2) or 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 and reanalyze all impacted samples since last acceptable BSC.	If reanalysis is not possible, apply B-flag to all results for specific nuclide(s) in all samples associated with the failed BSC.	A detector's background should be determined immediately after calibration, with or without a counting container, depending on the inherent radionuclide activity levels in the counting container. The counting interval for the long count shall be between one and four times the nominal counting interval of the test sources. (MARLAP 18.5.6.2) Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-17. Gamma Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument Contamination Check (ICC) **(Short count for controlling gross contamination)**	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); The tolerance limit or control chart: ± 3% or 3σ of the mean activity. (MARLAP 18.5.6.2)	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 and reanalyze all affected samples since last acceptable ICC.	If reanalysis cannot be performed, apply Q-flag to all results in all samples associated with the failed ICC	Integrate spectrum from ~50 – 2,000 keV to check for gross contamination. (MARLAP 18.5.6.2) Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-17. Gamma Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch. (MARLAP 18.4.1)	Z_{Blank} | ≤ 3. Investigate recurrent results with $\|Z_{Blank}\| \geq 2$ (MARLAP 18.4.1) or In-house control limits of ± 3 σ of the mean.	Recount the blank to confirm results unless all sample results are > 5 times the blank activity. Inspect MB control chart for indication of significant bias. If required, reprep and reanalyze Method Blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Unless required by method or program, Method Blanks are not used to correct sample activities, but only to monitor for contamination. (MARLAP 18.4.1) Blank matrices must be the same as the associated samples (i.e., radon- free distilled or deionized water, representative solid material, physically and chemically identical filter media.)

Table B-17. Gamma Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	$\lvert Z_{LCS} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{LCS} \rvert \geq 2$ (MARLAP 18.4.3) or Use in-house control chart limits of ± 3σ of the mean. In-house control limits may not fall more than 25% from the known LCS value. Acceptance criteria for LCS recovery may be specified by the project.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. If required, reprep and reanalyze the LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	LCS matrices must be the same as the associated samples and shall contain nuclides within the energy ranges of all those nuclides to be reported. LCS must be counted for a sufficient time to meet the required project minimum activity. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-17. Gamma Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Duplicate	One per preparatory batch per matrix.	$\lvert Z_{Dup} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Dup} \rvert \geq 2$ (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is < 3; or the relative percent difference (RPD) is < 25%.	Check for lab error. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration - <u>Voltage Plateau</u> (ICALV) **(Separate plateaus determined for alpha and beta activity)**	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1)	Verify manufacturer's specifications. (MARLAP 18.5.6.1) Plot voltage vs. count rate to determine proper operating voltages (MARLAP 18.5.6.1) or Slope of the plateau less than 5% over a range of 100V.	Correct problem, then repeat ICALV.	Flagging criteria are not appropriate.	Use a traceable calibration source (CS). Series of 1-minute counts in < 50V steps from ~300V to ~1500V. No samples may be run until plateau calibration criteria are met.
Initial Calibration – <u>Efficiency</u> (ICALE)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1)	Verify manufacturer's specifications for detector efficiency for both alpha and beta counting modes using electroplated sources. (MARLAP 18.5.6.1) A 1σ counting uncertainty of ≤ 1% shall be achieved for all detector efficiency determinations. (MARLAP 18.5.6.1)	Correct problem, then repeat ICALE.	Flagging criteria are not appropriate.	Use a traceable calibration source (CS). Detector's counting efficiency, using traceable calibration sources, shall be determined for each radionuclide used to analyze test sources. No samples may be run until efficiency calibration criteria are met.

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration – <u>Cross-Talk Factors</u> (ICALCT)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1)	Verify manufacturer's specifications for cross-talk in alpha and beta channels. (MARLAP 18.5.6.1)	Correct problem, then repeat ICALCT.	Flagging criteria are not appropriate.	Use a traceable calibration source (CS). Determine cross-talk factors for each nuclide, matrix and method. For mass loaded test sources, determine cross-talk factors for the nuclide as a function of test source mass. No samples may be run until cross-talk calibration criteria are met.

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration – <u>Self-Absorption Curve</u> (ICALSA)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1)	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 (MARLAP 18.5.6.1) or Best fit of data with correlation coefficient closest to 1.00 and the smallest standard error.	Correct problem, then repeat ICALSA.	Flagging criteria are not appropriate.	Use a traceable calibration source (CS). Minimum of seven mass attenuated standards. No samples may be run until mass attenuation calibration criteria are met.

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Efficiency Calibration Verification (IECV)	After ICALE for alpha and beta and prior to analysis of samples.	A tolerance limit or control chart shall be established immediately after the initial counting efficiency calibration and after instrument loss of control. A tolerance limit or control chart shall be set at ± 3% or 3σ of the mean (MARLAP 18.5.6.1) or Value of second source calibration for each isotope within ± 10% of initial calibration value.	Verify second source standard and repeat IECV to check for errors. If that fails, identify and correct problem and repeat IECV or ICALE and IECV, as appropriate.	Flagging criteria are not appropriate.	Use traceable second source standard that matches sample test source configuration (type, size, and position relative to the detector). No samples may be run until calibration has been verified.

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After a counting gas change and daily for short test-source counting intervals. For longer test-source counting times, a detector response check for a multi-sample shelf unit shall be conducted prior to test source counting, while a detector response check for a sequential sample counter shall be performed before and after the sample batch. (MARLAP 18.5.6.1)	Response checks shall have a tolerance limit or control chart limits ± 3% or 3σ of the mean. (MARLAP 18.5.6.1)	Check control chart for trends. Determine cause, correct problem, rerun calibration verification. If that fails, then repeat ICALE. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	Minimum of 2,000 net counts for each energy level. Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch. (MARLAP 18.4.1)	Z_{Blank} | ≤ 3. Investigate recurrent results with $\|Z_{Blank}\| \geq 2$ (MARLAP 18.4.1) or In-house control limits of ± 3 σ of the mean.	Recount the blank to confirm results, unless all sample results are > 5 times the blank activity. Inspect MB control chart for indication of significant bias. If required, reprep and reanalyze Method Blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Unless required by method or program, Method Blanks are not used to correct sample activities, but only to monitor for contamination. (MARLAP 18.4.1) Blank matrices must be the same as the associated samples (i.e., radon-free distilled or deionized water, representative solid material, physically and chemically identical filter media).

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Background Subtraction Count (BSC) Measurement (Long count for subtracting background from blanks or test sources)	Performed at least on a monthly basis. Determine alpha and beta background initially and after efficiency calibration. (MARLAP 18.5.6.1)	Use a statistical test to determine a change in the background count rate value. (MARLAP 18.5.6.4) or Within ± 3σ of mean activity of recent BSCs (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 and reanalyze all impacted samples since last acceptable BSC.	If reanalysis of samples is not possible, apply B-flag to all results in all samples associated with the failed BSC.	Detector background measured using a contamination-free source mount. Activity must meet project requirements. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Instrument Contamination Check (ICC) **(Short count for controlling gross contamination)**	Daily or when working with long count times, before and after each analytical batch. Check after counting high activity samples.	Use a statistical test to determine a change in the background count rate value (MARLAP 18.5.6.4) or 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. Reanalyze all affected samples since last acceptable ICC.	If reanalysis cannot be performed, apply Q-flag to all results in all samples associated with the failed ICC.	Develop detector response control chart immediately after calibration and loss of control. Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	$\lvert Z_{LCS} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{LCS} \rvert \geq 2$ (MARLAP 18.4.3) or Use in-house control chart limits of ± 3σ of the mean. In-house control limits may not fall more than 25% from the known LCS value. Acceptance criteria for LCS recovery may be specified by the project.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. If required, reprep and reanalyze the LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	LCS matrices must be the same as the associated samples. LCS must be counted for a sufficient time to meet the required project minimum activity. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch. (*MS not required when yield tracers are employed)*	If activity of the sample > 5 times the spiking level, then $\lvert Z_{MS} \rvert \leq 3$ (MARLAP 18.4.3) or Within 60-140% recovery.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-18. Gas Flow Proportional Counting

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Duplicate	One per preparatory batch per matrix.	$\lvert Z_{Dup} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Dup} \rvert \geq 2$ (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is < 3; or the relative percent difference (RPD) is < 25%.	Check for lab error. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Tracers (if used)	Added to each sample as yield monitor.	Isotopic yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Carriers (if used)	Added to each sample as chemical yield monitor.	Chemical yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-19. Liquid Scintillation Counter Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) **(Efficiency, ROI)**	Prior to initial use, following repair or loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.4)	Verify manufacturer's specifications for counting efficiency. (MARLAP 18.5.6.4) Establish energy ROIs for nuclides of interest. (MARLAP 18.5.6.4)	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Use a traceable calibration source (CS) radionuclide sources, typically unquenched LS cocktails tagged with ^{3}H and/or ^{14}C. No samples may be run until efficiency calibration criteria are met and energy ROIs are established for radionuclides of interest.

Table B-19. Liquid Scintillation Counter Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Calibration (QCAL) **(Quench curve)**	After each ICAL. Prior to method application, matrix, and cocktail changes or if control of system cannot be re-established or demonstrated. (MARLAP 18.5.6.4) .	A mathematical function and quench curve shall be developed so that the 95 percent confidence limit of the function is ≤ 5% over the expected quench range of the sources. Individual calibration sources shall be counted to achieve ROI measurement uncertainty of ≤ 1% (MARLAP 18.5.6.4) or Minimum 10,000 counts for each data point. Correlation coefficient for quench curve is > 0.995.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Use a traceable calibration source (CS). When establishing a quench curve, a minimum of five calibration sources of different quench factors shall be used. No samples may be run until all calibration criteria are met.

Table B-19. Liquid Scintillation Counter Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Calibration (QCAL) **(Standard Addition)**	Once after each ICAL.	Statistically evaluate replicate test-source analyses (MARLAP 18.5.6.4) or The duplicate error ratio (DER) between the sample and the duplicate is < 3; or the relative percent difference (RPD) is < 25%.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Use a traceable calibration source (CS). Add a spike to a duplicate processed sample or add a spike to a sample that has been counted and then recount. No samples may be run until calibration (Standard Addition) has been verified.
Initial Calibration Verification (ICV)	Once after each ICAL.	Value of each second source nuclide ± 10% of initial calibration value.	Verify second source standard and repeat ICV to check for errors. If that fails, correct problem and repeat ICV or ICAL and ICV as appropriate.	Flagging criteria are not appropriate.	Use a traceable second source standard for each nuclide. No samples may be run until calibration has been verified.

Table B-19. Liquid Scintillation Counter Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Counting efficiency performance check performed on day-of-use basis. Prior to use for short counting intervals. Before and after a test source batch for longer counting intervals. (MARLAP 18.5.6.4) For batch sequences that run more than a day, performance check is performed at the beginning and end of the batch, as long as it is not longer than a week.	Response checks shall have a tolerance limit or control chart set at ± 3% or 3σ of the mean. (MARLAP 18.5.6.4)	Check control chart for trends. Determine cause, correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	ROI for unquenched reference standards (typically 3H and/or 14C). Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-19. Liquid Scintillation Counter Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch. (MARLAP 18.4.1)	$\lvert Z_{Blank} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Blank} \rvert \geq 2$ (MARLAP 18.4.1) or In-house control limits of ± 3σ of the mean.	Recount the blank to confirm results, unless all sample results are > 5 times the blank activity. Inspect MB control chart for indication of significant bias. If required, reprep and reanalyze Method Blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Unless required by method or program, Method Blanks are not used to correct sample activities, but only to monitor for contamination. (MARLAP 18.4.1) Blank matrices must be the same as the associated samples (i.e., radon-free distilled or deionized water, or representative of the material media).

Table B-19. Liquid Scintillation Counter Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Background Subtraction Count (BSC) Measurement (Unquenched Blank; applicable when MSA is used)	Prior to initial use and monthly. (MARLAP 18.5.6.4)	Use a statistical test to determine a change in the unquenched background ROI count rate value (MARLAP 18.5.6.4) or 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 and reanalyze all impacted samples since last acceptable BSC.	If reanalysis cannot be performed, apply B-flag to all results for specific nuclide(s) in all samples associated with the failed BSC.	Unquenched sealed background vial not used for background subtraction. Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Background Measurement (MBM) (Quenched Blank)	Each batch. (MARLAP 18.5.6.4)	Use a statistical test to determine a change in the quenched background ROI count rate value (MARLAP 18.5.6.4) or Within ± 3σ of mean activity of recent MBMs for each ROI to be determined (minimum of 3 MBMs).	Check control chart for trends and recount. Determine cause, correct problem. If background activity has changed, re-establish MBM and reanalyze all impacted samples since last acceptable MBM.	If reanalysis cannot be performed, apply B-flag where count rate < 10 times that in the affected ROI(s) associated with the failed MBM.	MBM test source matches STS configuration (type, size and position relative to the detector). Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-19. Liquid Scintillation Counter Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	$\lvert Z_{LCS} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{LCS} \rvert \geq 2$ (MARLAP 18.4.3) or Use in-house control chart limits of ± 3σ of the mean. In-house control limits may not fall more than 25% from the known LCS value. Acceptance criteria for LCS recovery may be specified by the project.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. If required, reprep and reanalyze method LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	LCS matrices must be the same as the associated samples. LCS must be counted for a sufficient time to meet the required project minimum activity. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch. (*MS not required when yield tracers or carriers are employed)*	If activity of the sample > 5 times the spiking level, then $\lvert Z_{MS} \rvert \leq 3$ (MARLAP 18.4.3) or Within 60-140% recovery.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-19. Liquid Scintillation Counter Analysis

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Duplicate	One per preparatory batch per matrix.	$\lvert Z_{Dup} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Dup} \rvert \geq 2$ (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is < 3; or the relative percent difference (RPD) is < 25%.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Tracers	Added to each sample as yield monitor.	Isotopic yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Carriers	Added to each sample as chemical yield monitor.	Chemical yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) **(Efficiency)**	Prior to initial use, following repair or loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.3) Monthly for 6 months for new Lucas cells, annually thereafter per each cell/detector pair.	Verify manufacturer's specifications. (MARLAP) The operating voltage for the photomultiplier detector shall not exceed a slope of 2%/100V.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Use a traceable calibration source (CS). No samples may be run until calibration criteria are met.
Initial Calibration Verification (ICV)	After initial calibration.	Within ± 10% of initial calibration value.	Verify second source and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate.	Flagging criteria are not appropriate.	Use a traceable second-source standard. No samples may be run until calibration has been verified.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Monthly (or one in twenty). Per each cell/detector pair.	Response checks shall have a tolerance limit or control chart set at ± 3% or 3σ.	Check control chart for trends. Determine cause, correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable CCV. For data already reported, contact client to identify potential data quality issue.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Background Subtraction Count (BSC) Measurement	After initial calibration or prior to use, with each cell/detector pair.	Use a statistical test to determine a change in the background count rate value or 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 and reanalyze all impacted samples since last acceptable BSC.	If reanalysis cannot be performed, apply B-flag to all results in all samples associated with the failed BSC.	Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument Contamination Check (ICC)	Prior to use for each cell/detector pair.	Use a statistical test to determine a change in the background count rate value or Within ± 3σ of mean activity of recent ICCs (minimum of 3 ICC 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. Reanalyze all affected samples since last acceptable ICC.	If reanalysis cannot be performed, apply Q-flag to all results in all samples associated with the failed ICC.	Activity must meet project objectives. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch. (MARLAP 18.4.1)	$\lvert Z_{Blank} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Blank} \rvert \geq 2$ (MARLAP 18.4.1) or In-house control limits of ± 3σ of the mean.	Recount the blank to confirm results, unless all sample results are > 5 time the blank activity. Inspect MB control chart for indication of significant bias. If required, reprep and reanalyze Method Blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Unless required by method or program, Method Blanks are not used to correct sample activities, but only to monitor for contamination. (MARLAP 18.4.1) Blank matrices must be the same as the associated samples (i.e., radon-free distilled or deionized water, representative solid material, physically and chemically identical filter media).

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	$\lvert Z_{LCS} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{LCS} \rvert \geq 2$ (MARLAP 18.4.3) or Use in-house control limits of LCS ± 3σ of the mean. In-house control limits may not fall more than 25% from the known LCS value. Acceptance criteria for LCS recovery may be specified by the project.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. If required, reprep and reanalyze the LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	LCS matrices must be the same as the associated samples. LCS must be counted for a sufficient time to meet the required project minimum activity. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch. (*MS not required when chemical yield tracers or carriers are employed).*	If activity of the sample > 5 times the spiking level, then $\lvert Z_{MS} \rvert \leq 3$. (MARLAP 18.4.3) or Within 60-140% recovery.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Duplicate	One per preparatory batch per matrix.	$\lvert Z_{Dup} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Dup} \rvert \geq 2$ (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is < 3; or the relative percent difference (RPD) is < 25%.	Check for lab error. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Tracers (if used)	Added to each sample as isotopic yield monitor.	Isotopic yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Carriers (if used)	Added to each sample as chemical yield monitor.	Chemical yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-21. GC/MS Analysis of Air Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Canister Cleanliness Check	Each canister prior to shipment.	No reported analytes detected > ½ LOQ.	Correct problem, then repeat cleaning of canister and recertify.	Flagging is not appropriate.	Applicable only when laboratory supplies sampling canisters to client. For accreditation purposes, the laboratory must have the ability (procedures in place) to certify each canister. If the project uses the DoD/DOE QSM to define their requirements, then individual cans must be certified. Batch certification is only permitted if that is what the project requests.
Tune Check	Prior to ICAL and prior to each 24-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.

Table B-21. GC/MS Analysis of Air Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	At instrument set-up, prior to sample analysis.	Calculated RSD for the RRF of each target analyte in the calibration must be less than 30%; or Linear least squares regression for each analyte: $r^2 \geq 0.99$; or Non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic with one calibration point at the same concentration as the daily CCV. The lowest calibration standard concentration at or below the LOQ. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte.

Table B-21. GC/MS Analysis of Air Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units of the mean RRT of the calibration standards. RRTs may be updated based on the daily CCV.	Correct problem, then rerun ICAL.	NA.	RRTs shall be compared with the most recently updated RRTs. After maintenance is performed which may affect retention times, RRTs may be updated based on the daily CCV.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 30% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-21. GC/MS Analysis of Air Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 24 hours of analysis time; and at the end of the analytical batch run.	Concentration the same as the mid-point calibration standard (or lower). All reported analytes within ± 30% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) until a passing CCV is attained, and then reanalyze all associated samples since last acceptable CCV. Alternatively, perform an ICAL (including appropriate instrument QC) if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs), these additional requirements must also be met.

Table B-21. GC/MS Analysis of Air Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, standards, blanks, and QC sample.	**ICAL Standards** The area response for each internal standard must be within 40% of the mean area response of the calibration standards for each internal standard. The retention time shift for each internal standard at each calibration level must be within 20 seconds of the mean retention time of the calibration standards for each internal standard. **Field Samples, Blanks & QC** RT of each IS must be within +/-0.33 minutes of the most recent initial calibration.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	Must include at a minimum bromochloromethane, Chlorobenzene-d5, and 1, 4-difluorobenzene.

Table B-21. GC/MS Analysis of Air Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per analytical batch (after the first CCV), prior to analysis of any field samples.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Conduct investigation to determine the source of the contamination and take appropriate corrective actions. Rerun MB and all associated samples. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated analytical batch. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	The MB is a certified clean canister of the same type as the sample that has not left the laboratory. The blank has been pressurized with humidified zero air and carried through the same analytical procedures as a field sample. Results may not be reported without a valid MB. If a TIC search is required for any samples, then a TIC search is also required for the associated Method Blank.

Table B-21. GC/MS Analysis of Air Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)/ LCS Replicate	One pair per analytical batch.	One canister standard is analyzed two times in the analytical run. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD between the LCS and LCS Replicate for each target analyte must be < 30%.	Correct problem, and then reanalyze the LCS and all samples in the associated analytical batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated analytical batch.	Must contain all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the daily CCV is prepared in a canister, then the initial and closing CCVs can serve as the LCS and LCS Replicate as well. Both the CCV and LCS acceptance criteria would apply.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of DFTPP from method 8270. Tune check can be acquired as a full scan.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune. In addition to the full scan tune check, optimization for the analytes of interest is recommended.
Deuterated Monitoring Compounds (DMCs) (surrogates)	All field and QC samples.	PAH analysis: DMCs required for polyaromatic hydrocarbon (PAH) target analytes: fluoranthene-d_{10} and 2-methylnaphthalene-d_{10}. Minimum RRF for PAH DMCs: 0.40. All DMCs: Requires 50-150% recovery until in-house limits can be established.	Correct problem, and then reprep and reanalyze all samples with failing DMCs if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated samples and analytes if acceptance criteria are not met and explain in the Case Narrative.	For non-PAH target analytes, other DMCs with similar chemistry must be assigned. Laboratories may use the same extract for full scan and SIM analysis if the SIM-specific DMCs are added prior to extraction.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Performance Checks	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until the performance checks are within criteria. DDT breakdown and tailing factors are considered overall measures of port inertness and column performance and are required checks for SIM operation. DDT breakdown and tailing factor checks can be acquired as a full scan.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	At instrument set-up, prior to sample analysis.	Each analyte must meet one of the following options: RSD for each analyte ≤ 20% [If pentachlorophenol is a target analyte, an RSD of ≤ 40% allowed] or Linear least squares regression for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels required for ICAL with one calibration point at the same concentration as the daily CCV. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units of the mean RRT of the calibration standards. RRTs may be updated based on the daily CCV.	Correct problem, then rerun ICAL.	NA.	RRTs shall be compared with the most recently updated RRTs. Characteristic ions must maximize in the same scan or within one scan of each other. After any maintenance is performed which could affect retention times, RRTs may be updated based on the daily CCV.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value. If pentachlorophenol is a target analyte, a %D from true value of ± 50% is allowed.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	Concentration the same as the mid-point calibration standard (or lower). All reported analytes within ± 20% of true value. If pentachlorophenol is a target analyte, a %D from true value of ± 50% is allowed. All reported analytes within ± 50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) until a passing CCV is attained, and then reanalyze all associated samples since last acceptable CCV. Alternatively, perform an ICAL (including appropriate instrument QC) if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs), these additional requirements must also be met.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, Standards, blanks, and QC sample.	Retention time within ± 10 seconds from retention time 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 initial CCV is used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	Internal Standard is spiked no greater than 0.40 ng/µL concentration. According to the EPA Contract Laboratory Program Statement of Work (CLP SOW), this is the concentration of internal standard specified for SIM analysis. The SOW indicates calibration standards range from 0.10 to 1.0 ng/µL, so 0.40 ng/µL is mid- range. 1, 4-dichlorobenzene-d4 is ignored for SIM.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparation batch, prior to analysis of any field samples.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Conduct investigation to determine the source of the contamination and take appropriate corrective actions. Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated analytical batch.	Laboratories may use the same extract for full scan and SIM analysis provided the applicable DMCs and IS are spiked at the appropriate concentrations. Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparation batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, and then reanalyze the LCS and all samples in the associated analytical batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated analytical batch.	Must contain all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparation batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparation batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 40% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative.	The MSD must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. All data must be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Characteristic ions for MS confirmation	Minimum 3 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 must be > 0. Confirmation requires S/N ratio of ≥ 3 for each quant and confirmation ion.	No data can be reported without MS confirmation.	NA.	Need 3 structurally significant ions that are logical fragments – not isotopic clusters. Internal standard and DMC can use fewer than 3 ions.

Table B-23. Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples Other than Explosives

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Preparation Processing	Each required ISM sample, PT, LCS, and Blank. Entire sample must be dried, ground, and sieved unless otherwise stipulated in the QAPP.	Refer to method specific tables for blank and other QC acceptance criteria. Where method specific tables require the use of DoD/DOE QSM Appendix C limits, those limits are not applicable to analysis after preparation using table B-23 and laboratories shall develop in-house acceptance criteria. Laboratories shall develop in-house surrogate acceptance criteria.	Refer to method specific tables for respective corrective actions.	Refer to method specific tables for respective flagging criteria.	Surrogates are added prior to sample preparation procedures. Holding times begin with the collection of the first increment. At least one Grinding Blank per batch must be processed after the LCS or after a client identified sample with known contamination, or at the end of the batch. A Grinding Blank using clean solid matrix (such as Ottawa sand) must be prepared (e.g., ground and subsampled) and analyzed in the same manner as an ISM sample. Do not include vegetation or debris in the portion of the sample that passes through the sieve unless that is a project specific requirement.

Table B-23. Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples Other than Explosives

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Grinding	Initial demonstration at start up and any time major equipment is changed or when a reduction in the number or time of grinding cycles occurs. Each required ISM sample, PT, LCS, and Blank.	Grind entire sample that passes through the 10 mesh sieve such that final particle size is <75 µm. The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to <75 µm by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA	Flagging is not appropriate.	Grinding and sieving is an iterative process, so cycles and duration can be varied to reduce heat if all ISM and QC samples are treated the same. Grinding may introduce a bias and is not recommended for certain compounds. Each ISM sample, PT, LCS, and Grinding Blank must use the same grinding process (i.e., same time intervals and number of grinding cycles).
Subsampling	Each ISM sample, LCS, blank and Matrix Spike sample. All ISM sample types must be incrementally subsampled after processing.	Entire sample is mixed and spread out evenly on a large flat surface (e.g., baking tray), and 30 or more randomly located increments are removed from the entire depth and breadth to obtain the appropriate subsample size.	NA	Flagging is not appropriate	The total subsample weight collected can vary based on the requirements of the respective digestion/extraction process.

Table B-23. Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples Other than Explosives

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
ISM laboratory replicates	At the subsampling step, performed on one ISM sample per batch. Cannot be performed on any sample identified as a blank (e.g., Field Blank, Method Blank, Grinding Blank) or other QC.	For results above the LOQ, RSD or RPD must not exceed 20%.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	If reported per the client, apply J-flag to all samples within that batch if acceptance criteria are not met and explain in the Case Narrative.	Sample selection for performing replicates is based upon samples expected to contain the highest concentrations or as specified for the project. Percent RSD can be used for 3 or more replicates, otherwise use RPD or other appropriate metric. Field replicates do not replace the requirement for the laboratory to generate laboratory duplicates during subsampling.

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) ± 3 sample standard deviations.

An addendum to the 2012 study was conducted in the fall of 2018 to establish control limits for PFAS compounds for analysis by LCMSMS Compliant with DoD/DOE QSM 5.1 Table B-15. This LCS study incorporated the contributions from 11 DoD ELAP and DOECAP-AP accredited/approved laboratories. In all, over 49 thousand records were analyzed, and LCS limits were set for approximately 49 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 across at least 4 laboratories) for having sufficient data. Control limits were calculated as the sample mean (percent recovery) ± 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

Table C-1. Method 1668 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-2. Method 1668 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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
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
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

Table C-4. Method 6010 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-5. Method 6020 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-6. Method 6020 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 (MOD) 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 (MOD) 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
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

Table C-19. Method 8141 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-20. Method 8141 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-21. Method 8151 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-24. Method 8260 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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
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

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-26. Method 8270 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 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
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

Table C-27. Method 8270 SIM Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 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

Table C-28. Method 8270 SIM Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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
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
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

Table C-32. Method 8310 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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 Solid Matrix

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

Table C-35. Method 8330 Solid Matrix

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 - 8330B Series 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
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

Table C-36. Method 8330 - 8330B Series Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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

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
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

Table C-41. Method 9056 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

Table C-43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
74-83-9	Bromomethane	2657	98.6	11.8	63	134
106-97-8	Butane	587	96.2	10.9	64	129

Table C-43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
91-20-3	Naphthalene	2439	97.5	13.4	57	138
111-84-2	Nonane	2617	95.4	10.8	63	128

Table C-43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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

Table C-44. Method PFAS by LCMSMS Compliant with QSM Table B-15 Aqueous Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2991-50-6	2-(N-Ethylperfluorooctanesulfonamido) acetic acid	1210	97.9	12.2	61	135
2355-31-9	2-(N-Methylperfluorooctanesulfonamido) acetic acid	1219	100.9	11.7	65	136
757124-72-4	Fluorotelomer sulphonic acid 4:2	789	103.2	13.2	63	143

27619-97-2	Fluorotelomer sulphonic acid 6:2	1673	102.4	12.5	64	140
39108-34-4	Fluorotelomer sulphonic acid 8:2	1657	102.3	11.6	67	138
31506-32-8	N-methylperfluorooctanesulfonamide	404	104.1	12.0	68	141
375-73-5	Perfluorobutanesulfonic acid	1832	100.8	9.4	72	130
375-22-4	Perfluorobutanoic acid	1270	100.6	9.2	73	129
335-77-3	Perfluorodecanesulfonic acid	1361	97.7	14.8	53	142
335-76-2	Perfluorodecanoic acid	1722	100.4	9.5	71	129
307-55-1	Perfluorododecanoic acid	1714	102.8	10.2	72	134
375-92-8	Perfluoroheptanesulfonic acid	1552	101.9	10.7	69	134
375-85-9	Perfluoroheptanoic acid	1837	101.4	9.5	72	130
355-46-4	Perfluorohexanesulfonic acid	1849	99.7	10.3	68	131
307-24-4	Perfluorohexanoic acid	1797	100.4	9.4	72	129
68259-12-1	Perfluorononanesulfonic acid	780	97.7	9.5	69	127
375-95-1	Perfluorononanoic acid	1846	99.9	10.0	69	130
754-91-6	Perfluorooctanesulfonamide	1453	101.9	11.4	67	137
1763-23-1	Perfluorooctanesulfonic acid	1744	102.7	12.4	65	140
335-67-1	Perfluorooctanoic acid	1962	102.2	10.1	71	133
2706-91-4	Perfluoropentanesulfonic acid	812	99.0	9.2	71	127
2706-90-3	Perfluoropentanoic acid	1695	100.7	9.3	72	129
376-06-7	Perfluorotetradecanoic acid	1714	101.8	10.0	71	132
72629-94-8	Perfluorotridecanoic acid	1696	104.8	13.0	65	144
2058-94-8	Perfluoroundecanoic acid	1746	100.8	10.6	69	133

Table C-45. Method PFAS by LCMSMS Compliant with QSM Table B-15 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2991-50-6	2-(N-Ethylperfluorooctanesulfonamido) acetic acid	249	99.7	12.9	61	139
2355-31-9	2-(N-Methylperfluorooctanesulfonamido) acetic acid	254	103.9	13.3	63	144
757124-72-4	Fluorotelomer sulphonic acid 4:2	266	103.1	13.7	62	145
27619-97-2	Fluorotelomer sulphonic acid 6:2	575	101.9	12.4	64	140

Table C-45. Method PFAS by LCMSMS Compliant with QSM Table B-15 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
39108-34-4	Fluorotelomer sulphonic acid 8:2	544	100.8	11.8	65	137
375-73-5	Perfluorobutanesulfonic acid	624	100.5	9.2	72	128
375-22-4	Perfluorobutanoic acid	333	102.9	10.5	71	135
335-77-3	Perfluorodecanesulfonic acid	336	96.2	12.4	59	134
335-76-2	Perfluorodecanoic acid	569	101.0	10.4	69	133
307-55-1	Perfluorododecanoic acid	565	101.7	10.8	69	135
375-92-8	Perfluoroheptanesulfonic acid	511	101.0	10.3	70	132
375-85-9	Perfluoroheptanoic acid	652	101.2	10.0	71	131
355-46-4	Perfluorohexanesulfonic acid	639	98.5	10.5	67	130
307-24-4	Perfluorohexanoic acid	614	100.8	10.2	70	132
68259-12-1	Perfluorononanesulfonic acid	338	96.8	9.1	69	125
375-95-1	Perfluorononanoic acid	650	100.3	9.3	72	129
754-91-6	Perfluorooctanesulfonamide	377	102.2	11.5	67	137
1763-23-1	Perfluorooctanesulfonic acid	518	101.9	11.3	68	136
335-67-1	Perfluorooctanoic acid	663	101.1	10.4	69	133
2706-91-4	Perfluoropentanesulfonic acid	335	97.9	8.1	73	123
2706-90-3	Perfluoropentanoic acid	588	100.2	10.3	69	132
376-06-7	Perfluorotetradecanoic acid	551	101.3	10.5	69	133
72629-94-8	Perfluorotridecanoic acid	548	102.3	12.1	66	139
2058-94-8	Perfluoroundecanoic acid	587	99.9	12.0	64	136

Appendix D: Non-Destructive Assay (NDA)

This appendix addresses quality assurance and control measures to be implemented by the NDA measurement organization. There are two subsections: one concerning quality assurance requirements that must be performed and documented, and another addressing quality control measures with criteria for acceptable performance and associated action limits.

1.0 QUALITY ASSURANCE

1.1 NDA System Calibration

This section delineates requirements for establishment of a traceable NDA measurement system "initial calibration," confirmation of the "initial calibration" and the continuing verification of such. Procedures shall be developed and implemented for NDA measurement system calibration methods and processes. Per the purpose of this Appendix, the term calibration is referred to and defined in three separate ways: 1) initial calibration, 2) calibration confirmation, and 3) calibration verification.

The "initial calibration" is that fundamental calibration that addresses and accounts for the response of a NDA measurement system to radioactive materials present in the waste containers or process components of interest (measurement items). The "calibration confirmation" is a thorough corroboration of the "initial calibration" using traceable working reference materials (WRMs) and representative waste matrix/ process component configurations. The "calibration verification" is a periodic verification of the "initial calibration" to ensure on-going long-term data quality compliance through the period of NDA operations.

Procedural steps for calibration are not specified here. However, those elements that must be considered during the "initial calibration," "calibration confirmation," and "calibration verification" are enumerated. This allows the NDA measurement organization autonomy in devising and implementing techniques and analytical procedures for these three calibration definitions. Through these three mechanisms, the NDA measurement organization shall demonstrate the calibration and associated uncertainty is compliant with applicable client and/or end-user requirements initially and throughout the contract period.

1.1.1 Initial NDA System Calibration

A NDA measurement system "initial calibration" shall be performed to ensure the measurement system response provides valid data of known and documented quality. Calibrations shall be performed using traceable WRMs obtained from suppliers maintaining a nationally recognized

reference base and an accredited measurement program. Full documentation of the calibration technique, process, and results is required. For cases where there is an insufficient number and denomination of traceable radioactive material standards to support the "initial calibration," the NDA organization can develop alternate calibration strategies based on available resources. Alternate strategies shall be clearly documented and technically justifiable.

The development and establishment of an "initial calibration" shall address the following as applicable:

a) SOPs shall be in place to specify steps/activities necessary to develop and determine the "initial calibration" including, but not limited to, specification of traceable radioactive sources or their alternates, geometrical positioning of sources, traceable source/matrix media configurations, acquisition of NDA system response data, computational methods, analysis of response data to determine a robust calibration, calibration acceptance criteria, calibration applicability and qualifiers, and calibration uncertainty.

b) The "initial calibration" shall be performed through the use of traceable working reference materials unless exceptions have been stipulated and documented. For mass calibrations (i.e., calibrations that use a direct measurement of the same isotopes, matrices, and containers that will subsequently be measured in unknown items), the radioactive material mass and matrix characteristics must span and bracket the range of anticipated values for the measurement items. For calibrations based on instrument response modeling, sufficient information shall be provided in the method description and calibration regimen to assure that the calibration measurements and model appropriately spans and brackets the anticipated analysis space (e.g., provide mechanisms to account for anticipated geometries, radioactive material mass, chemical composition, and matrix characteristics). For enrichment determinations using the enrichment meter technique, the initial calibration must span the range of enrichments in anticipated unknown item measurements.

c) The measurement uncertainty associated with the application of the "initial calibration" shall be established using a sound and technically defensible technique. Methods for the estimation of total measurement uncertainty (TMU) shall be developed and documented. Where applicable, the calibration uncertainty shall include terms for mass, matrix characteristics and configurations, and radioactive material properties. These methods shall consider, at a minimum, uncertainty components, the calibration uncertainty model (method of uncertainty component propagation), estimates of uncertainty introduced by differences between item characteristics, and calibration modeling assumptions. For example, if the model assumes a homogeneous distribution of the isotope of interest, the uncertainty introduced if items are not homogeneous using a worst case distribution, as determined through a documented engineering judgment including supporting data, must be determined.

d) The NDA measurement method capability related to each initial calibration must be defined and documented. As applicable, this capability includes waste matrix types, process equipment types, geometries, configurations, radioactive material types, matrix density range, hydrogenous material range, radioactive material mass range, radioactive material compound, and other parameters affecting instrument response. The intent of defining the capability is to delineate those source/matrix configurations where the calibration is applicable and where it is not.

e) Where surrogate materials are used to simulate waste matrices, their configuration(s) must be nominally representative of the actual waste item population. The design of surrogate matrix configurations must be documented. Surrogate materials used to produce a given matrix configuration shall be carefully specified, procured and the resultant physical properties and configuration documented.

f) If NDA method manuals, national standards, or a mandated NDA calibration methods do not specify the number of traceable WRMs to span the mass/activity and radioactive material compound(s) characteristics of the waste/process component, a minimum number must be determined and technically justified. NDA organization must document this number and their denominations in a calibration SOP or other applicable document. This requirement does not necessarily apply to NDA methods that rely on modeling. However, the method used to assure that the calibration and model appropriately spans and brackets the anticipated analysis space (e.g., provide mechanisms to account for anticipated geometries, radioactive material mass, chemical composition, and matrix characteristics) as per item (b) above must be technically justified and documented. For NDA methods that do not necessarily require calibration with source material similar in nature to the waste or process items (e.g., neutron counting), those source(s) used are still required to be traceable. However, accounting of the efficiency variation because of the composition of the actual radioactive material shall be assessed and corrected for (e.g., Californium (^{252}Cf) fission neutron spectrum counter efficiency versus uranyl fluoride (UO_2F_2) neutron spectrum efficiency).

g) The “initial calibration” process shall be clearly documented including the calibration measurement configurations, data acquisition parameters, acquired data, data reduction methods, resultant calibration factors or expressions, statistical analyses, and uncertainties. Records containing information pertinent to the calibration process shall be retained including but not limited to:

   i) WRM and/or surrogate waste matrix configurations used to acquire instrument response data, calibration determination techniques;
   ii) SOP(s) used;

   iii) data acquisition parameters;

iv) NDA system identification;

v) analytical software used;

vi) traceable standard identifications;

vii) analytical support equipment information; and

viii) electronic file storage locations.

Records shall be sufficient to allow reproduction of the “initial calibration.”

h) The “initial calibration” shall be re-established when repairs or changes are made to the measurement system that are likely to affect one or more calibration parameters.

Examples that may require repeating the “initial calibration” include, but not are limited to:

i) major NDA system repairs or modifications;

ii) replacement of vital NDA measurement system components (e.g., collimator, multi-channel analyzer (MCA), neutron generator);

iii) change in collimator depth and/or aperture not accounted for in a model; and

iv) significant software modification and/or changes.

1.1.2 Calibration Confirmation

A confirmation of the “initial” NDA measurement system calibration shall be performed. In this context, confirmation means the “initial calibration” shall be assessed and determined to be correct and true by the objective collection of evidence supporting the calibration was properly established.

a) The “calibration confirmation” process is to produce objective evidence demonstrating the applicability and correctness of the “initial calibration” relative to the waste forms and process components of interest. The recommended method is to assemble test item(s) consisting of traceable source/matrix configuration(s) nominally representative of the waste form and/or process components to be characterized. They cannot be the same configurations used to establish the “initial calibration.” They must contain a known and traceable radioactive element/isotope, mass/activity, and/or enrichment in a known and representative matrix configuration. The confirmation test item(s) are then measured using the “initial calibration” of the NDA system. The number of differing test item configurations used to confirm the calibration is to be determined by the NDA

organization and documented. The reported "calibration confirmation" measurement result must agree, with criteria as established by the NDA organization, with the known element/isotope, mass/activity and/or enrichment of the confirmation test item(s). The NDA organization acceptance criteria shall not exceed the criteria as presented in Section 1.1.3 unless technically justified and documented.

b) The radioactive sources used for "calibration confirmation" purposes shall, to the extent practicable, be representative of the actual radioactive material compositions and chemical compounds as found in the measurement item inventory of interest.

c) Radioactive material standards used for "calibration confirmation" are to be traceable to a nationally recognized reference base (e.g., National Institute of Standards and Technology [NIST] or New Brunswick Laboratory [NBL]). The traceable standards used for "calibration confirmation" shall not be related to (from the same feedstock or lineage) those used to perform the "initial calibration." Noncompliance with this requirement, due to lack of a sufficient variety of traceable standards, can be temporarily waived provided an adequate alternate confirmation strategy is devised.

d) "Calibration confirmation" acceptance is assessed through the degree of agreement between the known "calibration confirmation" test item value and that as per the NDA confirmation measurement result. The NDA organization is to determine and document representative "calibration confirmation" source/matrix surrogate configuration(s). The NDA organization may also develop "calibration confirmation" bias and precision acceptance criteria specific to the NDA system and measurement items under consideration. Recommended "calibration confirmation" acceptance criteria are delineated in Section 1.1.3.

e) "Calibration confirmation" results outside NDA organization defined acceptance criteria require implementation of corrective action(s) as applicable. Calibration confirmation results are not to exceed the maximum allowable acceptance criteria of Section 1.1.3 unless the NDA organization has specifically determined and documented greater limits with the requisite technical justification.

f) For the case where a corrective action was required and subsequently implemented, the "calibration confirmation" process is to be repeated. Acceptable results must be obtained and documented before the NDA system is considered operational. Where a "calibration confirmation" failure was determined to be due to a minor issue (e.g., wrong constant, wrong efficiency file, or an inappropriate test item), the entire "calibration confirmation" measurement regimen may not need to be repeated. This is acceptable provided it is the true cause of the failure. All corrective actions and their effects, supporting data, results, etc., shall be documented and retained.

g) In the case where the “calibration confirmation” was acceptable for certain types or categories of radioactive material/waste matrix configurations, but unacceptable for other categories with distinctly different source/matrix properties, conditional acceptance of the “calibration confirmation” can be made. The NDA organization, however, must clearly identify which categories of source/matrix configurations are approved for NDA measurement and which are not. The technical basis for accepting certain source/matrix categories shall be documented and available for review. Recalibration or corrective action efforts should be implemented and documented for source/matrix categories not meeting acceptance criteria for “calibration confirmation.”

h) The “calibration confirmation” process shall be performed following an “initial calibration” or when there are indications warranting a re-assessment of the “initial calibration,” (e.g., the source/matrix configuration of measurement items varies relative to the source/matrix configurations used to develop the “initial calibration.”) Additional causes for a performing a “calibration confirmation” include:

   i) major NDA system repairs or modifications,

   ii) replacement of NDA measurement system components, e.g., detector, neutron generator or supporting electronic components that have the potential to affect data quality,

   iii) re-calibration,

   iv) significant changes to the NDA system software, and

   v) relocation of the system (applies primarily to fixed stationary systems).

i) Records must be retained to permit reconstruction of any NDA measurement system “calibration confirmation”(e.g., NDA method, measurement system configuration, confirmation date, primary radioactive isotope(s), mass or concentration and response, calibration factor(s), or equations/coefficients used to convert NDA instrument response to mass/concentration). Documentation must explicitly connect the “calibration confirmation” data/records to the “initial calibration.”

1.1.3 Calibration Confirmation Acceptance Criteria

a) Bias and precision limits are used to determine the acceptability of “calibration confirmation” measurements. The specified limits should be “upper limits” to be applied to all NDA measurement techniques over all matrix configurations. The recommended “calibration confirmation” limits are not specifically tied to end-user requirements, rather they are nominal performance levels expected of NDA systems. Failure to comply with

these bias and precision limits is used as an indicator that more capable measurement techniques need to be developed.

b) NDA measurement system bias and precision should be determined through the acquisition of replicate measurements using matrix container and/or process component mock-ups combined with traceable WRMs. The source/matrix configurations are to be representative of the actual measurement item population of interest. The number of different source/matrix test configurations and replicate measurements of each shall be determined by the NDA organization and documented. The "calibration confirmation" bias is to be determined in terms of %Bias [(mean measured value - known value)/known value]*100 or %R (mean measured value/known value)*100. The bias shall not be outside the limits as per Table D-1 at the 95% confidence level.

c) Precision is reported as percent relative standard deviation (RSD). The RSD shall not exceed the value listed in the last row of Table D-1 for twenty replicate measurements of the "calibration confirmation" source/matrix test item(s). Equivalent RSD limits for a number of different replicate values are tabulated in Table D-2.

Table D-1. Calibration Confirmation Acceptance Limits

Confirmation Range	%Bias	%R
bias (lower limit)	-30	70
bias (upper limit)	30	130
precision	20% RSD at the 95% confidence level for 15 replicates	

Table D-2. Upper Limits for RSD vs. Number of Replicates

Number of Replicates	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Max RSD[a]	**1.8**	**6.6**	**10.0**	**12.3**	**14.0**	**15.2**	**16.2**	**17.1**	**17.7**	**18.3**	**18.8**	**19.3**	**19.7**	**20.0**

a – the values listed are derived from the measured standard deviation of the replicate measurements using:

$$\frac{s}{\mu} \times 100\% < \sqrt{\frac{(0.292)^2 \times \chi^2_{0.05,n-1}}{n-1}} \times 100\%$$

Where:

s = measured standard deviation

n = number of replicates

μ = known or true value

$\chi^2_{0.05,n-1}$ = critical value for the upper 5% tail of a one sided chi-squared distribution, with n-1 degrees of freedom,

0.292 constant corresponds to a 95% upper confidence bound on the true system precision limit of 29.2%.

d) NDA service providers may develop alternate methods and limits for bias and precision. Such alternate methods and limits must be technically defensible and clearly documented.
e) Failure to comply with the bias and precision requirements for "calibration confirmation" requires development of a corrective action plan (CAP). The CAP shall include detail on the nature of the failure, its suspected causes, methods to evaluate potential causes, and activities proposed to identify and rectify the deficiency. The CAP results shall be documented and show why the failure occurred and what actions were taken to prevent a re-occurrence. The "calibration confirmation" shall be performed again after the corrective actions in the CAP have been implemented and the results documented.

1.1.4 Calibration Verification

"Calibration verification" is a measure designed to provide continual and long-term information on the stability of the "initial calibration" while minimizing the impact on NDA operational schedules and resources. The "calibration verification" test item(s) must meet the bias acceptance criteria delineated in Section 1.1.3. A "calibration verification" shall be performed at least once every five operational days for each measurement system and calibration in use. A five day operational period is defined as a rolling tally of five days where NDA operations were in effect, not necessarily consecutive. The start point for the five day operational period is from the start of approved operations or the first operational day after the previous rolling five day tally was completed. The five day operational "calibration verification" requirement may be extended to a maximum of thirty operational days provided the NDA organization can demonstrate and technically justify the long term stability of the NDA system per established acceptance criteria.

Calibration verification test items are typically selected from or assembled from the traceable standards and matrix containers or process component mock-ups used in the "calibration confirmation" process. The "calibration verification" test item is to be submitted to NDA operations in a "blind" manner, where applicable, and processed through the measurement routine as though it were an actual measurement item. The "calibration verification" test items are to be selected and/or configured and submitted such that during a 12-month period the operational space of the NDA system "initial calibration" is spanned. The "calibration

verification" is a point check in the calibration realm. It is not required that each waste matrix type comprising the operational space of the NDA system be tested. However, it is expected that the "calibration verification" configurations vary over the operational space. The NDA organization is responsible for specification, assembly and selection of "calibration verification" test items, and meeting the applicable rolling operational day period (i.e., minimum five days, maximum thirty days).

Acceptable performance for a "calibration verification" measurement result in terms of bias, trending measures, and so forth shall be determined and documented by the NDA organization. It is recommended that the "calibration confirmation" acceptability requirements of Section 1.1.3 be considered in this process. A CAP for out-of-control "calibration verification" results is to be prepared by the NDA organization. The CAP shall include a provision requiring the evaluation of measurement item data potentially affected by the failed "calibration verification" measure. The "calibration verification" protocol, monitoring, acceptance criteria, action levels, etc. are to be clearly documented and readily available for review. The "calibration verification" data is to be control charted and monitored for trends over time.

The NDA organization can utilize other methods of "calibration verification" provided they are technically justifiable and documented.

1.2 NDA Method Detection Limit

A methodology shall be in place to determine NDA measurement system detection limit for those radionuclides specified per the client/end-user requirements. It shall be re-determined each time there is a significant change in the measurement method or matrix configuration. Instruments performing low-level waste discrimination measurements must have a minimum detectable activity (MDA)/lower limit of detection (LLD) sufficient to meet the acceptance criteria. The methodology for determination of the MDA/LLD is to be documented by the NDA organization.

The LLD is that level of radioactivity which, if present, yields a measured value greater than the critical level (Lc) with a 95% probability, where the Lc is defined as that value which measurements of the background will exceed with 5% probability (the LLD may be defined in a different manner to comply with specific client needs). Because the LLD is a measurement-based parameter, it is not feasible to calculate LLDs for radionuclides that are not determined primarily by measurement (e.g., ^{99}Tc). In such cases, the NDA organization shall derive the equivalent of an LLD (i.e., a reporting threshold for a radionuclide(s) when technically justified). This value may be based on decay kinetics, scaling factors, or other scientifically based relationships and must be adequately documented in site records.

The minimum detectable activity is that activity of an analyte in a sample that will be detected with a probability β of non-detection (Type II error) while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample

(Type I error). For the purposes of this document, the alpha (α) and beta (β) probabilities are both set at 0.05 unless otherwise specified.

1.3 Infinite Thickness

For a given radioactive material thickness (deposit or buildup), a thickness may be reached beyond which there is no increase in counts for an increase in thickness. At this point, infinite thickness has been reached. This phenomenon is typically only observed in gamma-ray counting. The NDA organization shall have a documented process for identifying infinite thickness when performing measurements.

Some common techniques include:

- Transmission Factor - ASTM C1133-89, 'Standard Test Method for NDA of Special Nuclear Material in Low Density Scrap and Waste by Segmented Passive Gamma-Ray Scanning,' ASTM, 1989.

- Peak ratio - Software such as Multi-Group Analysis for Uranium.

1.4 NDA Measurement Uncertainty

NDA organizations shall have and apply methods and procedures for estimating total measurement uncertainty (TMU) for all reported values. The NDA organization shall perform a preliminary identification of uncertainty components and produce measurement uncertainty estimates for the waste population to be characterized prior to generating characterization data for the client/end-user. An estimate of the measurement uncertainty for the measurement item inventory of interest is to be performed and documented. The estimate shall be based on knowledge of the measurement method performance and make use of previous experience and validation data from similar measurement apparatus and configurations when available. The estimated measurement uncertainties must be evaluated per client and/or end-user needs and requirements. The method used to calculate TMU for the purpose of demonstrating compliance with client and/or end-user requirements must be documented and technically justified.

The NDA organization shall have a method to determine total measurement uncertainty for each NDA system employed including:

a) Develop a document or plan that delineates the approach to TMU determination, defines measurement uncertainty components, and determines a method for acquiring data/information on components of variance and processing of acquired data and information to arrive at technically defensible TMU for the measurement item population of interest.

b) Procedure or applicable document that provides specific direction on the acquisition of NDA system measurement data for use in deriving the TMU.

c) Produce documentation that clearly describes the processing of acquired data, accounting for all significant variables, and the application of methods to determine the TMU.

d) Clearly define how the TMU is expressed (e.g., 95% confidence level, percent, one-sigma, etc.)

e) The TMU determination method must be clearly documented; NDA organizations that utilize commercial off-the-shelf data analysis and uncertainty software are still accountable to produce clear documentation of the TMU approach, components of variance, and technique for arriving at the TMU value.

1.5 NDA Measurement Traceability

The calibration of NDA instrumentation and support measurement devices (e.g., weight scale) used for NDA characterization purposes shall have traceable calibrations established and documented before being put into service. Traceability is the ability to relate individual measurement results through an unbroken chain of calibrations to a nationally recognized reference base (e.g., NIST, etc.). For NDA measurements, traceable materials include radioactive WRMs, certified weights for scale calibrations, and thickness measurement methods.

a) The NDA organization shall have a program and procedures for establishing a traceable calibration as well as QC checking of its NDA instrumentation and support equipment. This program shall include a system for selecting, procuring, using, and controlling traceable reference standards for NDA measurement instrumentation and support equipment. For cases where traceable working reference materials are not yet available, the NDA organization may propose alternate methods that are technically defensible and clearly documented.

b) Traceable sources used for calibration shall be traceable for all attributes used for the calibration (e.g., a ^{252}Cf source shall be certified in its neutron yield and isotopic composition used to calculate the decay rate, and a mixed nuclide source used to perform an efficiency calibration of a gamma-ray detector shall be certified for the yield of each gamma ray energy used in the calibration and the decay properties of the contributing nuclides).

c) The NDA organization shall have a procedure(s) for the specification, procurement and acceptance of WRMs. The WRM certifications shall be acquired and maintained, and traceable to a nationally recognized reference base (e.g., NIST, NBL).

d) The NDA service provider shall retain records for all WRMs including the manufacturer/vendor, the manufacturer's Certificate of Traceability, the date of receipt, and a certificate expiration date.

e) Traceable standards shall be verified at a minimum of every five years. Standards with an expiration date less than five years shall be verified at a period equal to the time expiration time interval. Verification of a standard is accomplished through an assessment of its usable attribute to the NDA application (e.g., ^{235}U 185.7 keV gamma-ray emission rate and neutron emission rate). The area number of means by which a standard can be deemed verified as acceptable for use.

   i) The standard can be sent to a qualified facility maintaining measurement systems traceable to a certified reference material (CRM) for a determination of the standard attribute of interest. In this case the standard is simply given an updated attribute value and returned to the NDA organization with a revised or new certificate.

   ii) Another method is to cross compare the standard with another traceable standard possessing the same attribute in a calibrated and operational measurement system. An evaluation of the results can produce a verification of the standard that is about to or has expired. The NDA organization must determine the acceptable uncertainty in the verified value relative to the NDA characterization process at hand.

   The verification method used and standard verification acceptability criteria shall be documented. The results of the verification are to be documented and maintained as a QA record.

f) WRM Certificates of Traceability shall contain information and data that clearly details traceability to a CRM.

g) Checks needed to maintain confidence in the status of WRMs shall be carried out according to defined procedures and schedules.

h) The NDA service provider shall have procedures for the safe handling, transport, storage, and use of WRMs in order to prevent contamination or deterioration and protect their integrity.

1.6 NDA Measurement System Software

Software quality assurance (SQA) requirements must be implemented by NDA organizations that utilize software as part of NDA waste characterization, developed in-house or acquired.

When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage, or retrieval of NDA measurement data, the NDA organization shall have documentation or SOPs for software related activities. This documentation includes but is not limited to the following as applicable:

a) For software acquired from a commercial vendor or other third party, evidence of software quality control (QC), verification and validation (V&V), and other pertinent data shall be acquired and maintained by the NDA organization. Software verification is the process of evaluating software to determine whether the products of a given development phase satisfy the conditions imposed at the start of that phase (IEEE-STD-610). Software validation is the process of evaluating software during or at the end of the development process to determine whether it satisfies specified requirements. (IEEE-STD-610)

b) For software developed or modified in-house by the NDA organization, software development planning and QA controls shall be identified in documented plans. The following activities shall be addressed in such plans/procedures:

    i) software development and testing;

    ii) software V&V;

    iii) software configuration control; and

    iv) software operation and maintenance.

c) Computer software developed by the NDA organization shall be documented per applicable software development quality standards. Such standards usually require documentation, including:

    i) software specification document;

    ii) software design document;

    iii) software test plan; and

    iv) software V&V document (Note: Commercial off-the-shelf software [e.g., word processing, database and statistical programs in general] used within its designed application range are usually considered to be sufficiently validated). However, NDA

organization developed software and/or modifications to commercial software must be validated. Installation and operability checks shall also be performed.

d) Software change procedures shall include requirements for the requesting, testing, quality assurance, approving, and implementation of changes.

e) Data, including, but not limited to decay constants, branching ratios, material attenuation values, neutron yields, and master gamma libraries used in the reduction of processing of NDA measurement data to a reportable quantity, whether electronic or hardcopy, shall be placed under a control system so only authorized individuals have access.

f) Working data or source files (e.g., nuclear data libraries, master gamma libraries, geometry files, and efficiency files) shall be controlled by the NDA organization to prevent unauthorized access or inadvertent changes and controlled to document changes by authorized users to allow for re-creatability of the data used.

g) When commercial software is used that has the capability of performing user-defined calculations or macros (e.g., spreadsheet), all user-defined components shall be verified before initial use and after changes. Documentation of such shall be readily available for review. Appropriate protections must be included to preclude inadvertent changes to user-defined equation or macros. Printouts from any spreadsheet should include that information used to calculate the result.

h) Software version control methods must be in place to document the software version currently used as well as data reports with the date and time of generation and the software version used to generate the data report. Software that includes user-defined calculations and/or macros shall also track revisions to the user-defined customization using version information.

i) Confidentiality of data entry or collection, data storage, data transmission and data processing.

j) Computers and automated equipment are to be maintained to ensure proper function and must have appropriate environmental and operating conditions necessary to maintain the integrity of NDA measurement data and information.

k) Procedures are to be established and implemented for the maintenance of security of data, including the prevention of unauthorized access to and the unauthorized amendment of computer records.

l) An inventory of all applicable software used to generate NDA characterization data shall be maintained that identifies the software name, version, classification, and exemption

status (DOE 0 414.C or latest version), operating environment, and the person and organization responsible for the software.

m) Maintain a historical file of software, software operating procedures, software changes, and software version numbers.

1.7 Acceptable Knowledge

NDA methods typically directly quantify one or more of the prevalent radionuclides known to be present in the waste and process component items. Other radionuclides may be present, some of which are not readily quantifiable through the NDA method being employed. NDA measurement campaigns often require that radionuclide not directly measureable by NDA methods be quantified and/or the minimum detectable activity determined and reported.

For radionuclides to be reported per contractual requirements, but not quantifiable through existing NDA techniques, isotopic ratios or radionuclide scaling factors based on acceptable knowledge (AK) of the facility process are commonly employed. The radionuclides and isotopes that are quantifiable through the NDA methods are used in conjunction with AK derived ratios and scaling factors to quantify the radionuclides not directly quantifiable. To use AK to determine such ratios and scaling factors, the NDA organization must technically justify the AK data and its use with NDA measurement information. The AK ratios or scaling factors must be appropriate to the generation point of the waste, process component, etc.

a) <u>AK Documentation:</u>

The use of AK information concerning the radiological composition of a waste type or process component must be documented either in an AK summary report for that waste type/component or other controlled document. Should this information be contained in AK package(s) prepared to meet other general waste characterization requirements, it need not be duplicated in other controlled documents that address the radiological properties of the waste stream. However, all relevant information must be included in the AK record.

All ratios or scaling factors used must be technically sound and based on known, documented relationships or correlations. Uncertainties reported when using ratios and scaling factors are used must include the uncertainty in the ratio or scaling factor.

The type and quantity of supporting documentation may vary by waste stream and shall be compiled in a written record that includes a summary identifying all sources of information used to delineate the waste stream's isotopic distribution or radionuclide scaling factors. The basis and rationale for the delineation shall be clearly summarized in an AK report and traceable to referenced documents. Assumptions made in this

rationale shall be identified. The following information should be included as part of the AK written record:

i) map of the site with the areas and facilities involved in waste generation and process equipment identified;

ii) facility mission description as related to radionuclide-bearing materials and their management (e.g., routine production, fuel research and development, and experimental processes);

iii) description of the specific site locations (such as the area or building) and operations relative to the isotopic composition of the uranium bearing wastes and process components they generated;

iv) waste identification or categorization schemes used at the facility relevant to the waste material's isotopic distribution (e.g., the use of codes that correlate to a specific isotopic distribution and a description of the isotopic/radionuclide composition of each waste stream);

v) information regarding the waste's physical and chemical composition that could affect the isotopic distribution (e.g., processes used to remove ingrown daughters or alter its expected contribution based solely on radioactive decay kinetics); and

vi) statement of all numerical adjustments applied to derive the material's isotopic distribution (e.g., scaling factors, decay/in-growth corrections and secular equilibrium considerations).

Documentation must be sufficient to enable independent calculation of the scaling factor or ratio of interest.

b) <u>Supplemental AK Information:</u>

Supplemental AK information should be obtained dependent on availability. The amount and type of this information cannot be mandated, but information should be collected as appropriate to support contentions regarding the waste's isotopic distribution. This information is used to compile the waste's AK written record. Supplemental AK documentation that may be used includes but is not limited to information from the following sources:

i) safeguards and security, materials control and accountability, and other nuclear materials control systems or programs and the data they generated;

ii) reports of nuclear safety or criticality, accidents/excursions involving the use of special nuclear material (SNM), or nuclear material;

iii) waste packaging procedures, waste disposal, building or nuclear material management area logs or inventory records, and site databases that provide information on SNM or nuclear materials;

iv) test plans, research project reports, or laboratory notebooks that describe the radionuclide content of materials used in experiments;

v) information from site personnel (e.g., documented interviews); and

vi) historical analytical data relevant to the isotopic distribution of the waste stream.

c) AK Discrepancy Resolution:

If there is any form of discrepancy between AK information related to isotopic ratios or composition, the NDA organization is responsible for having the sources of the discrepancy evaluated to determine information credibility. Information that is not credible or information that is limited in its applicability to the NDA characterization effort will be identified as such, and the reasons for dismissing it will be justified in writing. Limitations concerning the information will be documented in the AK record and summarized in the AK report. In the event the discrepancy cannot be resolved, the site will perform direct measurements for the impacted population of containers or process items. If discrepancies "result in a change to the original determinations, the AK summary will be updated.

1.8 NDA Data Reporting, Review, and Verification

a) NDA Measurement Data Reporting:

The NDA organization is to document individual NDA measurement item results in a standard report format. For each NDA measurement item (waste container/ process component) there shall be a separate report. The NDA measurement item reports shall contain or reference the location of information sufficient to fully describe all input data, NDA measurement configuration information, acquisition parameters, analysis technique, software version, QC data, etc. to allow reconstruction of the reported results.

i) Title and contact information, including:

a. report title (e.g., "NDA Measurement Item Report");

b. name of NDA organization;

c. client contact name for which report is to be delivered and NDA service provider point of contact responsible for ensuring the submittal of the report in the approved manner; and

d. identification of project name, site, or facility NDA measurement items is associated with.

ii) Measurement item identification and QC information:

a. measurement item identification/designator and other identifiers/designations as applicable (e.g., the clients own identifier);

b. date(s) of NDA data acquisition;

c. analysis, background, and QC file names;

d. measurement item description;

e. NDA field worksheet file name, log name, or other identifier;

f. gross/net weight, if applicable;

g. NDA measurement live time; and

h. location of NDA measurement system, site name, facility name, building name, and other identifying information.

iii) Primary radionuclide measurement results:

a. primary NDA measurement quantitation method (e.g., gamma, neutron);

b. primary radioisotopes and their associated TMUs in appropriate units (e.g., gram, activity, activity concentration, MDA, and % uncertainty);

c. total radionuclide mass, activity, concentration, and associated TMU;

d. ^{235}U fissile gram equivalent and associated TMU (gram); and

e. other primary quantities such as uranium enrichment weight percent (wt%) and associated wt% TMU.

iv) NDA acquisition and analysis information:

a. NDA detector or system identification;

b. name of ancillary data and/or information sheets associated with the NDA measurement item. These are often called NDA Field Worksheets and contain information pertinent to the analysis of the acquired data such as container fill height and measurement configuration (e.g., detector to item distance and operator signature/date);

c. identification of real time radiography examination files, if applicable;

d. the acquisition software identification and version; and

e. analysis software identification and version.

v) Comment/Narrative section:

a. name or reference to procedures used to acquire the NDA measurement data, analyze the data and acquire supporting data/information used in analysis;

b. name or reference to QC procedures utilized in the acquisition and processing of the data;

c. identification or reference to WRM and check source(s) used for calibration and/or QC activities;

d. identification of or reference to calibration procedures and records and/or location; and

e. if not specified elsewhere, definition of the quoted uncertainties (i.e., one σ, two σ). When TMU is reported differently on the batch cover sheet of the IMS, the method of expressing TMU shall be specified on the NDA measurement item report sheet or the applicable procedures referenced.

The NDA measurement item report is to have the analyst signature and date and the independent technical reviewer signature and date.

b) <u>NDA Data Review:</u>

All NDA measurement data must be reviewed and approved by qualified personnel prior to being reported. At a minimum, the data and analysis must be reviewed by an independent technical reviewer (a second qualified person). This reviewer shall be an individual other than the data generator (analyst) who is qualified to have performed the initial work. The independent technical reviewer shall verify, at a minimum, the following information:

i) NDA measurement system QC results are within established control limits and, if not, the data have been appropriately dispositioned using the nonconformance process. This shall include a complete summary of qualitative and/or quantitative data for all items with data flags or qualifiers;

ii) “calibration verification” measurements were performed and reviewed as acceptable;

iii) system data acquisition and reduction were conducted in a technically correct manner in accordance with current methods (verification of procedure and revision);

iv) calculations performed outside of software that is in the software QA program have been reviewed by a valid calculation program, a periodic spot check of verified calculation programs (not required with every report) and/or 100 percent check of all hand calculations;

v) proper constants such as half-lives, branching ratios, attenuation values, neutron yields, gamma libraries were used;

vi) data were reported in the proper units and correct number of significant figures;

vii) values that are not verifiable to within rounding or significant difference discrepancies must be rectified prior to completion of independent technical review;

viii) the data have been reviewed for transcription errors;

ix) calibrations have been documented; and
x) Standards used are traceable to nationally recognized certificates.

c) <u>NDA Data Verification:</u>

Data verification is a systematic confirmation by examination and provision of evidence that specified requirements have been met data to ensure that the required data quality characteristics have been obtained. The verification process ensures that applicable quality controls have been properly implemented and data validity per program requirements has been met. Verification activities are usually performed at the batch level where all QA elements ranging from NDA measurement reports to compliance with applicable regulations are collected, collated, and prepared for submittal. NDA measurement data reports are to be provided to the client on a batch basis as determined with and agreed to by the client.

i) Batch data reports are to be prepared for each measurement batch on standard form (hard copy or electronic equivalent). Batch data reports shall at a minimum include the following:

a. NDA organization name, NDA measurement system identification, batch number, NDA measurement item identifications included in the batch, date and signature release by authorized personnel;

b. Table of Contents;

c. QC data, backgrounds, replicate data, and control charts, etc. for the relevant batch time period; and

d. data verification per the NDA service provider QA Plan and as per applicable procedures.

ii) Batch reports must be reviewed and approved by qualified personnel before being submitted. Only appropriately trained and qualified personnel shall be allowed to perform data verification/review. Verification reviews shall ensure:

a. the QC documentation for the batch report is complete and includes as applicable a list of containers in the set or batch and applicable set or batch QC results;

b. data were collected as described in the planning documents and are complete and correct. All batch data reports must be approved by the project manager or designee. The project manager shall verify at a minimum the following information:

i. data generation-level verification have been performed by a second qualified person and appropriate signature release;

ii. batch review checklists are complete;

iii. batch reports are complete and data are properly reported (e.g., data are reported in the correct units and with the correct number of significant figures); and

iv. data comply with program objectives.

Results of the review may require that qualifiers be placed on the use of the data. Verification methods shall be planned and documented. The documentation shall include the acceptance criteria used to determine if the data are valid. For noncompliant data, corrective action procedures shall be implemented.

1.9 NDA Measurement Performance Evaluation

The NDA organization shall demonstrate that its NDA methods, calibrations and uncertainty estimates are applicable to the matrix/process components. Part of this demonstration of proficiency is the participation in performance evaluation (PE) programs as scheduled and conducted by specified qualification and approval agencies if available. Elements of the performance evaluation process include:

a. NDA organization shall demonstrate successful participation in applicable PE program(s). The NDA organization shall demonstrate continued proficiency throughout the term of operation. The testing will be single-blind and representative of the matrix types and configurations and analytes (^{235}U, ^{238}U, etc.) to be characterized.

b. Unacceptable NDA results for PE test sample(s), as determined per PE program criteria, will require the NDA organization to implement corrective action procedures and submit a corrective action plan to the PE program or applicable oversight agency. Results of the corrective action plan shall be documented and available for review.

c. Documentation of successful capability demonstration, such as a Certification Statement or letter of concurrence from the qualifying agency, must be acquired and retained by the NDA organization. All associated supporting data necessary to reproduce the PE measurement results as contained in the Certification Statement or equivalent document must be retained by the NDA organization.

d. Once the initial capability demonstration is successfully completed, continuing demonstration of method performance is to be accomplished through the periodic “calibration verification” measurements as well as all applicable QC requirements.

2.0 NDA QUALITY CONTROL

The purpose of a measurement control program is to test and ensure the stability of the measurement process and to gain additional information on measurement uncertainties where practicable. The measurement control program provides for the administration, evaluation, and control of measurement processes. The design of the measurement control program is to ensure the NDA measurement process provides data of sufficient quality (i.e., the measurement system is in control per defined criteria). The NDA organization can then make and document qualifying statements about the suitability and validity of measurement data as generated for the client and/or end-user.

QC measurements are to be performed in conjunction with and related to a batch of NDA measurement items. A batch is a grouping of similar measurement items to which a set of QC criteria is applied to demonstrate acceptability of the results. The batch size is specified to be 20

items such that when one replicate is performed per batch, a 5% check of the data is achieved. In addition to the replicate requirement are pre- and post-batch QC checks (e.g., background and energy calibration checks). A batch can be fewer than 20 items as in the case where there are fewer than 20 similar measurement items available for analysis or other driving circumstances such as throughput requirements.

For each measurement item batch, QC measures are to be performed before commencement of a batch and at the end of the batch. An analytical batch may span a period of more than one day, but the requirement to perform QC checks per day is not superseded. The replicate QC measure only has to be performed once per batch. Performance checks shall bracket the NDA measurements which comprise the batch. Out of control performance checks for a given NDA instrument shall cause the batch data to be considered suspect. Corrective actions shall be in place to evaluate the measurement item results for the affected batch.

2.1 QC Procedures

The NDA organization shall have procedures implementing applicable QCs for monitoring the validity of NDA measurements and the analytical results. The NDA QA program shall specify qualitative and quantitative acceptance criteria for the QC checks. The NDA QC measures and acquired information/data shall be documented or logged in such a way that trends are detectable. Statistical techniques shall be applied to the evaluation of acquired QC data and action levels specified. Procedures shall also be in place to implement the corrective action process when QC criteria are not satisfied. The QC program shall be periodically reviewed. In addition, the NDA service provider shall address the following:

a) Development of a QC plan with clearly defined roles and responsibilities. The QC program should assure objectivity and independence of action. The person assigned responsibility for the QC program shall be knowledgeable of the measurement system being controlled, statistical QC, and the process being monitored. The organization should provide sufficient separation of functions to avoid any conflict of interest.

b) Acquisition and maintenance of suitable WRMs and check sources to monitor measurement system performance during NDA characterization operations. Records concerning specification and acquisition of standards and sources, including an assessment of their uncertainties and procurement, shall be documented and retained.

c) QC checks shall include a means to evaluate the variability and/or repeatability of NDA measurement results.

d) Determination of measurement parameters and acceptance criteria necessary to ensure the accuracy of the NDA method using daily performance checks and analysis of

performance check data (e.g., control charts, trending analysis, and replicate measurements).

e) QC protocols as specified in the NDA organization method manual and/or procedure(s) shall be followed.

f) QC measurement parameter action levels shall be established and documented.

g) Written procedures shall be developed and documented to address out-of-control conditions and the subsequent re-qualification of the instrument.

2.2 NDA QC Requirements

Procedures cited in various ASTM, ANSI standards, NRC standard practices, and guidelines as referenced in Appendix A are recommended for use at all NDA measurement facilities. QC requirements must at a minimum include the following:

a) Background Measurements must be performed and recorded for neutron and gamma systems for each system in use at least once per day and twice for each batch. The once per day Background Measurement can serve as the beginning or ending background measurement required for the batch. The two Background Measurements for each batch shall bracket the start and end of the batch- one at the beginning of the batch and one at the end of the batch- unless technical justification to do otherwise is developed and documented. The count time for neutron and gamma background checks shall be at least as long as the measurement count time unless otherwise specified and documented by an appropriately qualified individual. The background measurement shall be evaluated before daily NDA measurements commence. Depending on environmental conditions, the background frequency may need to be increased to ensure data quality. Increases in the frequency of background measurements shall be determined and documented by an appropriately qualified individual (Note: Enrichment measurement systems that employ an infinite-thickness analysis technique do not require a background performance check). The recorded background data is to be monitored using control charts or tolerance charts to ensure the background environment is within statistical control. Contributions to background because of radiation from nearby radiation producing equipment, standards, or wastes must be controlled to the extent practicable or more frequent background checks must be performed.

b) Instrument Performance Measurement checks must be acquired for each NDA measurement system in use at least once per day and twice for each data batch. For each performance check, two measurements shall be used to bracket the batch- one before and one after the batch measurements are completed. Performance checks

include detection efficiency checks; matrix correction checks; and for spectrometric instruments, energy calibration and energy resolution checks. The NDA organization is to establish acceptable performance check ranges or limits as applicable. An out-of-control energy calibration check may cause measurement item results to be suspect since the last successful energy calibration check. Energy calibration checks can be performed at a greater frequency than once per day. Performance checks, as applicable, shall also be acquired on support equipment. The recorded performance measurement checks are to be monitored using control charts or tolerance charts to ensure the instrument performance is within statistical control.

c) Replicate Measurements are used to determine the repeatability of a measurement system that represents the intrinsic instrument variability. Repeatability variance is a short-term variance usually dominated by counting statistics. The Replicate Measurement is acquired by randomly selecting one measurement item that has been processed through the NDA system for the batch. This measurement item is then to be re-measured using the same NDA system, software, and acquisition/reduction parameters. Data analysis is to be performed independently for the two measurements. The second measurement of the item is to be performed any time before the start of the next data set or batch. This repeat measurement is then the replicate for that batch. A minimum of one Replicate Measurement is required for each batch. For a randomly selected Replicate Measurement item that corresponds to a measurement below the lower limit of quantitation (LLQ), the 95% uncertainty ranges of the pair of measurements must overlap.

When two replicates are utilized to assess repeatability, the data should be evaluated using the Relative Percent Difference (RPD) as follows:

$$\frac{|D-S|}{S} \times 100\% \leq 25\%$$

Where:

S = initial randomly selected measurement item

D = duplicate result for measurement item S

An acceptable RPD shall be less than or equal to 25% or other criteria specifically requested by the client. A control chart of the RPD shall be maintained for trending analysis. Procedures shall be established for the collection, processing, and periodic evaluation of replicate data. Alternate methods for determining repeatability and assessing its acceptability may be implemented by the NDA organization provided they are technically justifiable, documented, and available for review. The replicate data is to be monitored using control charts or tolerance charts to ensure the instrument reproducibility is within statistical control.

d) Check sources used for QC checks should be traceable, long-lived, and provide adequate counting statistics for a relatively short count time. If the check source is not traceable, it should be correlated with a traceable source or well-known, characterized, and documented.

e) All performance data shall be monitored on an as-recorded basis and over time using control charts and trending techniques. Most monitoring techniques assume that measurement data are distributed normally and that observations are independent. The assumption of normality should be assessed prior to implementation of a control regimen. The NDA organization is responsible for determining acceptance criteria for as-recorded and long term data trending. Recommended control chart limits and actions levels are contained in Table D-3. Corrective action plans or procedures shall be in place to manage out-of-control results and the associated measurement item data.

3.0 QC ACTION LEVELS AND RESPONSE

Quality control measurements shall be performed on a periodic basis as prescribed above and evaluated relative to established acceptance criteria. Quality control measurements shall also be reviewed and evaluated over time to determine continued acceptability of the assay system and to monitor trends. If daily quality control checks yield results that are outside the acceptable range(s), the required responses in Table D-3 must be followed. The NDA service provider may implement more restrictive control limits and other administrative limits as applicable. All control limits and associated actions are to be documented and maintained. Refer to Table D-3 Range of Applicability.

Table D-3. Range of Applicability.

Category	Acceptability Range[a]	Required Response
Acceptable Range	$\lvert Data \rvert$ [b] $\leq 2\sigma^c$	No action required
Warning Range	$2\sigma^c < \lvert Data \rvert \leq 3\sigma^c$	The performance check shall be rerun no more than two times. If the rerun performance result is within 2σ, then the additional performance checks shall be documented and work may continue. If the system does not fall within the ± 2σ after two rerun performance checks, then the required response for Action Range shall be followed.

Action Range	$\lvert Data \rvert$ > 3σ[c]	Work shall stop and the occurrence shall be documented and appropriately dispositioned (e.g., initiating a nonconformance report). The NDA system shall be removed from service pending successful resolution of the failure cause. All assays performed since the last acceptable performance check, are suspect, pending satisfactory resolution. At a minimum, "calibration verification" is required prior to returning the system back to service.

a - American National Standards Institute. *Nondestructive Assay Measurement Control and Assurance*, ANSI N15.36.

b - absolute value.

c - the standard deviation is only based on the reproducibility of the data check measurements themselves. This is not TMU.

Appendix E: The Hanford Analytical Services Quality Assurance Requirements Documents (HASQARD) Specific Requirements Checklist

The HASQARD is a Department of Energy Hanford Site-wide sampling and analytical standard issued by DOE Richland Operations Office and DOE Office of River Protection. The HASQARD is a contractual requirement in all prime contracts with DOE RL and ORP and its compliance is mandatory. The HASQARD provides a single set of analytical criteria acceptable to the Hanford Site regulators and establishes the QA/QC requirements to achieve a consistent level of quality across the Hanford Site for analysis of environmental cleanup samples at onsite laboratories and offsite commercial laboratories. The Hanford specific requirements checklist contains analytical QA/QC requirements that are currently not found in the DoD/DOE QSM. These requirements, in addition to the other requirements of the DoD/DOE QSM, must be implemented by commercial laboratories having subcontracts with Hanford Site prime contractors in order to demonstrate compliance with the HASQARD.

HASQARD Checklist Item Number	Line of Inquiry	Notes
1.0	**Hanford Analytical Quality Assurance Requirements Documents (HASQARD) Revision 4 Volume 1 (Programmatic)**	
1.1	The QA program shall identify all positions given the responsibility and authority to do the following: Stop unsatisfactory work. The plan shall identify the chain of command through which any employee may initiate a stop-work order where detrimental ethical, contractual, quality, safety, or health conditions exist	
1.2	Laboratory operations covered by procedures shall include cleaning of glassware.	
1.3	The laboratory shall have technical and test procedures as appropriate to the scope and complexity of the procedures or work requested: • Approaches to address background corrections • Procedures, when automatic quantitation algorithms are overridden	

HASQARD Checklist Item Number	Line of Inquiry	Notes
1.4	The laboratory shall track authorized departures from established procedure steps and periodically evaluate if formal procedure revision is appropriate.	
1.5	Quality-related information to be analyzed shall include but not be limited to the following, as appropriate: • Lessons learned	
1.6	Laboratories must have a records control system standard operating procedure which shall include the following: • Access to and control of the files including accountability for any records removed from storage. • Procedures for filing of supplemental information and disposing of superseded records. • Replacement, restoration, or substitution of lost or damaged records. • If applicable, protection of records from magnetic sources	
1.7	**Intentionally Left Blank**	
1.8	The laboratory shall have a procedure requiring written approval to be received from all affected clients, prior to disposal of any records associated with DOE analytical data.	
1.9	Procurement documents shall identify requirements for a software supplier's reporting of software errors to the purchaser and, as appropriate, the purchaser's reporting of software errors to the supplier.	
1.10	If the laboratory's statement of work requires that a vendor have an approved QA program, then the laboratory shall verify that the vendor's software QA program and submitted documentation meet the requirements specified in the statement of work.	
1.11	The laboratory QA program shall identify the required distribution of internal audit reports, management review reports, and all related documentation.	
1.12	Management reviews shall include documented identification of noteworthy accomplishments as applicable.	
1.13	The laboratory QA program shall establish and implement a formal mechanism for reporting the status of the QA program to management.	
1.14	QA status reports to management shall be issued annually, at a minimum.	

HASQARD Checklist Item Number	Line of Inquiry	Notes
1.15	The QA status reporting system shall identify the following: • frequency schedule for the report • report recipient • report preparer • topics to be discussed in the report	
2.0	**Hanford Analytical Quality Assurance Requirements Documents (HASQARD) Revision 4 Volume 4 (Programmatic)**	
2.1	The laboratory shall have a system to notify and explain any unique client requirements to all staff performing work for the client. Unique requirements are those that differ from the procedures described in the laboratory procedures.	
2.2	The laboratory shall have a system in place to allow tracking of samples and holding times. The system shall allow the laboratory to assess if holding times shall be met or exceeded, and assess the number of samples available for analysis.	
2.3	If multiple calibration curves are used, analytical QC must be performed for each curve.	
2.4	Over-range samples are diluted or otherwise re-analyzed within the calibration range of the measurement system or, when necessary, reported using defined qualifiers to denote increased quantitative uncertainty.	
2.5	The following information shall be maintained regarding laboratory-prepared standards, reagents, reference materials, or other media potentially impacting the quality of reported results: • Dilution documentation, including volume/weight of standards, final volume, etc. • Traceability to any critical equipment used (e.g., balance IDs, pipette IDs) • Final concentration	
2.6	Instrument operational parameters (e.g. temperature, temperature change rate, flow, mobile phase carrier, solvent, counting time, attenuation, detector voltage) shall consider the manufacturer's recommendations or requirements and will be consistent, as applicable to the analytical method, between client samples, laboratory QC samples, and calibration standards.	

HASQARD Checklist Item Number	Line of Inquiry	Notes
2.7	Records associated with instrument calibration and control shall identify the corrective actions taken for unacceptable results.	
2.8	When recalibration is required at an unusual and/or increasing frequency, HASQARD Vol. 1, Section 5.4, "Recurring Conditions Adverse to Quality" shall be invoked as part of the corrective actions. (see next page for details)	

HASQARD Checklist Item 2.8

Insert additional DOE Only Requirement:

When recalibration is required at an unusual and/or increasing frequency, the following shall be invoked as part of the corrective actions.
The corrective action process shall describe the provisions for determining if corrective actions have not been effective in preventing recurrence of quality problems. Preventive action shall be initiated, as appropriate, considering the magnitude of potential problems. When preventive measures are implemented, their effect shall be monitored to ensure that desired quality objectives are satisfied and maintained.
Provisions for making corrective action determinations shall include, but not be limited to, the following:

- Determining the events leading to the adverse condition
- Determining the technical and work activities associated with the quality problem
- Ascertaining the quality problem's generic implications
- Determining the extent to which similar quality problems (or precursors to the problem) have been recognized
- Determining the effectiveness of any corrective actions that were taken
- Determining the impacts on the completed work
- Determining actions that can be taken by the responsible organization to preclude recurrence
- Determining if stopping the work associated with the activity is necessary.

2.9	The following information shall be traceable to the raw data output: • date of sample collection, • date of sample preparation, • date of analysis run, • sample identification numbers, • analyst or instrument operator, • type of analysis, • procedure number, including revision number	
2.10	The laboratory shall establish SOPs on how to verify that all readings or output are accurate to ensure accuracy of data entry, proper calculation, and appropriate data reduction.	
2.11	Basic rules for significant figures and for calculating values and retaining the number of significant figures shall be based upon an authoritative source or accepted standard such as the ASTM E-29, *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications*.	
2.12	When a figure is to be rounded to fewer digits than the total number available, the rounding-off procedure shall be based upon an authoritative source or accepted standard such as that described in ASTM E-29. A brief interpretation of the ASTM E-29 procedure follows: • When the first digit discarded is less than five, the last digit retained should not be changed. • When the first digit discarded is greater than five, the last figure retained should be increased by one. • When the first digit discarded is exactly five, followed only by zeros, the last digit retained should be rounded upward if it is an odd number, but no adjustment made if it is an even number.	
2.13	The laboratory sample data review process shall include data verification of sample identification number, analyst and date of analysis.	
2.14	Errors detected in the review process shall be referred to the analyst for corrective action.	
2.15	Sample preparation procedures shall be established by the laboratory and shall consider the manufacturer's recommendations or requirements (e.g., specific sample vial configuration/composition, reagent to analyte ratios, special reagent handling, etc.) where applicable.	
2.16	Purchased stock mixtures and reagents shall be labeled to indicate the date on which the mixtures/reagents were opened and the expiration date.	
2.17	Glassware cleaning shall be performed in a manner such that sample contamination is minimized.	

2.18	Laboratories shall clean common sample processing equipment (e.g., grinders) between samples and maximize use of disposable sample handling equipment (e.g., spatulas, pipettes, or balance pans) to minimize cross sample contamination.	
2.19	For preparation blank (method blank) failures, the client shall be notified prior to re-preparation and analysis when additional costs will be incurred.	
2.20	Preparation blank results shall not be subtracted from sample results unless client requirements specify otherwise.	
2.21	No adjustment of client sample results based on LCS or BS recovery is made in the laboratory report.	
2.22	No adjustment of the client sample results is made in the laboratory report based on the MS recovery.	
2.23	Samples with target analyte concentrations exceeding the blank contamination level by a factor of 20 or more can be reported unless client requirements dictate otherwise. In all other cases, all samples analyzed since the last acceptable initial calibration blank or continuing calibration blank shall be reanalyzed for the specific analyte failure.	
2.24	Calculations used for the following common data quality indicators shall be in agreement with those defined below: • percent relative standard deviation • relative percent difference • method accuracy based on sample spike • method accuracy based on standard	

HASQARD Checklist Item 2.24
Insert additional DOE Only Requirement:

Percent Relative Standard Deviation: The RSD is used when at least three replicate measurements are performed on a given technique. The RSD is computed using the following equation:

$$\mathrm{RSD} = \frac{s}{\bar{x}} * 100$$

Where s = standard deviation with n - 1 degree of freedom, n = total number of observed values, and $\bar{x}$ = mean of observed values.

Relative Percent Difference: The RPD is used when two measurements exist. The RPD is generally used to express the precision of duplicate or spike duplicate samples. The RPD is computed using the following equation:

$$\mathrm{RPD} = \frac{|x_1 - x_2|}{\bar{x}} * 100$$

Where $x_{1,2}$ are the observed values and $\bar{x}$ = mean of observed values.

Method Accuracy Based on Sample Spike: Accuracy for the sample is expressed as the percent recovery (%R) of a MS (or MSD) sample. The percent recovery is calculated based on the following equation:

$$\%\mathrm{R} = \frac{(\mathrm{SSR} - \mathrm{SR})}{\mathrm{SA}} * 100$$

Where SSR = spiked sample result, SR = sample result, and SA = spike added.

Method Accuracy Based on Standard: Accuracy for the sample is expressed as the percent recovery (%R). The percent recovery of a standard is calculated according to the following equation:

$$\%\mathrm{R} = \frac{A_m}{A_k} * 100$$

Where A_m = measured value of the standard analyte and A_k = known value of the spike added.

3.0	**Hanford Analytical Quality Assurance Requirements Documents (HASQARD) Revision 4 Volume 4 (Organics Data Quality)**	
3.1	Preparation blank or method blank acceptability shall be demonstrated as follows: 1) the blank shall be less than the estimated quantitation limit (EQL) of the associated samples, or 2) if the blank is equal to or above the EQL, it shall not exceed 5% of the measured concentration present in the associated samples, or 3) when a decision level is specified, the blank shall not exceed 5% of the decision level unless (2) above applies.	
3.2	Surrogate recoveries are reported as measured (i.e., no sample recovery corrections are performed based on surrogate recovery).	
3.3	Total organic carbon, total inorganic carbon, and total carbon measurements should also include continuing calibration checks every 15 samples and at the end of the analytical run.	
4.0	**Hanford Analytical Quality Assurance Requirements Documents (HASQARD) Revision 4 Volume 4 (Inorganics Data Quality)**	
	No additional requirements	
5.0	**Hanford Analytical Quality Assurance Requirements Documents (HASQARD) Revision 4 Volume 4 (Radiochemistry Data Quality)**	
5.1	Preparation or method blank acceptability shall be demonstrated as follows: 1) the blank shall be less than the estimated minimum detectable concentration (MDC) or 2 x the total propagated uncertainty (TPU) of the associated samples, or 2) if the blank is equal to or above the estimated MDC or 2 x TPU, it shall not exceed 5 percent of the measured activity present in the associated samples, or 3) when a decision level or client required detection limit is specified, the blank shall not exceed 5 percent of the decision level or client required detection limit unless (2) above applies.	
5.2	Blank spike (BS) control is demonstrated by target analytes being within either the preset limit of 80 to 120 percent, or within statistically determined limits when the ±20 percent criteria cannot be reliably achieved.	
5.3	The criteria for selection and recovery (maximum and minimum limits) of matrix spike (MS) materials shall be specified. Nominal recovery limits for MS should be 75 to 125 percent. Alternately, statistical evaluation of laboratory performance over time for samples of similar matrix and concentration level may be used.	

5.4	Radiochemical duplicate relative percent difference (RPD) criterion is set at 20 percent for aqueous liquids and 30 percent for all others. This criterion shall apply when both sample results are >5 times the MDC and the individual uncertainties are < 20 percent. Or alternatively, the duplicates should agree within two standard deviations. The laboratory can also perform an evaluation using the mean difference.	
5.5	The criteria for selection and recovery (maximum and minimum limits) of carriers shall be specified, as use may be considered unique to the specific isotope being determined. Nominal recovery limits for carriers should be 40 to 110 percent.	