Department of Defense (DoD)

Department of Energy (DOE)

Consolidated

Quality Systems Manual (QSM) for Environmental Laboratories

Based on ISO/IEC 17025:2005(E)

and

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

DoD Quality Systems Manual Version 5.0

DOE Quality Systems for Analytical Services Version 3.0

July 2013

Department of Defense (DoD)

Quality Systems Manual (QSM) for Environmental Laboratories

Based on ISO/IEC 17025:2005(E)

and

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

DoD Quality Systems Manual Version 5.0

July 2013

Department of Defense (DoD)

Quality Systems Manual (QSM) for Environmental Laboratories

Version 5.0

7/17/13

Kevin Coats
Army Principal

Date

7/23/13

Cornell Long
Air Force Principal

Date

7/17/13

Jordan Adelson, Ph.D.
Navy Principal, EDQW Chair

Date

Department of Energy (DOE)

Quality Systems for Analytical Services

Based on ISO/IEC 17025:2005(E)

and

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

DOE Quality Systems for Analytical Services Version 3.0
July 2013

Department of Energy (DOE)

Quality Systems for Analytical Services (QSAS)

Version 3.0

10/31/2013

Jorge A. Ferrer — **Date**
Manager, Department of Energy Consolidated Audit Program
Oak Ridge Office

10/30/2013

Anita R. Bhatt — **Date**
Director, Radiological and Environmental Sciences Laboratory
Idaho Operations Office

10/30/2013

George E. Detsis — **Date**
Manager, Analytical Services Program
Office of Health, Safety and Security

Preface

The Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) Operations Team developed this manual called the *DoD/DOE Quality Systems Manual (QSM) for Environmental Laboratories*. The 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 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 qualification in accordance with the DOECAP and DOE related contract awards. 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 specific provisions in those standards are superseded by this document. All references to the term "accreditation" in this manual refer to the DoD ELAP only.

To alleviate issues of copyright and provide a manual that is freely available to all, this manual is presented in a new format, which must be used in conjunction with the TNI and ISO/IEC 17025:2005(E) standards. DoD/DOE specific language is presented as text and appendices in the order in which topics are addressed in the TNI standard. 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. Current accreditation to DoD QSM version 4.2 is considered equivalent to accreditation to this manual. DoD ELAP Accreditation Bodies will accredit laboratories to this version of the standard during their normal accreditation cycles.

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 DOECAP team.

Table of Contents

VOLUME 1, MODULE 1: PROFICIENCY TESTING

1.0 INTRODUCTION 1

2.0 REQUIREMENTS FOR ACCREDITATION (SECTION 2 IS DOD ONLY) 1

2.1 INITIAL ACCREDITATION 1
2.2 CONTINUING ACCREDITATION 2

3.0 REQUIREMENTS FOR PARTICIPATION (SECTION 3 IS DOE ONLY) 3

3.1 INITIAL INCLUSION 3
3.2 CONTINUED PARTICIPATION 4

VOLUME 1, MODULE 2: QUALITY SYSTEMS GENERAL REQUIREMENTS

1.0 INTRODUCTION, SCOPE, AND APPLICABILITY 6

1.1 INTRODUCTION 6
1.2 SCOPE DOD/DOE (CLARIFICATION) 6

2.0 NORMATIVE REFERENCES 7

3.0 TERMS AND DEFINITIONS 7

3.1 ADDITIONAL TERMS AND DEFINITIONS 7
3.2 SOURCES 11
3.3 EXCLUSIONS AND EXCEPTIONS 11

4.0 MANAGEMENT REQUIREMENTS 11

4.1 ORGANIZATION 11
4.2 MANAGEMENT 11
4.3 DOCUMENT CONTROL 15
4.4 REVIEW OF REQUESTS, TENDERS AND CONTRACTS 15
4.5 SUBCONTRACTING OF ENVIRONMENTAL TESTS 15
4.6 PURCHASING SERVICES AND SUPPLIES 16
4.7 SERVICE TO THE CLIENT 16
4.8 COMPLAINTS 16
4.9 CONTROL OF NONCONFORMING ENVIRONMENTAL TESTING WORK 17
4.10 IMPROVEMENT 17
4.11 CORRECTIVE ACTION 17
4.12 PREVENTIVE ACTION 17
4.13 CONTROL OF RECORDS 17
4.14 INTERNAL AUDITS 19
4.15 MANAGEMENT REVIEWS 19
4.16 DATA INTEGRITY INVESTIGATIONS 19

5.0 TECHNICAL REQUIREMENTS 20

5.1 GENERAL 20
5.2 PERSONNEL 20
5.3 ACCOMMODATION AND ENVIRONMENTAL CONDITIONS 21
5.4 ENVIRONMENTAL METHODS AND METHOD VALIDATION 22
5.5 CALIBRATION REQUIREMENTS 25
5.6 MEASUREMENT TRACEABILITY 27
5.7 COLLECTION OF SAMPLES 28
5.8 HANDLING SAMPLES AND TEST ITEMS 28
5.9 QUALITY ASSURANCE OF ENVIRONMENTAL TESTING 32

ED_006532_00005078-00007

5.10 Reporting the Results 32

6.0 HAZARDOUS AND RADIOACTIVE MATERIALS MANAGEMENT AND HEALTH AND SAFETY PRACTICES 35

6.1 Radioactive Materials Management and Control 35
6.2 Toxic Substances Control Act (TSCA) Material 36
6.3 Laboratory Safety and Health 36
6.4 Waste Management and Disposal 37

VOLUME 1, MODULE 3: QUALITY SYSTEMS FOR ABSESTOS TESTING

1.0 ASBESTOS TESTING 39

1.6 Demonstration of Capability 39
1.7 Technical Requirements 39

VOLUME 1, MODULE 4: QUALITY SYSTEMS FOR CHEMICAL TESTING

1.0 CHEMICAL TESTING 41

1.5 Method Validation 41
1.6 Demonstration of Capability (DOC) 43
1.7 Technical Requirements 43

VOLUME 1, MODULE 5: QUALITY SYSTEMS FOR MICROBIOLOGICAL TESTING

VOLUME 1, MODULE 6: QUALITY SYSTEMS FOR RADIOCHEMICAL TESTING

1.0 RADIOCHEMICAL TESTING 51

1.1 Introduction 51
1.2 Scope 51
1.3 Terms and Definitions DoD/DOE (Clarification) 51
1.4 Method Selection 51
1.5 Method Validation 51
1.7 Technical Requirements 55
1.8 Method Specific Directions DoD/DOE (Requirements) 63

VOLUME 1, MODULE 7: QUALITY SYSTEMS FOR TOXICITY TESTING

APPENDIX A: REPORTING REQUIREMENTS

1.0 COVER SHEET 73

2.0 TABLE OF CONTENTS 73

3.0 CASE NARRATIVE 73

4.0 ANALYTICAL RESULTS 74

5.0 SAMPLE MANAGEMENT RECORDS 75

6.0 QA/QC INFORMATION 75

7.0 DATA REPORTS FOR THIRD PARTY REVIEW OR VALIDATION 76

APPENDIX B: QUALITY CONTROL TABLES

TABLE -1. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY 77

TABLE- 2. ORGANIC ANALYSIS BY HIGH-PERFORMANCE LQIUD CHROMATOGRPAHY 82

ED_006532_00005078-00008

TABLE – 3. NITROAROMATICS, NITRAMINES, AND NITRATE ESTERS ANALYSIS BY HPLC, LC/MS, OR LC/MS/MS (METHOD 8330B) 86

TABLE – 4. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY 93

TABLE - 5. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRPAHY/LOW-RESOLUTION MASS SPECTROMETRY (METHOD 8280) 98

TABLE- 6. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRPAHY/HIGH-RESOLUTION MASS SPECTROMETRY (METHOD 8290) 105

TABLE – 7. INORGANIC ANALYSIS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) 110

TABLE – 8. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY 113

TABLE – 9. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY (ICP/MS) 117

TABLE – 10. INORGANIC ANALYSIS BY COLORIMETRIC HEXAVALENT CHROMIUM 122

TABLE – 11. CYANIDE ANALYSIS 125

TABLE – 12. COMMON ANIONS ANALYSIS BY IC 129

TABLE - 13. PERCHLORATE BY MASS SPECTROMETRY METHODS 132

TABLE -14.CHEMICAL WARFARE AGENTS BY GC/MS 139

TABLE -15. PERFLUORINATED COMPOUNDS BY LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY 144

TABLE – 16. ALPHA SPECTROMETRY 148

TABLE -17. GAMMA SPECTROMETRY 154

TABLE – 18. GAS FLOW PROPORTIONAL COUNTING 160

TABLE - 19. LIQUID SCINTILLATION COUNTER ANALYSIS 167

APPENDIX C: LABORATORY CONTROL SAMPLE (LCS) CONTROL LIMITS AND REQUIREMENTS

1.0 INTRODUCTION 173

2.0 LCS LIMIT TABLES 173

TABLE 1. METHOD 1668 SOLID MATRIX 173
TABLE 2. METHOD 1668 WATER MATRIX 174
TABLE 3. METHOD 6010 SOLID MATRIX 176
TABLE 4. METHOD 6010 WATER MATRIX 177
TABLE 5. METHOD 6020 SOLID MATRIX 178
TABLE 6. METHOD 6020 WATER MATRIX 180
TABLE 7. METHOD 6850 SOLID MATRIX 181
TABLE 8. METHOD 6850 WATER MATRIX 181
TABLE 9. METHOD 7196 SOLID MATRIX 181
TABLE 10. METHOD 7196 WATER MATRIX 182
TABLE 11. METHOD 7470-7471 SERIES SOLID MATRIX 182
TABLE 12. METHOD 7470-7471 SERIES WATER MATRIX 182

ED_006532_00005078-00009

TABLE 13. METHOD 8015 (MOD) SOLID MATRIX....182
TABLE 14. METHOD 8015 (MOD) WATER MATRIX....183
TABLE 15. METHOD 8081 SOLID MATRIX....183
TABLE 16. METHOD 8081 WATER MATRIX....184
TABLE 17. METHOD 8082 SOLID MATRIX....185
TABLE 18. METHOD 8082 WATER MATRIX....186
TABLE 19. METHOD 8141 SOLID MATRIX....186
TABLE 20. METHOD 8141 WATER MATRIX....187
TABLE 21. METHOD 8151 SOLID MATRIX....189
TABLE 22. METHOD 8151 WATER MATRIX....189
TABLE 23. METHOD 8260 SOLID MATRIX....190
TABLE 24. METHOD 8260 WATER MATRIX....195
TABLE 25. METHOD 8270 SOLID MATRIX....200
TABLE 26. METHOD 8270 WATER MATRIX....205
TABLE 27. METHOD 8270 SIM SOLID MATRIX....210
TABLE 28. METHOD 8270 SIM WATER MATRIX....212
TABLE 29. METHOD 8290 SOLID MATRIX....214
TABLE 30. METHOD 8290 WATER MATRIX....214
TABLE 31. METHOD 8310 SOLID MATRIX....215
TABLE 32. METHOD 8310 WATER MATRIX....216
TABLE 33. METHOD 8321 SOLID MATRIX....217
TABLE 34. METHOD 8321 WATER MATRIX....218
TABLE 35. METHOD 8330 SOLID MATRIX....218
TABLE 36. METHOD 8330 - 8330B SERIES WATER MATRIX....219
TABLE 37. METHOD 8330B SOLID MATRIX....220
TABLE 38. METHOD 9010 - 9020 SERIES SOLID MATRIX....221
TABLE 39. METHOD 9010 - 9020 SERIES WATER MATRIX....221
TABLE 40. METHOD 9056 SOLID MATRIX....222
TABLE 41. METHOD 9056 WATER MATRIX....222
TABLE 42. METHOD RSK-175 WATER MATRIX....223
TABLE 43. METHOD TO-15 GAS MATRIX....223

APPENDIX D: NON DESTRUCTIVE ASSAY (NDA)

1.0 QUALITY ASSURANCE....228

1.1 NDA SYSTEM CALIBRATION....228
1.2 NDA METHOD DETECTION LIMIT....235
1.3 INFINITE THICKNESS....236
1.4 NDA MEASUREMENT UNCERTAINTY....236
1.5 NDA MEASUREMENT TRACEABILITY....236
1.6 NDA MEASUREMENT SYSTEM SOFTWARE....238
1.7 ACCEPTABLE KNOWLEDGE....239
1.8 NDA DATA REPORTING, REVIEW, AND VERIFICATION....241
1.9 NDA MEASUREMENT PERFORMANCE EVALUATION....245

2.0 QUALITY CONTROL....245

2.1 QC PROCEDURES....246
2.2 NDA QC REQUIRMENTS....247

3.0 QC ACTION LEVELS AND RESPONSE....248

ED_006532_00005078-00010

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 is DoD Only)

2.1 Initial Accreditation

2.1.1 Initial Accreditation for DoD ELAP

To obtain initial accreditation for 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-6010, or lead-soil-6020) that corresponds to their 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 (ISO) 17043 (General Requirements for Proficiency Testing) from an International Laboratory Accreditation Council (ILAC) approved signatory Accreditation Body. Laboratories seeking DoD ELAP accreditation have the option to obtain PT samples from the Mixed Analyte Performance Evaluation Program (MAPEP). MAPEP is required for all laboratories that possess a radioactive materials license for analysis of radiological samples. MAPEP PT samples for analyte suites that do not contain radioactive materials can be accepted by laboratories without a radioactive materials license.

2.1.3 PT Samples not from ISO 17043 Accredited PT Provider

When PT samples cannot be obtained from an ISO 17043 accredited PT provider, the laboratory shall obtain permission to use non-ISO 17043 PT providers from their Accreditation Body 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 the DoD ELAP Accreditation Body. Other measures

ED_006532_00005078-00011

(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.

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 twelve (12) months old. The analysis date between PT samples shall be at least fifteen (15) calendar days apart if two or more successive PT samples are performed. The fifteen (15) calendar day requirement does not apply to the MAPEP program. Laboratories that participate in the MAPEP program shall follow the MAPEP program requirements.

2.1.6 PT Study Determination

The success or failure of any analyte-matrix-method combinations for a PT study shall be determined by the PT provider under the requirements of the governing regulatory or ISO 17043 statistically derived program.

2.1.7 PT Samples Same as Regular Environmental Samples

In all cases, PT samples must be evaluated the same as regular environmental samples. A laboratory shall employ the same quality control, sequence of 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. Each PT sample shall be analyzed approximately six (6) months apart (i.e., any time frame from four (4) to eight (8) months apart is considered acceptable) if two PT samples are analyzed. 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 fifteen (15) calendar days apart. The fifteen (15) calendar day requirement does not apply to the MAPEP program. Laboratories that participate in the MAPEP program shall follow the MAPEP program requirements.

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.

ED_006532_00005078-00012

3.0 Requirements for Participation (Section 3 is DOE Only)

3.1 Initial Inclusion

3.1.1 Initial Inclusion into the DOECAP Program

The laboratory shall demonstrate successful participation for a minimum of one year in an ISO 17043 accredited PT program. The single blind studies must be related to regulatory or environmental programs, matrix types, or analytes for each of the analytical disciplines (i.e., inorganic, organic, radiochemistry) that each laboratory will perform in support of DOE field offices. A laboratory is only required to analyze samples containing analytes, and samples of matrices, applicable to data they report under DOE contracts.

3.1.2 PT Samples for Initial Inclusion

MAPEP is required for all laboratories that possess a radiological materials license and that perform inorganic, semi-volatile organic, or radiochemical analyses for DOE. Laboratories that perform volatile organic and wet chemistry analyses to DOE will be required to maintain proficiency in ISO 17043 accredited PT program for all matrices 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 to receive MAPEP samples that contain radiological materials. However, MAPEP PT samples for organic analytes do not contain radioactive materials and can be accepted by laboratories without a radioactive materials license. Participation in MAPEP for laboratories that do not have a radioactive materials license is permitted at the request of the laboratory or as required by DOE subcontract requirements. In either case, the results submitted by the laboratories will be subject to the same evaluation criteria as used for laboratories that have a radiological materials license.

MAPEP samples are not provided for volatile organics or polychlorinated biphenyls (PCBs) in any matrix. The laboratories must obtain volatile and PCB PT samples from other ISO 17043 accredited suppliers.

Other programs (such as Drinking Water) require program specific PT samples. The following are required ISO 17043 PT providers for these other programs:

RadCheM™ PT Program, conducted by Environmental Resource Associates (or equivalent programs offered by other commercial suppliers if such suppliers become ISO 17043 accredited in the future), for radioactivity measurements in drinking water.

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

NELAC Fields of Testing for SDWA-Water (formerly known as WS). Under the terms of this manual, a laboratory must participate in two single blind, single concentration PT studies

ED_006532_00005078-00013

provided by an approved supplier. The PT suppliers must be approved by the PEOP/PEPA administered by the NELAP.

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

Other Recommended Programs include:

DMR QA program for NPDES analysis.

NELAC Fields of Testing for RCRA Solid. Under the terms of this manual, a laboratory may participate in two single-blind, single-concentration Proficiency Evaluation (PE) studies provided by an approved supplier. The PE suppliers must be approved by the Proficiency Testing Provider Accreditor/ Proficiency Testing Oversight Body (PTPA/PTOB) administered by the NELAP.

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. The results of all PT programs will be utilized in the reports produced for DOE laboratory users. Therefore, DOE will provide the laboratories operating to this manual instruction for ensuring the results of commercial PT studies are made available to DOE and the sites that have contracts with the laboratories.

3.2.2 Failure to Meet Criteria

Reporting an unacceptable value, as calculated by the PT program, may result in a probationary period until the next reporting period for that analyte. Any applicable analyte for which individual laboratory results are entered as NR or "not reported" will not be considered an acceptable result. Any individual analyte failures must be corrected within the next PT program performance cycle period. If the laboratory fails two consecutive evaluations, the laboratory may not receive samples for analysis by the failed method until an acceptable PT score has been achieved. The decision to withhold sample shipments will be at the discretion of the individual DOE contract holder. The laboratory can demonstrate proficiency in remedial MAPEP PT studies by acceptable performance in an unscheduled evaluation by the same PT program or by participation in the next regularly scheduled MAPEP study.

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

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

ED_006532_00005078-00014

i. Consistent bias, either positive or negative, at the "Warning" level (greater than +/- 20% bias) for a targeted analyte in a given sample matrix for the two most recent test sessions (e.g., Sr-90 in air filter 13 "+W" (+26%), Sr-90 in air filter test 14 "+W" (+28%));
ii. Quality issues (flags other than "Acceptable") that were not identified by the above for a targeted analyte in a given sample matrix over the last three test sessions, (e.g., Am-241 in soil test 12 "-N" (-47%), Am-241 in soil test 13 "+W" (+24%) in soil test 13 "+W" (+24%). Am-241 in soil test 14 "-N" (-38%)); and
iii. Any other performance indicator and/or historical trending that demonstrate an obvious quality concern (e.g., consistent "False Positive" results for Pu-238 in all tested matrices over the last three test sessions).

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

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

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

ED_006532_00005078-00015

Volume 1, Module 2: Quality Systems General Requirements

1.0 INTRODUCTION, SCOPE, AND APPLICABILITY

1.1 Introduction

1.2 Scope DoD/DOE (Clarification)

The following is a clarification of *TNI 1.2:*

The Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) Operations Team 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 qualification in accordance with the DOECAP and DOE related contract awards. 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 only.

This manual is presented in a new format, which is designed for use in conjunction with the TNI (September 2009) and ISO/IEC 17025:2005(E) standards. DoD/DOE specific language is presented as text and appendices in the order in which topics are addressed in the TNI standard. 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 Operations Team strongly encourages the involvement of project chemists and laboratories during project-planning activities.

ED_006532_00005078-00016

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

3.0 TERMS AND DEFINITIONS

3.1 Additional Terms and Definitions

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

Accreditation (DoD Only Clarification): Refers to accreditation in accordance with the DoD ELAP.

Accreditation Body (DoD Only Clarification): Entities recognized in accordance with the DoD ELAP 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.

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.

ED_006532_00005078-00017

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.

Digestion: A process in which a sample is treated (usually in conjunction with heat and acid) to convert the 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.

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

ED_006532_00005078-00018

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.

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.

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).

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: Concentration range where the instrument provides a linear response.

Measurement Performance Criteria (MPC): Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.

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

ED_006532_00005078-00019

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. The quantitation range 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.

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 environment 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.

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

ED_006532_00005078-00020

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

3.2 Sources

3.3 Exclusions and Exceptions

4.0 MANAGEMENT REQUIREMENTS

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

4.1.5 DoD/DOE (Requirement)

The following shall be implemented in addition to *ISO Clause 4.1.5 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., 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 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 Accreditation Bodies, DOECAP Operations Teams, or to personnel on behalf of DoD/DOE shall be in English.

4.2.3 DoD/DOE (Requirement)

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

Top management shall be responsible for:

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

ED_006532_00005078-00021

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 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.

ED_006532_00005078-00022

4.2.8.2 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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 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 4.2.8.4 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;

ED_006532_00005078-00023

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. 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 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 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)**

4.2.8.5 DoD/DOE (Guidance)

The following is guidance to *TNI 4.2.8.5 a) through f):*

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 to *TNI 4.2.8.5 f) i) through xxiii):*

xxiv) equipment/instrument maintenance;
xxv) computer hardware and software; and
xxvi) troubleshooting.

ED_006532_00005078-00024

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)

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

The following shall be implemented in addition to *TNI 4.5.1 through 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 in support of Environmental Restoration projects must be accredited in accordance with the DoD ELAP. Subcontracted laboratories performing analytical services for the DOE must be approved by the appropriate DOE subcontractor representative.

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 systems elements (such as data review) or outsourced personnel must comply with the laboratory's overall management system, must comply with the requirements of this standard, and are subject to review/approval by the DoD/DOE customer.

ED_006532_00005078-00025

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

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; and
f) Accreditation or certification scopes/certificates.

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., Quality Assurance Project Plan (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)

ED_006532_00005078-00026

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

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. Notification shall be performed according to a written procedure. Records of corrections taken to resolve the nonconformance shall be submitted to the customer(s) in a timely and responsive manner.

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

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

The following shall be implemented in addition to *ISO Clauses and TNI 4.11.1 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.

Approved corrective actions developed to address findings during DoD ELAP or DOECAP assessments must be implemented. Any changes to approved corrective action plans must be approved by the DoD ELAP Accreditation Bodies or the DOECAP Operations Team, as appropriate.

DoD/DOE (Guidance)

The following is guidance to *ISO Clause 4.6.1:*

Willful avoidance of approved corrective action implementation may result in loss of DoD ELAP accreditation or in DOECAP Priority I findings. As a result, work may be discontinued until implementation is verified by the DoD ELAP Accreditation Body or DOECAP Operations Team, 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.

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.

ED_006532_00005078-00027

4.13.3 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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.

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

4.13.4 DoD/DOE (Requirement)

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.

ED_006532_00005078-00028

4.13.10 DOE Only (Requirement)

The laboratory shall have a system in place to record incidents involving spillage of customer samples or significant spillage of chemicals.

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

The following shall be implemented in addition to *ISO Clauses and TNI 4.14.1 through 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.

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 must 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)

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)

ED_006532_00005078-00029

5.0 TECHNICAL REQUIREMENTS

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

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

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 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 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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

ED_006532_00005078-00030

"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 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 is 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)

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.

ED_006532_00005078-00031

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 at a minimum, every 14 days. The data from the analysis of the Storage Blanks shall be available for review.

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

5.3.5 DOE Only (Requirement)

The following shall be implemented in addition to *ISO Clause 5.3.5:*

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

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

5.4.6 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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

ED_006532_00005078-00032

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.

5.4.7.1 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 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.

ED_006532_00005078-00033

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, requirements to be met by the software change, testing, QC, approving, implementing changes, and establishing priority of change requests;
   - 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;

ED_006532_00005078-00034

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)

5.5.5 DoD/DOE (Requirement)

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

h) Date placed in service;
i) Condition when received (e.g., new, used, reconditioned);
j) Operational status;and
k) Instrument configuration and settings.

5.5.13.1 DoD/DOE (Requirement)

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

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

5.5.13.1 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 5.5.13.1 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 SI, International System of Units.

5.5.13.1 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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:

ED_006532_00005078-00035

Performance Check	Frequency	Acceptance Criteria
Balance calibration 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
Verification 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]	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, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	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)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)

ED_006532_00005078-00036

Performance Check	Frequency	Acceptance Criteria
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, the nominal volume is the volume 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; a background reading is taken; and verified with a radiological source]	Per Laboratory SOP

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

5.6.1 and 5.6.2 DoD/DOE (Requirement)

The following shall be implemented in lieu of *TNI sections 5.6.1 and 5.6.2:*

General ISO/IEC 17025:2005(E), Clauses 5.6.1 and 5.6.2 are applicable to this standard.

5.6.4.2 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 5.6.4.2 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 5.6.4.2 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.

ED_006532_00005078-00037

5.6.4.2 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 5.6.4.2 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.

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 to *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.

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

ED_006532_00005078-00038

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).

c) Shipping containers 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. 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)).

d) The laboratory shall have a system in place to record incidents involving spillage of customer samples or significant spillage of chemicals.

5.8.6 DoD/DOE (Requirement)

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

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.

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.

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:

ED_006532_00005078-00039

a) Continued preservation of the sample is in question (e.g., the sample may not be compatible with the preservation); or
b) Deterioration of the preservation is suspected.

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 is remaining.

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.

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:

a) A sample is in someone's custody if:
   i) It is in one's actual physical possession;
   ii) It is in one's view, after being in one's physical possession;
   iii) It has been in one's physical possession and then locked or sealed so that no one can tamper with it; and/or
   iv) It is kept in a secure area, restricted to authorized personnel only.
b) The COC records shall identify all individuals who physically handled individual samples.
c) **DoD/DOE(Guidance)**
   The following is guidance to *TNI 5.8.8 c):*
   In order to simplify record keeping, the number of people who physically handle the sample should be minimized.
d) **DoD/DOE (Guidance)**
   The following is guidance to *TNI 5.8.8 d):*
   It is recommended that a designated sample custodian be appointed to be responsible for receiving, storing, and distributing samples.
e) **DoD/DOE (Guidance)**
   The following is guidance to *TNI 5.8.8 e):*

ED_006532_00005078-00040

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.

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

g) The COC forms shall remain with the samples during transport or shipment.

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

i) **DoD/DOE (Guidance)**

The following is guidance to *TNI 5.8.8 i):*

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

j) 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.

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:

a) Time of day and calendar date of each transfer or handling;

b) Signatures of all personnel who physically handled the samples;

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

d) Common carrier records.

5.8.9 DoD/DOE (Requirement)

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

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.

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

ED_006532_00005078-00041

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.

Further instructions on waste management and disposal are contained in Section 6.4 (DOE Only).

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 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.

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

5.10 DoD/DOE (Requirement)

The following shall be implemented in addition to *ISO clause 5.10:*

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

ED_006532_00005078-00042

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.

5.10.2 DoD/DOE (Requirement)

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

In addition, the name of a contact person and their phone number must also be included in the laboratory information.

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; and
g) Where management system requirements are met, a statement of compliance/noncompliance 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.

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 quantitation range).

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

N – Non-target analyte. The analyte is a tentatively indentified 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).

ED_006532_00005078-00043

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.]

DoD Only (Guidance)
The following is Guidance to DoD/DOE 5.10.3.1.1

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

5.10.5 DoD/DOE (Requirement)
The following shall be implemented in addition to *ISO Clause 5.10.5:*

When included, opinions and interpretations shall only be contained in the case narrative.

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.

5.10.11 DoD/DOE (Requirement)
The following shall be implemented in addition to *TNI 5.10.11 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.

DoD/DOE (Guidance)
The following is guidance to *TNI 5.10.11:*

A practical approach for determining start time follows:

ED_006532_00005078-00044

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 quantitation range.

6.0 Hazardous and Radioactive Materials Management and Health and Safety Practices

(All of Section 6 is a DOE Only Requirement)

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

All DOE sites submitting environmental and waste samples to environmental laboratories shall disclose known or suspected hazards associated with the samples. Based on a good faith effort, 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 receive 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 compliance management of all samples, including regulatory storage and disposal of all samples and associated derived wastes. Some DOE sites permit the return of sample residuals and prior arrangements must be established prior to the receipt of samples. In most cases, derived wastes must be disposed by the laboratory.

6.1 Radioactive Materials Management and Control

6.1.1

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

6.1.2

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

ED_006532_00005078-00045

6.1.3
The laboratory shall have facilities and procedures in place to handle the isotopes, quantity, and physical form of radioactive material specified on the radioactive material license. The laboratory shall ensure adherence to all radioactive materials license and procedural requirements.

6.1.4
The Radiation Safety Officer (RSO) listed in the Radioactive Materials License shall be available to monitor the radioactive materials and control programs and provide rapid response to any radiological emergencies. The laboratory shall have an alternate or backup RSO that shall have the necessary training and experience to perform the duties of the RSO in the event that the RSO is not available.

6.1.5
The laboratory shall have in place a radioactive materials inventory program capable of tracking standards, tracers, and all radiological samples. The radioactive material inventory shall be updated according to the schedule established by laboratory Radioactive 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
Radioactive and mixed wastes shall be segregated from non-radioactive waste.

6.2 Toxic Substances Control Act (TSCA) Material

6.2.1
The laboratory shall comply with all federal regulations governing TSCA materials control and protection.

6.2.2
The laboratory shall segregate all radioactive TSCA materials from all other analytical samples and residues.

6.2.3
The laboratory shall have a procedure for return of radioactive TSCA materials for which there is no commercial treatment or disposal options to the customer.

6.3 Laboratory Safety and Health

6.3.1
The laboratory shall comply with all state and federal regulations governing laboratory health and safety.

6.3.2
A laboratory safety inspection program shall be in place that includes routine inspections of laboratory areas for safety related concerns.

ED_006532_00005078-00046

6.3.3
Chemical hazards labeling on chemical containers shall be in accordance with the laboratory's approved Chemical Hygiene Plan.

6.3.4
On an annual frequency, all visitors, maintenance personnel, and auditors shall have a recorded safety orientation prior to entering the laboratory. All visitors shall be briefed on the safety practices and policies.

6.3.5
The laboratory shall have a Hazardous Waste Operator and Emergency Response (HAZWOPER) trained person on staff. Backup personnel with appropriate training for the Emergency Response (HAZWOPER) trained personnel shall be required.

6.3.6
The laboratory shall have reentry procedures defined in the Emergency Action Plan.

6.4 Waste Management and Disposal

6.4.1
The laboratory shall comply with all federal and state regulations governing waste management and disposal.

6.4.2
The laboratory shall have a waste management plan in place which is capable of:

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

6.4.3
The waste management plan shall include, but not be limited to, the following:

a) Administrative programs to demonstrate compliance for effluent discharges as required by regulatory agencies and applicable DOE Orders;
b) Training procedures, schedules, and management of training records in the areas of waste management, shipping, waste handling, and radioactive materials control;
c) Radioactive volumetric and surface release policies;
d) Permits and licenses to handle hazardous and radioactive waste;
e) Policy or direction on how to conduct waste brokering and Transport, Storage, and Disposal Facility (TSDF) evaluation to ensure proper disposition of waste;
f) Tracking of individual sample container from receipt to final disposition; and

ED_006532_00005078-00047

g) Waste minimization and pollution prevention programs including substitution (when permitted), segregation, and recycling.

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. Reviews of waste brokering and TSDF evaluations shall be performed every three years, unless there are changes in the facilities operations that require the reviews to be conducted on a more frequent basis (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 shall be maintained. A list of the facilities that are approved shall be maintained. Refer to EPA public domain Enforcement and History Online (ECHO) and "envirofacts" websites for information on TSDFs.

6.4.4
The laboratory shall remove or deface all samples container labels prior to container disposal such that they are rendered illegible.

6.4.5
Analytical process waste shall be segregated and removed to a designated storage area to minimize the potential for cross contamination.

6.4.6
Laboratory analysis derived waste characterization shall be repeated at a frequency adequate to account for all known variation in the waste streams.

6.4.7
Samples that are consumed during analysis must be included in the sample accountability tracking.

6.4.8
The laboratory shall have provisions for the disposition of excess samples.

6.4.9
For excess samples that are bulked and drain disposed, the laboratory is aware of the requirements for the receiving Publicly Owned Treatment Works (POTW) or wastewater treatment system and has a program that meets and demonstrates compliance with these requirements

ED_006532_00005078-00048

Volume 1 Module 3: Quality Systems for Asbestos Testing

1.0 Asbestos Testing

1.6 Demonstration of Capability

1.6.2.2 DoD/DOE (Requirement)

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

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

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 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 1.7.1.1.1 b)*:

Use a gold standard grid 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 1.7.1.2.2:*

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

ED_006532_00005078-00049

1.7.1.3.1 DoD/DOE (Requirement)

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

a) Both stereoscope and polarized light microscope must be aligned and checked for function and optimized for correct operation before every use by every analyst.
b) All alignments and function checks must be documented in the proper log book or electronic record.

ED_006532_00005078-00050

Volume 1 Module 4: Quality Systems for Chemical Testing

1.0 Chemical Testing

1.5 Method Validation

1.5.1 DoD/DOE (Requirement)

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

c) The laboratory must evaluate non-standard methods (including laboratory-developed methods) using quality control procedures and acceptance criteria that are consistent with those of similar standard methods or technologies, and must include the following:

   i) Scope;
   ii) Calibration;
   iii) Interferences/Contamination;
   iv) Analyte identification;
   v) Analyte quantitation;
   vi) Selectivity;
   vii) Sensitivity;
   viii) Precision; and
   ix) Bias.

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

e) Methods must be validated when modifications cause changes in stoichiometry, technology, mass tuning acceptance criteria, or quantitation ions to occur.

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 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

ED_006532_00005078-00051

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 1.5.2.1 f):*

f) Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ 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. 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) The LOD shall be verified quarterly. 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. All verification data will be in compliance, reported, and available for review.

ED_006532_00005078-00052

1.5.2.2 DoD/DOE (Requirement)

The following shall be implemented in lieu of *TNI 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.

1.5.2.2 DoD/DOE (Requirement)

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

e) For DoD/DOE, at a minimum, the LOQ shall be verified quarterly. 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.

1.6 Demonstration of Capability (DOC)

1.6.2 DoD/DOE (Requirement)

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

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

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 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.

ED_006532_00005078-00053

1.7.1.1 DoD/DOE (Requirement)

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

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

When sample results exceed the quantitation range, the laboratory shall dilute and reanalyze the sample (when sufficient sample volume and holding time permit) to bring results within the quantitation range. For metals analysis with a single-point calibration, the laboratory may report a sample result above the quantitation range if the laboratory analyzes and passes a CCV that exceeds the sample concentration but is within the linear dynamic range (provided the CCV is analyzed in the same manner as the sample). Results outside the quantitation 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 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 1.7.2 c) i) through iii):*

iv) The concentration of the 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 1.7.2 d):*

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

ED_006532_00005078-00054

1.7.2 DoD/DOE (Requirement)

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

i) 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 spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.

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

iii) If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.

iv) Corrective action(s) and recalibration must occur if the above scenario fails. 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 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 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;

ED_006532_00005078-00055

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

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 reporting 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) **DoD Only (Requirement)** 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.

ED_006532_00005078-00056

1.7.3.3 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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 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 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 shall be noted in the case narrative, or a LCS Duplicate (LCSD) may 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 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 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.

1.7.3.5 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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 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 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

ED_006532_00005078-00057

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 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 and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater;
   ii) The concentration of any common laboratory contaminant in the blank exceeds the LOQ;
   iii) If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. 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 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.

ED_006532_00005078-00058

1.7.4.2 DoD/DOE (Guidance)

The following is guidance to *TNI 1.7.4.2 b):*

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.

ED_006532_00005078-00059

Volume 1, Module 5: Quality Systems for Microbiological Testing

No additions or clarifications were made to Module 5. TNI and ISO/IEC 17025:2005(E) standards shall be followed.

ED_006532_00005078-00060

VOLUME 1, MODULE 6: Quality Systems for Radiochemical Testing

1.0 RADIOCHEMICAL TESTING

1.1 Introduction

1.2 Scope

1.3 Terms and Definitions DoD/DOE (Clarification)

The following is a clarification of *TNI 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.

1.4 Method Selection

1.5 Method Validation

1.5.2.1 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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) 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

ED_006532_00005078-00061

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. Curie can be used to calculate the MDA.

i) With a Blank Population:

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

K = efficiency * $e^{-\lambda t}$ * aliquot fraction * tracer recovery*Yield

T_S = count time of the sample in minutes

s_b = standard deviation of the blank population where the blank population is in net blank counts in count time T_S

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

Identification of blanks to be used in the population:

1. The number of blanks to use in the population;
2. How the blank population changes; and
3. 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.

ED_006532_00005078-00062

ii) Without a Blank Population:

MDA for samples without a blank population can be determined if based on appropriate Curie 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.

iii) 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 aliquant 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:

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

iii) The MDA for the analysis shall be a maximum of 10% of the sample activity.

iv) If sample-specific MDAs are calculated and reported, that shall be clearly stated in the data package.

v) 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.

ED_006532_00005078-00063

1.5.2.1.2 DoD/DOE (Requirement)

a) Decision Level (DL): In the context of analyte detection, the minimum measured value (e.g., of the instrument signal or the analyte concentration) required to give confidence that a positive (nonzero) amount of analyte is present in the material analyzed. The DL is sometimes called the critical level (Lc) or critical value (MARLAP). It is the quantity of analyte at or above which an *a posteriori* decision is made that a positive quantity of the analyte is present. 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) DL Factors and Conditions: DLs are determined *a posteriori* based on sample-specific sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency.

c) DL Calculation: The basic DL calculation shall be based on concepts developed by L. A. Currie, "Limits for Qualitative Detection and Quantitative Determination, *Analytical Chemistry*, March, 1968, Vol. 40, or MARLAP Chapter 20. The following general equation below can be used to calculate the decision level.

d) The DL 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 DL 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

ED_006532_00005078-00064

W = the weight or volume of the sample.

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

DLs for samples without a blank population can be determined if based on appropriate L. A. Currie or MARLAP calculations using a CSU.

1.5.4 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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: All measurement uncertainties shall be propagated and reported with each result. The formula for calculating the Combined Standard Uncertainty (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 2 sigma.

The uncertainty of a count may be estimated as the square root of counts except when there are zero (0) counts. In the case of zero (0) counts, the uncertainty of the count is assumed to be the square root of one count.

Systematic Error shall include, but is not necessarily limited to:

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.

1.7 Technical Requirements

1.7.1 DoD/DOE (Requirement)

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

a) Initial Calibration:
   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 prepared from

ED_006532_00005078-00065

NIST/international traceable standards, they shall be "working reference materials" as defined in ASTM C1128 (current version).

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.

x) Check sources shall be used only to verify that efficiencies have not changed. They shall not be used to determine efficiencies.

1.7.1 DoD/DOE (Requirement)

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

b) Instrument Calibration Verification (Performance Checks)

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 Full-Width-Half-Maximum (FWHM) resolution of the alpha or gamma detector shall be evaluated prior to instrument use and following repair or loss of control (MARLAP 18.5.6.2). The measured FWHM resolution shall be trended.

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

It is important to use calibration or QC sources that will not cause detector contamination from recoil atoms from the source.

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 1.7.1 c)* including *I through iv):*

c) Background Measurement

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.

Successive long background measurements may be evaluated as background check measurements.

Low levels of contamination not detected in a shorter background counting time may bias the results of sample analyses. The duration of the background check measurement shall be of sufficient duration (i.e., at least as long as the sample

ED_006532_00005078-00066

count time) to quantify contamination that may impact routine sample measurements.

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

If the background check is conducted less frequently than daily, any associated sample results shall not be released for use until a (bracketing) background check is measured and has met all acceptance criteria. 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.

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

i) For gamma spectroscopy systems, long background measurements (to be used for background corrections) shall be performed on at least a monthly basis. The duration of the background measurement shall be sufficient to quantify contamination that may affect routine sample measurements (the count time for the background measurement shall be at least as long as the sample count time.)

ii) For alpha spectroscopy systems, monthly background determinations shall be performed for each Region of Interest (ROI). The duration of the background measurement shall be sufficient to quantify contamination that may affect routine sample measurements.

   Backgrounds for alpha spectrometers should be rechecked after being subjected to high-activity samples. Labs must have procedures in place to define high activity and counting procedures to check for gross contamination from high activity samples.

iii) For gas-proportional counters, long background measurements (to be used for background corrections) shall be performed on a monthly basis, at minimum.

   Backgrounds for gas flow proportional counters should be rechecked after being subjected to high-activity. Labs must have procedures in place to define high activity.

iv) For scintillation counters, the duration of the background measurement shall be sufficient to quantify contamination that may affect routine sample measurements.

   The daily instrument check shall include a check with an unquenched, sealed background vial (which should never be used to correct sample results for background measurements, since it is not in the same configuration as samples).

ED_006532_00005078-00067

1.7.2 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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 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 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.
The batch blank matrix shall be the same as the samples, as can be reasonably achieved, and shall be documented in the Case narrative

e) Blank Acceptance Criteria:
A method blank shall be one per preparatory batch. (MARLAP 18.4.1)
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 ±3 σ of the mean.
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.

f) The following batch blank matrices shall be used for all radiochemistry analyses:
   i) Distilled or deionized water, radon free;
   ii) Characterized solid material representative of the sample matrix;
   iii) Filters, physically and chemically identical filter media, analyte free (if supplied to the laboratory by customer).

ED_006532_00005078-00068

1.7.2.2 DoD/DOE (Requirement)

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

j) The LCS shall be counted for a sufficient time to meet the required detection limit.

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 ± 3 σ 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 be of the same element as the sample analyte and shall be at least five times, but not greater than 20 times the RL with the following exceptions:

   i) 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.

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

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

n) LCS shall be traceable to the NIST or accepted international standard, or shall be a working reference material as described in ASTM C 1128 (current version), and may be used repeatedly for different analytical batches as long as it is appropriate for the matrix and geometry of the batch.
The analyte need not be the same as the sample analyte, but shall fall in the approximate energy region of the spectrum as the analyte(s) (i.e., low, mid-range, or high energy).

1.7.2.3 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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 within the control limits of 60 - 140%, or as specified by client. Matrix spike samples for

ED_006532_00005078-00069

which the sample activity is greater than five times the spiking level are not required to meet this criterion. If activity of the MS > 5 times the unspiked sample, use $|Z_{MS}| \leq 3$. (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.

xiii) Counting: The matrix spike shall be counted for a sufficient time to meet the required detection limit.

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

1.7.2.3 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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:

$|Z_{Dup}| \leq 3$ (MARLAP 18.4.1) if using MARLAP; or the duplicate error ratio (DER) between the sample and the duplicate is <3; or 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 subsampling shall be described in the case narrative.

ED_006532_00005078-00070

1.7.2.3 DoD/DOE (Requirement)

The following shall be implemented in addition to *TNI 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.

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

The chemical yield for each sample determined using an indirect yield measurement method shall fall within the range 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.

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

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

ii) Spectral resolution requirements are met and there are no indications of spectral interferences; and

iii) Detection limit requirements are met.

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

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

1.7.2.3 DoD/DOE (Requirement)

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

d) Carrier

ED_006532_00005078-00071

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.

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

The chemical yield for each sample determined using an indirect yield measurement method shall fall within the range 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.

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

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

ii) Spectral resolution requirements are met and there are no indications of spectral interferences; and

iii) Detection limit requirements are met.

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 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:

ED_006532_00005078-00072

i) DOE / EH - 0173T "Environmental Regulatory Guide for Radiological Effluent Monitoring and Environmental Surveillance, January 1991.

ii) 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 1.7.2.5 a) through c):*

d) Water purity shall be at least distilled or deionized water.

i) Standards shall be verified prior to initial use.

Preparations of standards solutions used for a period of time exceeding one year shall be verified annually, at a minimum, and documented in a logbook.

At least three verification measurements of a standard shall be used to determine the mean value and standard deviation of the verification results.

The mean value shall be within 5% of the decay corrected certified value.

The 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.

If all criteria are met, the certified value shall be used.

ii) 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 1.7.2.7 a) through c):*

d) The detection/quantification requirements for contamination control sampling should be consistent with the lowest level of sample analyte or MDA equivalent. Samples shall be segregated by activity levels in sample receipt, processing areas, and storage areas.

1.8 Method Specific Directions DoD/DOE (Requirements)

The following shall be implemented in addition to *TNI 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 used for alpha spectrometry shall be tested by the laboratory for contribution in the ROIs of the analytes of interest. All tracers shall be of the same element or of an element with the same chemistry for the separations. If a significant contribution is found, the method for correction shall be site accepted prior to use.

ED_006532_00005078-00073

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 noted.

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:

      1. If the tracer recovery for the sample does not fall within 30% - 110%, reanalysis is required, beginning with preparation.
      2. If the FWHM for the tracer peak exceeds 100 keV and/or the peak energy does not fall within ± 40 keV of the known peak energy, reanalysis is required.
      3. 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.
      4. 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.

f) Efficiency determination and background determination for each ROI. Alpha spectrum regions of interest shall be selected with consistency from analyte to analyte.

g) Energy Calibration:

   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.

ED_006532_00005078-00074

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

Detector response (efficiency) is not used in the calculation of results when tracers are used in the analysis, but only used to calculate the estimated yield, which is also not used, except as a general method performance indicator.

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 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.

ED_006532_00005078-00075

1.8.2 Radon Scintillation (Lucas Cell)

a) Procedures for sample analyses by Lucas Cell shall incorporate and adhere to ASTM D3454 (current version), Standard Test Method for Radium-226 in Water. Where the word "should" is used in ASTM D3454, performance shall be in accordance with the statement unless otherwise provided in this document. Reference is to the current version of the method. When references are updated, an implementation schedule shall be determined by the lab.

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.

e) When consistent with MQO, Rn-222 ingrowth times may be shortened to the degree permitted by EPA Method 903.1

1.8.3 Liquid Scintillation Counting

a) Tritium in Water: Water samples for tritium analysis and all associated QC samples shall be distilled prior to analysis unless specified otherwise by the client. The applicable preparation SOP shall specify the fraction to be collected. The same fraction shall be collected for samples and all associated QC samples.

b) Counting Vial Preparation: Samples shall be counted in vials equivalent to or superior to low potassium glass vials or high density polyethylene vials. Samples in polyethylene vials shall be counted within a time period not to exceed the manufacturer's specification for the cocktail used in the analysis. Analysis documentation shall contain sufficient information for this to be verified. Vials shall be prepared according to manufacturer's specification for the cocktail. The vials shall be "dark adapted" for a minimum of 30 minutes or according to the cocktail manufacturer's specifications before counting. The prepared vials shall be inspected to verify that the sample loaded properly in the cocktail.

c) Laboratory SOPs for methods using liquid scintillation counting shall incorporate and adhere to ANSI N42.15-1997 (or latest version), American National Standard Check Sources for and Verification of Liquid Scintillation Systems. 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. 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

ED_006532_00005078-00076

from a running average of background counts or a background quench curve, the most recent background count shall be used to calculate sample activities and MDAs. This is not a performance check, rather a background subtraction sample in a configuration equivalent to that of associated samples in the batch. It is used to generate the background subtraction data for the batch (using the results associated directly with that batch, results of a rolling mean, or background quench curve). The effect of quench on background shall be evaluated and corrected using a background quench curve if it is significant.

e) For analysis methods using quench curves to determine individual sample detection efficiency or background, the quench curves shall be generated at least yearly and verified after any instrument maintenance.

f) If the calibration method is constant quench, the detection efficiency shall be checked at least weekly when in use or with each counting batch.

g) Sample-Specific Conditions: The following are conditions that require reanalysis for a particular sample and analyte, beginning with the preparation or recounting, as appropriate.

   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: 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) Instrument Calibration: Shall be performed in accordance with the requirements in ANSI N42.25, Calibration and Usage of Alpha/Beta Proportional Counters.

ED_006532_00005078-00077

Where the word "should" is used in ANSI N42.25, calibration shall be performed in accordance with the statement. References are for the current version. When references change, an implementation schedule 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/crosstalk curves shall be the same as that of the prepared sample and QC sample planchets. The depth and shape (flat, flanged, ringed, etc.), in addition to the diameter, are factors that shall be the same for calibration sources as for samples.

   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 Crosstalk Curves:

   i) Self-absorption curves are required for both alpha and beta counting.

   ii) A crosstalk curve shall be established for alpha to beta crosstalk versus residue weight.

   iii) Beta to alpha crosstalk is not significantly affected by planchet residue weight, and is generally constant over the applicable weight range. Therefore, this crosstalk correction does not require residue weight consideration.

   iv) The data used to generate self-absorption and crosstalk 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 crosstalk of each detector used to count analytical samples or QC samples shall be checked daily with separate alpha and beta emitting sources. The only

ED_006532_00005078-00078

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) SOPs for sample analysis by gamma spectrometry shall incorporate and adhere to ANSI N42.14-1991 (or latest version), Calibration and Use of Germanium Spectrometers for the Measurement of Gamma Ray Emission Rate of Radionuclides, and/or ANSI N42.12-1994 (or latest version), Calibration and Usage of Thallium-Activated Sodium Iodide Detector Systems for Assay of Radionuclides. References are for the current version. When references change, an implementation schedule will be determined.

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 regard to attaining RLs, 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 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. This applies to Ra-226 soil samples only.

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 traceable NIST or accepted international standards or prepared from NIST/international traceable sources.

ED_006532_00005078-00079

1) Germanium Detectors: Refer to ANSI N42.14 for guidance on isotope specific efficiency and efficiency as a function of energy calibrations. The efficiency calibration measurements shall be at least 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).

Sodium Iodide Detectors: 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.

i) Germanium Detectors: Refer to ANSI N42.14, Section 5.1 for guidance on calibrating gamma-ray energy as a function of channel number at a fixed gain. The energy calibration measurements shall be made using at least 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.

ii) At least 10,000 net counts (total counts minus the Compton continuum and ambient background) shall be accumulated in each full-energy gamma-ray peak of interest (ASTM D 3649-98a).

iii) Energy calibration shall be linear and accurate to 0.5 keV.

iv) Sodium Iodide Detectors: Refer to ANSI N42.12, Section 4.3.2.

ED_006532_00005078-00080

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.

1.8.6 Conditions Requiring Reanalysis or Recount

If reanalysis is not possible, the client shall be contacted for specific guidance or requirements.

a) General Conditions:

i) If the RLs could not be achieved because of laboratory errors or oversights such as inadequate count times, inadequate aliquot size, inappropriate dilution, low detector efficiencies, high detector backgrounds, etc., then the sample shall be reanalyzed under more optimal conditions.

ii) If the RLs could not be achieved because of problems associated with the sample such as inadequate sample provided, elevated radioactivity levels, sample matrix interferences such as high amounts of suspended solids, multiphase liquids, etc., then such problems shall be explained in the Case narrative.

b) Sample and Analyte-Specific Conditions: Any one of the following are additional conditions that require reanalysis for a particular sample and analyte:

i) If, for any reason, sample or batch QC integrity becomes suspect (e.g., spillage, mis-identification, cross-contamination), all potentially affected samples shall be reanalyzed from a point before that at which the integrity came into question. If new batch QC must be prepared for reanalysis, samples for reanalysis shall be restarted at the normal point of initiation for the batch QC.

ii) All samples associated with expired standards.

c) Analytical Batch Conditions: Except where noted otherwise, any one of the following conditions requires reanalysis of the entire analytical batch, beginning with the preparation: batches that failed the Method Blank or LCS criteria.

d) Conditions Requiring a Re-count: If the RL was not achieved due to inadequate count duration, low detector efficiencies, or high detector backgrounds, the sample shall be re-counted under more optimal conditions, and the reasons for the re-count shall be documented in the case narrative.

ED_006532_00005078-00081

Volume 1, Module 7: Quality Systems for Toxicity Testing

No additions or clarifications were made to Module 7. TNI and ISO/IEC 17025:2005(E) standards shall be followed.

ED_006532_00005078-00082

Appendix A: Reporting Requirements

In the absence of client specified reporting criteria, the reporting requirements outlined below 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: cover sheet, table of contents, case narrative, analytical results, sample management records, and Quality Assessment/Quality Control (QA/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:

- Title of report (i.e., test report, test certificate);
- Name and location of laboratory (to include a point of contact, phone and facsimile numbers, and e-mail address);
- Name and location of any subcontractor laboratories, and appropriate test method performed (information can also be located in the case narrative as an alternative);
- Unique identification of the report (such as serial number);
- Client name and address;
- Project name and site location;
- Statement of data authenticity and official signature and title of person authorizing report release;
- 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
- 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:

- Describe any abnormalities and deviations that may affect the analytical results;
- 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
- Provide a summary of samples included in the report with the methods employed in order to assist the user in interpretation.

ED_006532_00005078-00083

The case narrative shall provide (Information need not be repeated if noted elsewhere in the data package):

- 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;
- A list of samples that were received but not analyzed;
- Date of samples received;
- Sample preservation or condition at receipt;
- A description of extractions or analyses that are performed out of holding times;
- A definition of all data qualifiers or flags used;
- 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;
- Identification of multiple sample runs with reason(s) identified (e.g., dilutions or multiple cleanups);
- Identification of samples and analytes for which manual integration was necessary; and
- 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: (Information need not be repeated if noted elsewhere in the data package):

- Project name and site location;
- Field sample ID number as written on custody form;
- Laboratory sample ID number;
- Preparation batch number(s);
- Matrix (soil, water, oil, air, etc.);
- Date and time sample collected;
- Date and time sample prepared;
- Date and time sample analyzed;
- Method numbers for all preparation, cleanup, and analysis procedures employed;
- Analyte or parameter with the Chemical Abstracts Service (CAS) Registry Number if available;
- Sample aliquot analyzed;

ED_006532_00005078-00084

- Final extract volume;
- Identification of analytes in which manual integration occurred, including the cause and justification;
- Analytical results with correct number of significant figures;
- 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);
- Any data qualifiers assigned;
- Concentration units;
- Dilution factors;
- All multiple sample run results shall be reported;
- Percent moisture or percent solids (all soils are to be reported on a dry weight basis); and
- 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:

- Chain-of-custody records;
- Shipping documents;
- 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);
- Telephone conversation or e-mail records associated with actions taken or quality issues; and
- Records of sample compositing done by the laboratory.

6.0 QA/QC Information

The minimum laboratory internal QC data package shall include:

- Method blank results;
- Percent recoveries for Laboratory Control Sample (LCS), Laboratory Control Sample Duplicates (LCSD), Matrix spike (MS), and Matrix Spike Duplicates (MSD);
- MSD or matrix duplicate Relative percent differences (RPD);
- Surrogate percent recoveries;
- Tracer recoveries;
- Spike concentrations for LCS, MS, surrogates;
- QC acceptance criteria for LCS, MS, surrogates;

ED_006532_00005078-00085

- Post-Digestion Spike (PDS) recoveries;
- In-house or project specified LCS control limits, as applicable;
- Serial dilutions (SD) percent difference; and
- Batch numbers (preparation, analysis, and cleanup).

7.0 Data Reports for Third Party Review or Validation

The data validation guidelines established in other Department of Defense/Department of Energy 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.

DoD data validation guidelines defines the minimum reporting requirements for each stage (formerly level) of data package as outlined below.

- A cover sheet, table of contents, and case narrative including all of the information specified in the above sections are required for all stages of data reports.
- **Stage 1**: Analytical results, Sample Management Records.
- **Stage 2**: **Stage 1** reporting requirements plus QA/QC Information, Instrument QA/QC Information, Instrument and Preparation logs.
- **Stage 3**: **Stage 2** reporting requirements plus Instrument Quantitation Reports.
- **Stage 4**: **Stage 3** reporting requirements plus Instrument Chromatograms and Spectra.
- **In addition**, standards traceability should be included in Stages 3 and 4 if a legal chain of custody is required.

ED_006532_00005078-00086

Appendix B: Quality Control Requirements

Table – 1. Organic Analysis by Gas Chromatography					
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.

ED_006532_00005078-00087

Table – 1. Organic Analysis by Gas Chromatography					
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.
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.

ED_006532_00005078-00088

Table – 1. Organic Analysis by Gas Chromatography					
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 Pesticides multi-component analytes (i.e. Toxaphene, Chlordane), 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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB 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.

ED_006532_00005078-00089

Table – 1. Organic Analysis by Gas Chromatography					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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 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.	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 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.

ED_006532_00005078-00090

Table – 1. Organic Analysis by Gas Chromatography					
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 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 with surrogate is present, reanalysis may not be necessary.	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 positive results must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or 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.

ED_006532_00005078-00091

Table – 2. Organic Analysis by High-Performance Liquid Chromatography (HPLC)					
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.	NA.	NA.	Calculated for each analyte and surrogate.
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.

ED_006532_00005078-00092

Table – 2. Organic Analysis by High-Performance Liquid Chromatography (HPLC)					
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 established RT windows. All reported analytes and surrogates within ± 15% true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. 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 > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB 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.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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.

ED_006532_00005078-00093

Table – 2. Organic Analysis by High-Performance Liquid Chromatography (HPLC)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the 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.	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 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.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use 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 with surrogate is present, reanalysis may not be necessary.	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.

ED_006532_00005078-00094

Table – 2. Organic Analysis by High-Performance Liquid Chromatography (HPLC)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Confirmation of positive results (second column)	All positive results 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.

ED_006532_00005078-00095

Table – 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.	Laboratory must have a procedure to determine when the sample is dry to constant mass. Record date, time, and ambient temperature on a daily basis while drying samples.	NA.	Flagging is not appropriate.	Commercial PT samples must reflect the grinding, extraction, and analysis steps as a minimum.
Soil sieving procedure	Each sample, LCS, and Method Blank.	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Do not intentionally include vegetation in the portion of the sample that passes through the sieve unless this is a project specific requirement. Collect and weigh any portion unable to pass through the sieve.	NA.	Flagging is not appropriate.	

ED_006532_00005078-00096

Table – 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 grinding procedure	Initial demonstration.	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.	
Soil grinding blank	Prior to grinding samples; after every 10 samples; and 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 a field sample. 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 any individual grinding blank is found to exceed the acceptance criteria, apply B-flag to the samples following that blank.	Grinding blanks may be composited for analysis. At least one grinding blank per batch must be analyzed.
Soil subsampling process	Each sample, duplicate, LCS, and Method Blank.	Entire ground sample is mixed, spread out on a large flat surface (e.g., baking tray), and 30 or more randomly located increments are removed from the entire depth to sum a ~10 g subsample.	NA.	Flagging is not appropriate.	

ED_006532_00005078-00097

Table – 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, one sample per batch. Cannot be performed on any sample identified as a blank (e.g., trip blank, field blank, method blank).	Three 10 g subsamples are taken from a sample expected to contain the highest levels of explosives within the quantitation range of the method. 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 if acceptance criteria are not met and explain in the case narrative.	
Aqueous sample preparation	Each sample and associated batch QC samples.	Solid phase extraction (SPE) using resin-based solid phase disks or cartridges is required.	NA.	Flagging is not appropriate.	The salting-out procedure is not permitted.
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.

ED_006532_00005078-00098

Table – 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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. 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.

ED_006532_00005078-00099

Table – 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 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, 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. 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 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, therefore is taken post grinding from same ground sample as parent subsample is taken. 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.

ED_006532_00005078-00100

Table – 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 Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the 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.	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use 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 with surrogate is present, reanalysis may not be necessary.	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.

ED_006532_00005078-00101

Table – 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 positive results 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.

ED_006532_00005078-00102

Table – 4. Organic Analysis by Gas Chromatography/Mass Spectrometry					
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, 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 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.

ED_006532_00005078-00103

Table – 4. Organic Analysis by Gas Chromatography/Mass Spectrometry					
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.	Required 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	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.

ED_006532_00005078-00104

Table – 4. Organic Analysis by Gas Chromatography/Mass Spectrometry					
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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. 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.	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.	

ED_006532_00005078-00105

Table – 4. Organic Analysis by Gas Chromatography/Mass Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB 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.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the 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. 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.

ED_006532_00005078-00106

Table – 4. Organic Analysis by Gas Chromatography/Mass Spectrometry					
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 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.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use 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 with surrogate is present, reanalysis may not be necessary.	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.

ED_006532_00005078-00107

Table - 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.	
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.

ED_006532_00005078-00108

Table - 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.

ED_006532_00005078-00109

Table - 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.
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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 calibration verification. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

ED_006532_00005078-00110

Table - 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**
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.	
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.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, re-prep and reanalyze MB 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.

ED_006532_00005078-00111

Table - 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**
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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.	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 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 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.	A laboratory must use the 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 criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

ED_006532_00005078-00112

Table - 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, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	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 with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	

ED_006532_00005078-00113

Table - 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. 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.	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).

ED_006532_00005078-00114

Table - 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.

ED_006532_00005078-00115

Table - 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; <u>and</u> 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; <u>and</u> 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, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. <u>End-of-run CCV</u>: 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	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 calibration verification. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

ED_006532_00005078-00116

Table - 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)			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.	
Method Blank (MB)	One per preparatory batch, run after calibration standards and before samples.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. 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.

ED_006532_00005078-00117

Table - 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**
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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 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. 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.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the 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.

ED_006532_00005078-00118

Table - 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**
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%.	Correct problem, then re-prep and reanalyze the samples with failed IS.	Apply Q-flag to results of all affected samples.	
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.	
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.

ED_006532_00005078-00119

Table – 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.
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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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.

ED_006532_00005078-00120

Table – 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.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB 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 reprepped or reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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 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.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.

ED_006532_00005078-00121

Table – 7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA)					
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 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.
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.

ED_006532_00005078-00122

Table – 8. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry					
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.

ED_006532_00005078-00123

Table – 8. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry					
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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low-level ICV)	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 (LLICV). Low-level calibration check standard should be less than or equal to the LOQ.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. 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.

ED_006532_00005078-00124

Table – 8. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. 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 < LOD (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.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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 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.	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.

ED_006532_00005078-00125

Table – 8. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry					
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 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.
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.

ED_006532_00005078-00126

Table – 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.

ED_006532_00005078-00127

Table – 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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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.
Low-level Calibration Check Standard (Low Level ICV)	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. Low-level calibration check standard should be less than or equal to the LOQ.

ED_006532_00005078-00128

Table – 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.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. 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.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.

ED_006532_00005078-00129

Table – 9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
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 < LOD (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.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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 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.	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.

ED_006532_00005078-00130

Table – 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 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.
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.

ED_006532_00005078-00131

Table – 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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

ED_006532_00005078-00132

Table – 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 > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. 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 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 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.
Matrix Spike (MS)	Once per preparatory batch.	A laboratory must use the 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.	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.

ED_006532_00005078-00133

Table – 10. Inorganic Analysis by Colorimetric Hexavalent Chromium					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
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 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.
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.	
Post-digestion Matrix Spike (solid matrix samples)	One per preparatory batch.	Recovery within 85 - 115%.	No specific corrective action, 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.

ED_006532_00005078-00134

Table – 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 std 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.

ED_006532_00005078-00135

Table – 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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. 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 cyanide results 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.

ED_006532_00005078-00136

Table – 11. Cyanide Analysis					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run; after every 10 field samples; and at end of the analysis sequence. (After ICV and each CCV).	No cyanide detected > LOD.	Correct problem and reanalyze all samples analyzed since the last acceptable calibration blank.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the 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.	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.

ED_006532_00005078-00137

Table – 11. Cyanide Analysis					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike Duplicate (MSD) and Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the 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.
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.

ED_006532_00005078-00138

Table – 12. Common Anions Analysis by 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.	Established 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.

ED_006532_00005078-00139

Table – 12. Common Anions Analysis by 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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. 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 > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB 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.

ED_006532_00005078-00140

Table – 12. Common Anions Analysis by 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 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 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. 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 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.

ED_006532_00005078-00141

Table – 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.

ED_006532_00005078-00142

Table – 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.

ED_006532_00005078-00143

Table – 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.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
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.

ED_006532_00005078-00144

Table – 13. Perchlorate by Mass Spectrometry Methods					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
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.
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.

ED_006532_00005078-00145

Table – 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 a valid reagent blank. 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 > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. 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.

ED_006532_00005078-00146

Table – 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 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.
Matrix Spike (MS)	One per preparatory batch per matrix.	A laboratory must use the 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. 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.

ED_006532_00005078-00147

Table – 13. Perchlorate by Mass Spectrometry Methods					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike Duplicate (MSD) or Laboratory Duplicate (LD)	One per preparatory batch per matrix.	A laboratory must use the 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.

ED_006532_00005078-00148

Table – 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.
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.

ED_006532_00005078-00149

Table – 14. Chemical Warfare Agents by GC/MS					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
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 prep batch.	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 prep batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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 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.	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.	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.	

ED_006532_00005078-00150

Table – 14. Chemical Warfare Agents by GC/MS					
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 > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB 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.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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. 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.

ED_006532_00005078-00151

Table – 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.	A laboratory must use the 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. 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. 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 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. 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.

ED_006532_00005078-00152

Table – 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 use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed. 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 with surrogate is present, reanalysis may not be necessary.	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.

ED_006532_00005078-00153

Table - 15. Perfluorinated Compounds by Liquid Chromatography/Mass Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial Calibration (ICAL) for all analytes and surrogates	Minimum of 5 calibration standards to establish linearity at method set-up and after major maintenance.	Each calibration point for each analyte must calculate to be within 75-125%, except the lowest cal point which must calculate to within 70-130%.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples may be run until ICAL has passed. Calibration can be linear (5 standards) or quadratic (6 standards); weighting is allowed.
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 and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging is not appropriate.	No samples may be run until calibration has been verified.
Continuing Calibration Verification (CCV)	Analysis of mid-level standard after every 10 field samples. All samples must be bracketed by the analysis of a standard demonstrating that the system was capable of accurately detecting and quantifying perfluorinated compounds.	All reported analytes and surrogates within ± 25% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected 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.	Flagging is only appropriate in cases where the samples cannot be reanalyzed.

ED_006532_00005078-00154

Table - 15. Perfluorinated Compounds by Liquid Chromatography/Mass Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Internal Standard (IS)	Addition of isotopically labeled analytes to every sample, batch QC sample, standard, instrument blank, and method blank.	Determine that the absolute areas of the quantitation ions of the IS(s) are within 50-150% from the average areas measured during initial calibration.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. For failed QC samples, correct problem, and rerun all associated failed field samples.	Apply Q-flag and discuss in the case narrative.	Failing internal standard should be thoroughly documented in the case narrative.
Tune Check	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standard must contain analytes of interest or appropriate substitute. Mass assignments of tuning standard within 0.5 amu of true value.	Retune instrument. If the tuning will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Flagging is not appropriate.	Problem must be corrected. Sample analysis shall not proceed without acceptable tuning.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB 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.

ED_006532_00005078-00155

Table - 15. Perfluorinated Compounds by Liquid Chromatography/Mass Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the 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. If in-house limits do not exist, use 70-130% until limits are established.	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.
Matrix Spike (MS)	One per preparatory batch per matrix.	A laboratory must use the 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. If in-house LCS limits do not exist, use 70-130% until limits are established.	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.	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.

ED_006532_00005078-00156

Table - 15. Perfluorinated Compounds by Liquid Chromatography/Mass Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch per matrix.	A laboratory must use the 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. If in-house LCS limits do not exist, use 70-130% until limits are established. MSD or MD: RPD of all analytes ≤ 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.	The data shall be evaluated to determine the source of error. Analyze MS/MSD for low concentration samples and Sample/MD for high concentration samples.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed. Limits may be set at 70-130% until sufficient data has been generated to establish in-house control limits.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. For failed QC samples, correct problem, and rerun all failed samples.	Apply Q-flag and discuss in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

ED_006532_00005078-00157

Table – 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 Two calibration peaks that are: 1) ≥700 keV apart; or 2) that bracket all peaks to be determined. 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.

ED_006532_00005078-00158

Table – 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 Observed peak centroid falls within ±20 keV from reference energy for each peak used in the initial energy calibration. FWHM ≤100 keV and within ±20 keV of corresponding calibration peaks in initial energy calibration.	Repeat ICV to check for error. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate.	Flagging criteria are not appropriate.	Use a 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 with a second source.
Continuing Calibration Verification (CCV) **(Pulsar check)**	Pulsar energy verification weekly, prior to analysis of samples. Use either Pulsar 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.	Recount and check control chart for trends. Determine cause, correct problem, and repeat CCV and all associated samples since last successful CCV.	Flagging criteria are not appropriate.	Pulsar check can be used to verify energy calibration when using radiotracers during analysis. No samples may be run until calibration has been verified.

ED_006532_00005078-00159

Table – 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 Pulsar 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 Observed peak centroid falls within 20 keV from reference energy for each peak used in the initial energy calibration. FWHM ≤100 keV and within 30 keV of corresponding calibration peaks in initial energy calibration.	Recount and check control chart for trends. Determine cause, correct problem, and repeat CCV and all associated samples since last successful CCV.	Flagging criteria are not appropriate.	Source check can be used to verify energy, FWHM and efficiency. No samples may be run until calibration has been verified.
Background Subtraction Count (BSC) Measurement	Prior to initial use or after initial calibration and monthly. (MARLAP 18.5.6.3)	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 where count rate <10 times that in the affected ROI(s) in the BSC.	BSC test source matches STS configuration (type, size and position relative to the detector). Activity must meet project objectives.

ED_006532_00005078-00160

Table – 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. Count duration ≥ longest STS count.	Z_{Blank} ≤3 for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1)	Check control chart for trends and recount. Determine cause and correct problem. If background activity has changed, re-establish BSC and reanalyze all infected samples.	If reanalysis cannot be performed, apply Q-flag to all affected result since last acceptable ICC where the STS count rate in the impacted ROI is ≤5 times that of STS. Explain in the case narrative.	
Method Blank (MB)	One per preparatory batch. (MARLAP 18.4.1)	$\|Z_{Blank}\| \leq 3$. 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. 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. 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. With project approval and appropriate qualification and narration, report results with a count rate >5 times that of the affected ROI in the MB.

ED_006532_00005078-00161

Table – 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.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. 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.	Results may not be reported without a valid LCS. Qualification is only appropriate in cases where the samples cannot be reanalyzed. 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. Acceptance criteria for LCS recovery may be specified by the project.
Matrix Spike (MS)	One per preparatory batch. (*MS not required when chemical yield tracers or carriers are employed).*	If activity of the MS > 5 times the unspiked sample, 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.	The data shall be evaluated to determine the source of difference.

ED_006532_00005078-00162

Table – 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.

ED_006532_00005078-00163

Table -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.	Traceable calibration source (CS) 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. No samples may be run until all calibration criteria are met.

ED_006532_00005078-00164

Table -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.	Traceable second-source standard 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.

ED_006532_00005078-00165

Table -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 not longer than a week.	Verify peak shift within tolerance limit; verify efficiency within control parameters; verify resolution in tolerance limit. Response checks shall have a tolerance limit or control chart set at ± 3% or 3σ of the mean. (MARLAP 18.5.6.2) or Peak Energy/Efficiency: low, mid, and high energies within 10% of the initial calibration value; FWHM: low, mid, and high energies within 10% of initial FWHM value.	Correct problem, rerun CCV. 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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

ED_006532_00005078-00166

Table -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)	Recount and check control chart for trends. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC and reanalyze or qualify all impacted samples since last acceptable BSC.	Apply B-flag to all results for specific nuclide(s) in all samples associated with the blank.	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)
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)	Recount the background. If still out of control, locate and correct problem; reanalyze or qualify all impacted samples since last acceptable ICC. If background activity has changed, re-establish BSC and reanalyze samples.	If corrective action fails, apply Q-flag to all results for specific nuclide(s) in all samples associated with the BSC.	Integrate spectrum from ~50 - 2,000 keV to check for gross contamination. (MARLAP 18.5.6.2)

ED_006532_00005078-00167

Table -17. Gamma Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per preparatory batch.	$\lvert Z_{Blank} \rvert \leq 3$ for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1) or No analytes detected > 2 times the blank Combined Standard Uncertainty (CSU). Blank result must not otherwise affect sample results.	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.	The results of method blanks typically 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.) Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

ED_006532_00005078-00168

Table -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. Qualification is only appropriate in cases where the samples cannot be reanalyzed.
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.

ED_006532_00005078-00169

Table – 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 and after loss of control. (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.	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.	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.

ED_006532_00005078-00170

Table – 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.	Determine crosstalk factors for each nuclide, matrix and method. For mass loaded test sources, determine crosstalk factors for the nuclide as a function of test source mass. No samples may be run until cross talk calibration criteria are met.
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.	Minimum of seven mass attenuated standards. No samples may be run until mass attenuation calibration criteria are met.

ED_006532_00005078-00171

Table – 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.	Correct problem and verify second source standard. Rerun IECV. If that fails, correct problem and repeat ICALE.	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.
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)	Within tolerance or control chart limits ± 3% or 3σ of the mean.	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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

ED_006532_00005078-00172

Table – 18. Gas Flow Proportional Counting					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per preparatory batch.	$\lvert Z_{Blank} \rvert \leq 3$ for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1) or No analytes detected > 2 times the blank Combined Standard Uncertainty (CSU). Blank result must not otherwise affect sample results.	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. 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.
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 and correct problem. If background activity has changed, re-establish BSC. All samples following the last acceptable background measurement must be reanalyzed.	If reanalysis of samples is not possible, apply B-flag to all results in all samples associated with the failed blank.	Detector background measured using a contamination-free source mount. Activity must meet project requirements.

ED_006532_00005078-00173

Table – 18. Gas Flow Proportional Counting					
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.	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).	Recount the background. If still out of control, locate and correct problem; reanalyze or qualify all impacted samples since last acceptable ICC. If background activity has changed, re-establish BSC and reanalyze samples.	If corrective action fails, apply Q-flag to all results for specific nuclide(s) in all samples associated with the BSC.	Develop detector response control chart immediately after calibration and loss of control.
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.

ED_006532_00005078-00174

Table – 18. Gas Flow Proportional Counting					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike (MS)	One per preparatory batch. *(MS not required when yield tracers are employed)*	If activity of the MS > 5 times the unspiked sample, $\lvert Z_{MS} \rvert \leq 3$. (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.	The data shall be evaluated to determine the source of difference.
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.	Recovery (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.

ED_006532_00005078-00175

Table – 18. Gas Flow Proportional Counting					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
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.

ED_006532_00005078-00176

Table - 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 appropriate reference 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.
Method Calibration (QCAL) **(Quench curve)**	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.	When establishing a quench curve, a minimum of five calibration sources of different quench factors shall be used. No samples may be run until calibration criteria are passed.

ED_006532_00005078-00177

Table - 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.	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.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Use a second source standard for each nuclide. No samples may be run until calibration has been verified.

ED_006532_00005078-00178

Table - 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 should have a tolerance limit or control chart set at ± 3% or 3σ of the mean. (MARLAP 18.5.6.4)	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 a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	$\lvert Z_{Blank} \rvert \leq 3$. 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. With project approval and appropriate qualification and narration, report results with a count rate >5 times that of the affected ROI in the MB.	Recount the blank to confirm results, unless all sample results are >5 times the blank activity. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. All samples following the last acceptable background measurement must be reanalyzed.	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. Blank matrices must be the same as the associated samples (i.e. radon free distilled or deionized water, or representative of the material media.

ED_006532_00005078-00179

Table - 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 and correct problem. 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 blank.	Unquenched sealed background vial not used for background subtraction. Activity must meet project objectives.
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) in the MBM.	MBM test source matches STS configuration (type, size and position relative to the detector).

ED_006532_00005078-00180

Table - 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. 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 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.
Matrix Spike (MS)	One per preparatory batch. *(MS not required when yield tracers or carriers are employed)*	If activity of the MS > 5 times the unspiked sample, $\lvert Z_{MS} \rvert \leq 3$. (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.	The data shall be evaluated to determine the source of difference.

ED_006532_00005078-00181

Table - 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.	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 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.

ED_006532_00005078-00182

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. 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 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 ± 3 sample standard deviations.

2.0 LCS Limit Tables

Table 1. Method 1668 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2051-60-7	PCB 1	148	91.7	14.6	48	136
56558-16-8	PCB 104	152	99.4	6.2	81	118
32598-14-4	PCB 105	179	105.6	7.2	84	127
74472-37-0	PCB 114	177	105.4	6.2	87	124
31508-00-6	PCB 118	180	107.7	9.6	79	137
65510-44-3	PCB 123	188	107.2	8.8	81	134
57465-28-8	PCB 126	181	100.8	7.3	79	123
2050-68-2	PCB 15	151	106	13.9	64	148
33979-03-2	PCB 155	153	98.7	7.5	76	121
38380-08-4	PCB 156	176	104.5	6.9	84	125

ED_006532_00005078-00183

Table 1. Method 1668 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 2. Method 1668 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2051-60-7	PCB 1	206	86.7	9.4	58	115
37680-73-2	PCB 101	107	103.8	9.5	75	132
56558-16-8	PCB 104	206	99.4	6.9	79	120
32598-14-4	PCB 105	258	104.7	9.3	77	133
74472-37-0	PCB 114	246	106.5	8.7	81	133
31508-00-6	PCB 118	212	104.9	7.7	82	128

ED_006532_00005078-00184

Table 2. Method 1668 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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

ED_006532_00005078-00185

Table 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

ED_006532_00005078-00186

Table 3. Method 6010 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 4. Method 6010 Water 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

ED_006532_00005078-00187

Table 4. Method 6010 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7439-98-7	Molybdenum	8251	101.1	4	89	113
7440-02-0	Nickel	12699	100.5	4.1	88	113
7440-05-3	Palladium	492	99.8	4	88	112
7723-14-0	Phosphorus	203	100.5	4.2	88	113
7440-09-7	Potassium	11006	99.9	4.7	86	114
7782-49-2	Selenium	13264	98.5	5.2	83	114
7440-21-3	Silicon	1525	100.6	6.1	82	119
7440-22-4	Silver	13770	99.1	5.1	84	115
7440-23-5	Sodium	10893	100.9	4.7	87	115
7440-24-6	Strontium	3782	101.3	3.8	90	113
7704-34-9	Sulfur	145	100.7	3.9	89	112
7440-28-0	Thallium	10063	99.5	4.7	85	114
7440-31-5	Tin	4502	101.3	4.4	88	115
7440-32-6	Titanium	5625	101.1	3.4	91	111
7440-61-1	Uranium	223	101.3	5.8	84	119
7440-62-2	Vanadium	12032	100.2	3.6	90	111
7440-66-6	Zinc	13549	100.6	4.6	87	115

Table 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

ED_006532_00005078-00188

Table 5. Method 6020 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7440-42-8	Boron	581	101.1	9	74	128
7440-43-9	Cadmium	2893	99.6	5.4	84	116
7440-70-2	Calcium	835	102.2	5.4	86	118
7440-47-3	Chromium	2420	100.8	6	83	119
7440-48-4	Cobalt	2005	99.7	5.1	84	115
7440-50-8	Copper	2548	101.3	5.8	84	119
7439-89-6	Iron	1131	102.7	7.1	81	124
7439-92-1	Lead	3228	101	5.7	84	118
7439-93-2	Lithium	162	97.8	7.5	75	120
7439-95-4	Magnesium	868	101.6	7.1	80	123
7439-96-5	Manganese	1830	100.3	5.1	85	116
7439-97-6	Mercury	226	99.9	8.8	74	126
7439-98-7	Molybdenum	1188	98.1	5.1	83	114
7440-02-0	Nickel	2617	101.4	5.8	84	119
7440-09-7	Potassium	803	102.3	5.7	85	119
7782-49-2	Selenium	3104	99.2	6.6	80	119
7440-22-4	Silver	2488	100.1	5.9	83	118
7440-23-5	Sodium	818	102.2	7.7	79	125
7440-24-6	Strontium	676	101.7	8.9	75	129
7440-28-0	Thallium	2589	100.1	5.9	83	118
7440-29-1	Thorium	341	98.4	5.7	81	116
7440-31-5	Tin	886	101.3	6.6	82	121
7440-32-6	Titanium	512	100.2	5.7	83	117
7440-61-1	Uranium	833	101.1	6.1	83	120
7440-62-2	Vanadium	1677	99.1	5.7	82	116
7440-66-6	Zinc	2352	100.1	6.2	82	119

ED_006532_00005078-00189

Table 6. Method 6020 Water 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
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

ED_006532_00005078-00190

Table 6. Method 6020 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 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 8. Method 6850 Water 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 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

ED_006532_00005078-00191

Table 10. Method 7196 Water 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 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 12. Method 7470 - 7471 series Water 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 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

ED_006532_00005078-00192

Table 14. Method 8015 (MOD) Water 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 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

ED_006532_00005078-00193

Table 15. Method 8081 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
5103-74-2	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 16. Method 8081 Water 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

ED_006532_00005078-00194

Table 16. Method 8081 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
5103-74-2	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 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

ED_006532_00005078-00195

Table 18. Method 8082 Water 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 19. Method 8141 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
86-50-0	Azinphos-methyl	325	96.7	19.6	38	156
35400-43-2	Bolstar [Sulprofos]	270	93.5	15.1	48	139
786-19-6	Carbophenothion	237	96.6	12.5	59	134
2921-88-2	Chlorpyrifos	333	93.3	15.5	47	140
56-72-4	Coumaphos	321	98.4	20.5	37	160
8065-48-3	Demeton	254	80.2	12.4	43	117
333-41-5	Diazinon	328	87.9	15.2	42	134
62-73-7	Dichlorvos [DDVP]	322	90.6	17.2	39	142
60-51-5	Dimethoate	264	77.5	20.6	16	139
298-04-4	Disulfoton	332	86	19.5	28	145
2104-64-5	EPN	300	90.6	15.5	44	137
563-12-2	Ethion	160	99.3	13.5	59	140
13194-48-4	Ethoprop	325	87.8	13.5	47	128
52-85-7	Fampphur	192	90.6	14.6	47	134
115-90-2	Fensulfothion	324	87.1	20	27	147
55-38-9	Fenthion	325	88.7	14.9	44	134
121-75-5	Malathion	322	91.2	15.2	46	137
298-00-0	methyl Parathion	330	93.6	14.8	49	138

ED_006532_00005078-00196

Table 19. Method 8141 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
126-68-1	O,O,O-Triethyl phosphorothioate	186	79.8	13.3	40	120
56-38-2	Parathion	313	94.3	14.9	50	139
298-02-2	Phorate	330	82.6	19.8	23	142
299-84-3	Ronnel	328	91.6	15.5	45	138
122-34-9	Simazine	120	93	16.3	44	142
22248-79-9	Stirophos [Tetrachlorovinphos, Gardona]	153	91.2	16.3	42	140
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	238	89	12.2	52	126
297-97-2	Thionazine	192	83.5	13.3	44	124
34643-46-4	Tokuthion [Protothiofos]	320	90.7	15.1	45	136
327-98-0	Trichloronate	326	88.3	17.2	37	140

Table 20. Method 8141 Water 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

ED_006532_00005078-00197

Table 20. Method 8141 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
62-73-7	Dichlorvos [DDVP]	682	88.3	16.4	39	138
60-51-5	Dimethoate	597	75.2	16.5	26	125
298-04-4	Disulfoton	753	85.1	16.3	36	134
2104-64-5	EPN	623	90	14.3	47	133
563-12-2	Ethion	345	93.3	17.1	42	145
13194-48-4	Ethoprop	620	88.8	12.2	52	125
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

ED_006532_00005078-00198

Table 21. Method 8151 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
93-76-5	2,4,5-T	1106	84.6	17.7	31	138
93-72-1	2,4,5-TP [Silvex]	1179	86.1	14.3	43	129
94-75-7	2,4-D	1256	86	19.3	28	144
94-82-6	2,4-DB	1030	88.2	17.9	34	142
19719-28-9	2,4-Dichlorophenylacetic Acid	1041	74	15.9	27	122
100-02-7	4-Nitrophenol	208	76.7	20	17	137
50594-66-6	Acifluorfen	206	79.8	18	26	134
1861-32-1	Dacthal (DCPA)	147	72.5	15.6	26	119
1918-00-9	Dicamba	1070	85.2	15.7	38	132
120-36-5	Dichloroprop	1033	91.4	21	28	155
94-74-6	MCPA	935	81.5	17.8	28	135
93-65-2	MCPP	807	88.7	18	35	143

Table 22. Method 8151 Water 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

ED_006532_00005078-00199

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 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

ED_006532_00005078-00200

Table 23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
17060-07-0	1,2-Dichloroethane-d4	5951	103.1	10.8	71	136
540-59-0	1,2-Dichloroethene	7748	99.9	7.3	78	122
78-87-5	1,2-Dichloropropane	12145	99.5	7.8	76	123
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	1269	97.8	11.3	64	132
108-70-3	1,3,5-Trichlorobenzene	4723	99.4	9.6	71	128
108-67-8	1,3,5-Trimethylbenzene	11080	98.4	8.4	73	124
541-73-1	1,3-Dichlorobenzene	11619	98.9	7.4	77	121
142-28-9	1,3-Dichloropropane	10713	99.1	7.3	77	121
542-75-6	1,3-Dichloropropene	3714	101.6	8.1	77	126
106-46-7	1,4-Dichlorobenzene	11848	97.5	7.6	75	120
105-05-5	1,4-Diethylbenzene	1896	96.6	5.9	79	114
123-91-1	1,4-Dioxane	7698	96.4	13.7	55	138
544-10-5	1-Chlorohexane	2543	100.4	9.8	71	130
594-20-7	2,2-Dichloropropane	10703	99.7	11.1	67	133
78-93-3	2-Butanone [MEK]	11514	99.6	16.3	51	148
126-99-8	2-Chloro-1,3-butadiene	6667	99	11.3	65	133
110-75-8	2-Chloroethyl vinyl ether	6957	96.1	17.6	43	149
95-49-8	2-Chlorotoluene	10838	98.5	7.9	75	122
591-78-6	2-Hexanone	11004	99.1	15.4	53	145
79-46-9	2-Nitropropane	4969	98.3	17.1	47	150
67-63-0	2-Propanol [Isopropyl alcohol]	1696	99.8	13.4	60	140
460-00-4	4-Bromofluorobenzene	6267	98.9	6.8	79	119
106-43-4	4-Chlorotoluene	10785	98.3	8.6	72	124
108-10-1	4-Methyl-2-pentanone [MIBK]	11364	99.6	11.6	65	135
67-64-1	Acetone	11089	99.6	21.4	36	164

ED_006532_00005078-00201

Table 23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
75-05-8	Acetonitrile	5697	98.5	14.8	54	143
107-02-8	Acrolein [Propenal]	7528	101.1	18	47	155
107-13-1	Acrylonitrile	8293	99.7	11.4	65	134
107-05-1	Allyl chloride	6908	101.1	11.2	68	135
71-43-2	Benzene	12853	99.2	7.4	77	121
100-44-7	Benzyl chloride	2743	92.1	9.4	64	120
108-86-1	Bromobenzene	10974	99.3	7.3	78	121
74-97-5	Bromochloromethane	11023	101.4	7.8	78	125
75-27-4	Bromodichloromethane	11850	101	8.5	75	127
75-25-2	Bromoform	11890	99.1	10.8	67	132
74-83-9	Bromomethane	11416	98.3	15	53	143
75-15-0	Carbon disulfide	11132	97.9	11.5	63	132
56-23-5	Carbon tetrachloride	12090	102.3	10.7	70	135
108-90-7	Chlorobenzene	12382	99.7	6.9	79	120
124-48-1	Chlorodibromomethane	11852	100.2	8.7	74	126
75-00-3	Chloroethane	11444	98.8	13.3	59	139
67-66-3	Chloroform	12344	100.3	7.6	78	123
74-87-3	Chloromethane	11876	93.3	14.3	50	136
156-59-2	cis-1,2-Dichloroethene	11645	99.9	7.6	77	123
10061-01-5	cis-1,3-Dichloropropene	11805	99.8	8.7	74	126
1476-11-5	cis-1,4-Dichloro-2-butene	977	106	12.4	69	143
110-82-7	Cyclohexane	8827	98.9	10.6	67	131
108-94-1	Cyclohexanone	3764	93.2	20.9	30	156
1868-53-7	Dibromofluoromethane	2142	98.1	6.8	78	119
74-95-3	Dibromomethane	10913	101.1	7.9	78	125
75-71-8	Dichlorodifluoromethane [Freon-12]	11467	88.9	20.1	29	149

ED_006532_00005078-00202

Table 23. Method 8260 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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

ED_006532_00005078-00203

Table 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

ED_006532_00005078-00204

Table 24. Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	24511	101.1	7.6	78	124
71-55-6	1,1,1-Trichloroethane	28223	102.7	9.6	74	131
79-34-5	1,1,2,2-Tetrachloroethane	27450	96.4	8.3	71	121
79-00-5	1,1,2-Trichloroethane	27338	99.5	6.5	80	119
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	21122	103	11.1	70	136
75-34-3	1,1-Dichloroethane	28154	101.3	8	77	125
75-35-4	1,1-Dichloroethene	29436	101	10	71	131
563-58-6	1,1-Dichloropropene	23631	102	7.8	79	125
87-61-6	1,2,3-Trichlorobenzene	24271	98.7	10.1	69	129
96-18-4	1,2,3-Trichloropropane	24525	97.5	8	73	122
526-73-8	1,2,3-Trimethylbenzene	2965	100.9	6.2	82	120
120-82-1	1,2,4-Trichlorobenzene	25290	99.8	10.1	69	130
95-63-6	1,2,4-Trimethylbenzene	27917	99.6	8	76	124
96-12-8	1,2-Dibromo-3-chloropropane	24955	94.9	11.1	62	128
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

ED_006532_00005078-00205

Table 24. Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
106-99-0	1,3-Butadiene	1202	100.6	19.2	43	158
541-73-1	1,3-Dichlorobenzene	26951	99.7	6.5	80	119
142-28-9	1,3-Dichloropropane	23811	99.1	6.5	80	119
542-75-6	1,3-Dichloropropene	9784	99.9	7.6	77	123
106-46-7	1,4-Dichlorobenzene	27715	98.3	6.5	79	118
105-05-5	1,4-Diethylbenzene	1980	98.4	6.4	79	118
123-91-1	1,4-Dioxane	17866	99	13.4	59	139
544-10-5	1-Chlorohexane	5790	99.6	8	76	124
540-84-1	2,2,4-Trimethylpentane [Isooctane]	5432	95.2	12.3	58	132
594-20-7	2,2-Dichloropropane	23775	99.7	13.2	60	139
75-85-4	2-Butanol	4332	92.7	9.1	66	120
78-93-3	2-Butanone [MEK]	26659	99.6	14.6	56	143
126-99-8	2-Chloro-1,3-butadiene	15673	100	11.7	65	135
110-75-8	2-Chloroethyl vinyl ether	18225	94.7	14.7	51	139
95-49-8	2-Chlorotoluene	23750	100	7.2	79	122
591-78-6	2-Hexanone	25368	97.9	13.5	57	139
91-57-6	2-Methylnaphthalene	3754	79.4	20.9	17	142
79-46-9	2-Nitropropane	10213	92.6	14.5	49	136
67-63-0	2-Propanol [Isopropyl alcohol]	2034	98.8	14.4	56	142
624-95-3	3,3-Dimethyl-1-butanol	6491	90.9	13.9	49	133
460-00-4	4-Bromofluorobenzene	9971	99.7	4.9	85	114
106-43-4	4-Chlorotoluene	23616	99.9	7.4	78	122
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

ED_006532_00005078-00206

Table 24. Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
107-02-8	Acrolein [Propenal]	16380	96.8	19.3	39	155
107-13-1	Acrylonitrile	20173	99	11.9	63	135
107-05-1	Allyl chloride	15758	99	10.4	68	130
71-43-2	Benzene	34376	99.4	6.9	79	120
100-44-7	Benzyl chloride	10675	90.1	15.9	42	138
108-86-1	Bromobenzene	23762	99.7	6.7	80	120
74-97-5	Bromochloromethane	24356	100.8	7.5	78	123
75-27-4	Bromodichloromethane	26888	101.8	7.8	79	125
75-25-2	Bromoform	27675	97.8	10.8	66	130
74-83-9	Bromomethane	26717	97	14.7	53	141
75-15-0	Carbon disulfide	25719	98.8	11.5	64	133
56-23-5	Carbon tetrachloride	28870	103.8	10.7	72	136
108-90-7	Chlorobenzene	29802	100	6.1	82	118
124-48-1	Chlorodibromomethane	27424	100	8.5	74	126
75-45-6	Chlorodifluoromethane	7197	84.4	14.9	40	129
75-00-3	Chloroethane	27069	99	13	60	138
67-66-3	Chloroform	29373	101.1	7.5	79	124
74-87-3	Chloromethane	27697	94.5	15	50	139
156-59-2	cis-1,2-Dichloroethene	27935	100.1	7.5	78	123
10061-01-5	cis-1,3-Dichloropropene	27197	99.5	8	75	124
1476-11-5	cis-1,4-Dichloro-2-butene	1524	101.5	14.9	57	146
110-82-7	Cyclohexane	20438	100.4	10	71	130
1868-53-7	Dibromofluoromethane	5702	99.1	6.5	80	119
74-95-3	Dibromomethane	24473	101.1	7.3	79	123
75-71-8	Dichlorodifluoromethane [Freon-12]	25410	92	20.1	32	152

ED_006532_00005078-00207

Table 24. Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

ED_006532_00005078-00208

Table 24. Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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	26108	103	12.8	65	141

ED_006532_00005078-00209

Table 24. Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
	[Freon-11]					
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 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

ED_006532_00005078-00210

Table 25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
608-27-5	2,3-Dichloroaniline	108	82.4	13	44	121
95-95-4	2,4,5-Trichlorophenol	4014	82.6	13.7	41	124
118-79-6	2,4,6-Tribromophenol	2930	85.7	15.4	39	132
88-06-2	2,4,6-Trichlorophenol	4183	82.1	14.5	39	126
120-83-2	2,4-Dichlorophenol	3794	80.9	13.7	40	122
105-67-9	2,4-Dimethylphenol	3886	78.4	16.2	30	127
121-14-2	2,4-Dinitrotoluene	4075	86.8	12.9	48	126
87-65-0	2,6-Dichlorophenol	1364	79.2	12.6	41	117
606-20-2	2,6-Dinitrotoluene	3706	85	13	46	124
53-96-3	2-Acetylaminofluorene	175	94	13.3	54	134
91-58-7	2-Chloronaphthalene	3569	77.5	12.1	41	114
95-57-8	2-Chlorophenol	3977	77.3	14.5	34	121
321-60-8	2-Fluorobiphenyl	3191	79.5	11.8	44	115
367-12-4	2-Fluorophenol	3008	75.2	13.3	35	115
91-57-6	2-Methylnaphthalene	5059	80.1	14	38	122
95-48-7	2-Methylphenol (o-Cresol)	4016	77	14.9	32	122
88-74-4	2-Nitroaniline	3639	85.4	13.8	44	127
119-75-5	2-Nitrodiphenylamine	279	88.1	11.6	53	123
88-75-5	2-Nitrophenol	3804	79.6	14.5	36	123
109-06-8	2-Picoline [2-Methylpyridine]	181	64.5	12.7	27	103
91-94-1	3,3'-Dichlorobenzidine	3521	71.3	16.5	22	121
56-49-5	3-Methylcholanthrene	188	95.1	13	56	134
99-09-2	3-Nitroaniline	3454	75.9	14.3	33	119
65794-96-9	3/4-Methylphenol [m/p-Cresol]	2900	76.5	14.1	34	119
534-52-1	4,6-Dinitro-2-methylphenol	3739	80.7	17.2	29	132
101-55-3	4-Bromophenyl phenyl ether	3708	85.1	13	46	124

ED_006532_00005078-00211

Table 25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
59-50-7	4-Chloro-3-methylphenol	3880	83.3	12.9	45	122
106-47-8	4-Chloroaniline [p-Chloroanlinie]	3435	61.3	14.9	17	106
7005-72-3	4-Chlorophenyl phenyl ether	3673	83	12.7	45	121
106-44-5	4-Methylphenol [p-Cresol]	1555	84.1	14.1	42	126
100-02-7	4-Nitrophenol	3976	80.6	17	30	132
99-55-8	5-Nitro-o-toluidine [2-Amino-4-nitrotoluene]	187	69.8	15.8	23	117
57-97-6	7,12-Dimethylbenz(a)-anthracene	338	96.2	15.3	50	142
83-32-9	Acenaphthene	5300	81.3	13.7	40	123
208-96-8	Acenaphthylene	5194	81.8	16.8	32	132
98-86-2	Acetophenone	2101	73.9	13.6	33	115
120-12-7	Anthracene	5250	85.2	12.7	47	123
1912-24-9	Atrazine	1428	87.1	13.4	47	127
103-33-3	Azobenzene	378	82.1	14.2	39	125
56-55-3	Benz(a)anthracene	5385	87.4	12.9	49	126
50-32-8	Benzo(a)pyrene	5500	86.9	13.9	45	129
205-99-2	Benzo(b)fluoranthene	5323	88.3	14.5	45	132
191-24-2	Benzo(g,h,i)perylene	5263	88.5	15.1	43	134
207-08-9	Benzo(k)fluoranthene	5386	89.6	14.2	47	132
100-51-6	Benzyl alcohol	2895	75.7	15.6	29	122
111-91-1	bis(2-Chloroethoxy)methane	3705	78.4	14.2	36	121
111-44-4	Bis(2-chloroethyl) ether	3711	75.4	14.9	31	120
39638-32-9	bis(2-Chloroisopropyl) ether	769	82	16.3	33	131
117-81-7	Bis(2-ethylhexyl) phthalate	4018	91.9	13.7	51	133
103-23-1	bis(2-Ethylhexyl)adipate	156	90.8	10.1	61	121
85-68-7	Butyl benzyl phthalate	3956	90.3	14	48	132

ED_006532_00005078-00212

Table 25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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

ED_006532_00005078-00213

Table 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

ED_006532_00005078-00214

Table 25. Method 8270 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
23950-58-5	Pronamide	179	93	12.4	56	130
129-00-0	Pyrene	5518	87.2	13.3	47	127
91-22-5	Quinoline	219	90	11.9	54	126
94-59-7	Safrole	176	87.8	13.6	47	129
1718-51-0	Terphenyl-d14	3111	90.5	12.3	54	127
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	136	94.4	14	52	137
297-97-2	Thionazine	139	94.6	10.7	62	127

Table 26. Method 8270 Water 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

ED_006532_00005078-00215

Table 26. Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
935-95-5	2,3,5,6-Tetrachlorophenol	266	85.6	11.7	50	121
608-27-5	2,3-Dichloroaniline	150	99.2	9.8	70	129
95-95-4	2,4,5-Trichlorophenol	5707	88.1	11.8	53	123
118-79-6	2,4,6-Tribromophenol	2059	91.5	16	43	140
88-06-2	2,4,6-Trichlorophenol	6136	87.2	12.4	50	125
120-83-2	2,4-Dichlorophenol	5330	84	12.2	47	121
105-67-9	2,4-Dimethylphenol	5298	77.5	15.6	31	124
51-28-5	2,4-Dinitrophenol	5127	82.9	20	23	143
121-14-2	2,4-Dinitrotoluene	6032	92.3	11.8	57	128
87-65-0	2,6-Dichlorophenol	1583	84	11.4	50	118
606-20-2	2,6-Dinitrotoluene	5107	90.7	11.2	57	124
53-96-3	2-Acetylaminofluorene	228	98.9	12.9	60	138
91-58-7	2-Chloronaphthalene	5084	78	12.8	40	116
95-57-8	2-Chlorophenol	5571	77.5	13.2	38	117
93951-73-6	2-Chlorophenol-d4	119	79.9	8.7	54	106
321-60-8	2-Fluorobiphenyl	2263	81.2	12.4	44	119
367-12-4	2-Fluorophenol	2022	68.8	16.6	19	119
91-57-6	2-Methylnaphthalene	6330	80.7	13.6	40	121
95-48-7	2-Methylphenol (o-Cresol)	5800	73	14.5	30	117
88-74-4	2-Nitroaniline	4855	90.8	12.1	55	127
119-75-5	2-Nitrodiphenylamine	272	97.3	11.3	64	131
88-75-5	2-Nitrophenol	5097	84.6	12.7	47	123
109-06-8	2-Picoline [2-Methylpyridine]	195	71.6	12.6	34	109
91-94-1	3,3'-Dichlorobenzidine	4815	77.9	16.9	27	129
56-49-5	3-Methylcholanthrene	237	94	12.8	56	133

ED_006532_00005078-00216

Table 26. Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
99-09-2	3-Nitroaniline	4808	84.4	14.5	41	128
65794-96-9	3/4-Methylphenol [m/p-Cresol]	3472	69.7	13.6	29	110
534-52-1	4,6-Dinitro-2-methylphenol	5097	90.1	15.5	44	137
101-55-3	4-Bromophenyl phenyl ether	5074	89.1	11.5	55	124
59-50-7	4-Chloro-3-methylphenol	5338	85.5	11.3	52	119
106-47-8	4-Chloroaniline [p-Chloroanlinie]	4687	75.3	14	33	117
7005-72-3	4-Chlorophenyl phenyl ether	5071	86.7	11.3	53	121
106-44-5	4-Methylphenol [p-Cresol]	2798	72.5	15.8	25	120
99-55-8	5-Nitro-o-toluidine [2-amino-4-nitrotoluene]	260	82.1	14.6	38	126
57-97-6	7,12-Dimethylbenz(a)-anthracene	373	97.1	11.9	61	133
83-32-9	Acenaphthene	6952	84.5	12.3	47	122
208-96-8	Acenaphthylene	6662	85.3	14.7	41	130
98-86-2	Acetophenone	2877	82.1	12	46	118
120-12-7	Anthracene	6792	89.6	11	57	123
140-57-8	Aramite	100	82.8	16.3	34	132
1912-24-9	Atrazine	2328	92.8	16.4	44	142
103-33-3	Azobenzene	578	88.5	9.3	61	116
56-55-3	Benz(a)anthracene	6867	91.6	11.1	58	125
50-32-8	Benzo(a)pyrene	7045	90.8	12.4	54	128
205-99-2	Benzo(b)fluoranthene	6767	92	12.9	53	131
191-24-2	Benzo(g,h,i)perylene	6624	92	13.9	50	134
207-08-9	Benzo(k)fluoranthene	6803	93.2	12.1	57	129
100-51-6	Benzyl alcohol	3349	71.2	13.5	31	112
111-91-1	bis(2-Chloroethoxy)methane	5094	83.9	11.9	48	120
111-44-4	Bis(2-chloroethyl) ether	5139	80.8	12.6	43	118

ED_006532_00005078-00217

Table 26. Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

ED_006532_00005078-00218

Table 26. Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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

ED_006532_00005078-00219

Table 26. Method 8270 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
87-86-5	Pentachlorophenol	6083	86.4	17.1	35	138
82-68-8	Pentchloronitrobenzene	618	94.5	13.4	54	135
62-44-2	Phenacetin	241	97.9	8.9	71	124
85-01-8	Phenanthrene	6822	89.6	10.2	59	120
298-02-2	Phorate	126	88.6	16.8	38	139
23950-58-5	Pronamide	249	97	10.5	65	129
129-00-0	Pyrene	7013	91.1	11.5	57	126
91-22-5	Quinoline	249	100.1	10.5	69	132
94-59-7	Safrole	233	90	9.7	61	119
1718-51-0	Terphenyl-d14	1893	91.7	13.9	50	134
3689-24-5	Tetraethyl dithiopyrophosphate [Sulfotep]	200	96.7	11.9	61	133
297-97-2	Thionazine	196	102	10.1	72	132

Table 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

ED_006532_00005078-00220

Table 27. Method 8270 SIM Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
50-32-8	Benzo(a)pyrene	2789	87.3	12.5	50	125
205-99-2	Benzo(b)fluoranthene	2790	90.3	12.6	53	128
191-24-2	Benzo(g,h,i)perylene	2739	87.8	13	49	127
207-08-9	Benzo(k)fluoranthene	2761	89.3	11.2	56	123
111-44-4	Bis(2-chloroethyl) ether	192	65.4	15.8	18	113
117-81-7	Bis(2-ethylhexyl) phthalate	181	108.9	13.9	67	150
85-68-7	Butyl benzyl phthalate	144	103.5	10.6	72	135
86-74-8	Carbazole	183	79.3	14.6	36	123
218-01-9	Chrysene	2812	87.5	10.2	57	118
84-74-2	Di-n-butyl phthalate	150	106.5	12.9	68	145
117-84-0	Di-n-octyl phthalate	144	105.5	16.8	55	156
53-70-3	Dibenzo(a,h)anthracene	2778	89.2	13.2	50	129
132-64-9	Dibenzofuran	282	71.9	12.2	35	108
84-66-2	Diethyl phthalate	147	99.3	10.9	67	132
131-11-3	Dimethyl phthalate	149	99.3	9.3	71	127
206-44-0	Fluoranthene	2782	87.3	10.7	55	119
86-73-7	Fluorene	2795	80.6	11.2	47	114
118-74-1	Hexachlorobenzene	201	81.9	14.2	39	125
193-39-5	Indeno(1,2,3-cd)pyrene	2812	89.6	13.5	49	130
62-75-9	N-Nitrosodimethylamine	117	90.7	10.9	58	124
91-20-3	Naphthalene	2823	74.7	12.2	38	111
4165-60-0	Nitrobenzene-d5	531	84.7	13.6	44	125
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
1718-51-0	Terphenyl-d14	1864	95.3	12.6	58	133

ED_006532_00005078-00221

Table 28. Method 8270 SIM Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
92-52-4	1,1-Biphenyl	106	77.3	7.3	56	99
90-12-0	1-Methylnaphthalene	2566	77.9	12.5	41	115
95-95-4	2,4,5-Trichlorophenol	488	84.1	13.4	44	124
118-79-6	2,4,6-Tribromophenol	164	83.7	12.7	46	122
606-20-2	2,6-Dinitrotoluene	118	67.2	15.8	20	115
91-58-7	2-Chloronaphthalene	717	72.4	12.7	34	111
321-60-8	2-Fluorobiphenyl	747	79.2	8.8	53	106
91-57-6	2-Methylnaphthalene	2984	76.5	12.6	39	114
83-32-9	Acenaphthene	3241	80.9	11.1	48	114
208-96-8	Acenaphthylene	3234	77.8	14.4	35	121
120-12-7	Anthracene	3224	85.8	11	53	119
56-55-3	Benz(a)anthracene	3277	89.3	10.1	59	120
50-32-8	Benzo(a)pyrene	3284	86.4	11.2	53	120
205-99-2	Benzo(b)fluoranthene	3248	89.7	12.3	53	126
191-24-2	Benzo(g,h,i)perylene	3178	86	14.1	44	128
207-08-9	Benzo(k)fluoranthene	3167	89.3	11.9	54	125
111-44-4	Bis(2-chloroethyl) ether	775	77.8	12.6	40	116
117-81-7	Bis(2-ethylhexyl) phthalate	275	114.1	19.6	55	173
85-68-7	Butyl benzyl phthalate	159	90.7	17.3	39	143
86-74-8	Carbazole	631	84	13.1	45	123
218-01-9	Chrysene	3215	88.3	10.4	57	120
84-74-2	Di-n-butyl phthalate	153	102.5	14.2	60	145
117-84-0	Di-n-octyl phthalate	157	103.3	19	46	160
53-70-3	Dibenzo(a,h)anthracene	3233	87.2	14.5	44	131
132-64-9	Dibenzofuran	864	77.5	14.1	35	120

ED_006532_00005078-00222

Table 28. Method 8270 SIM Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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

ED_006532_00005078-00223

Table 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 30. Method 8290 Water 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

ED_006532_00005078-00224

Table 30. Method 8290 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 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

ED_006532_00005078-00225

Table 31. Method 8310 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 32. Method 8310 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
90-12-0	1-Methylnaphthalene	432	73.3	11	40	106
91-57-6	2-Methylnaphthalene	448	73.4	10.7	41	106
83-32-9	Acenaphthene	493	78.5	11.2	45	112
208-96-8	Acenaphthylene	478	80.5	9.1	53	108
120-12-7	Anthracene	453	85.8	9.2	58	113
56-55-3	Benz(a)anthracene	493	89	11.6	54	124
50-32-8	Benzo(a)pyrene	445	89.1	10.3	58	120
205-99-2	Benzo(b)fluoranthene	467	88.7	11.6	54	124
191-24-2	Benzo(g,h,i)perylene	428	88.6	11.3	55	122
207-08-9	Benzo(k)fluoranthene	460	88.4	11.8	53	124
218-01-9	Chrysene	469	90.3	9.6	61	119
53-70-3	Dibenzo(a,h)anthracene	452	87.2	10.5	56	119

ED_006532_00005078-00226

Table 32. Method 8310 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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 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

ED_006532_00005078-00227

Table 34. Method 8321 Water 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 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

ED_006532_00005078-00228

Table 35. Method 8330 Solid Matrix						
606-20-2	2,6-Dinitrotoluene	556	102.4	5.4	86	119
35572-78-2	2-Amino-4,6-dinitrotoluene	562	103.8	5.7	87	121
88-72-2	2-Nitrotoluene	591	102	6	84	120
99-08-1	3-Nitrotoluene	614	103.3	8	79	127
19406-51-0	4-Amino-2,6-dinitrotoluene	594	104.2	6.7	84	124
99-99-0	4-Nitrotoluene	595	102.2	6.5	83	122
121-82-4	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	595	103.1	6.9	82	124
98-95-3	Nitrobenzene	598	103.9	7.9	80	128
55-63-0	Nitroglycerin	352	97.2	8.2	73	122
2691-41-0	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	581	99.1	7.5	77	122
78-11-5	PETN	326	100.9	7.5	78	123
479-45-8	Tetryl	584	101.8	11.9	66	138

Table 36. Method 8330 - 8330B series Water 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

ED_006532_00005078-00229

Table 36. Method 8330 - 8330B series Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
618-87-1	3,5-Dinitroaniline	150	94.3	7.6	71	117
99-08-1	3-Nitrotoluene	1643	98.8	8.8	73	125
19406-51-0	4-Amino-2,6-dinitrotoluene	1586	100.3	8	76	125
99-99-0	4-Nitrotoluene	1654	99.1	9.3	71	127
121-82-4	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	1833	99.1	10.4	68	130
80251-29-2	Hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine (DNX)	109	92.8	8.8	66	119
5755-27-1	Hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine (MNX)	249	94.3	12.5	57	132
98-95-3	Nitrobenzene	1743	99.3	11.4	65	134
55-63-0	Nitroglycerin	1076	100.4	8.8	74	127
2691-41-0	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	1755	100	11.8	65	135
78-11-5	PETN	1079	100.2	9	73	127
479-45-8	Tetryl	1597	95.8	10.7	64	128

Table 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

ED_006532_00005078-00230

Table 37. Method 8330B Solid Matrix						
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 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 39. Method 9010 - 9020 Series Water 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

ED_006532_00005078-00231

Table 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 41. Method 9056 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
24959-67-9	Bromide	2199	100.3	3.2	91	110
16887-00-6	Chloride	4948	98.5	4	87	111
16984-48-8	Fluoride	3251	99.7	4	88	112
14797-55-8	Nitrate	3192	99.7	3.9	88	111
14797-65-0	Nitrite	2583	98.9	3.9	87	111
14265-44-2	Phosphate	843	97.8	6.1	80	116
14808-79-8	Sulfate	4155	99.2	4.1	87	112

ED_006532_00005078-00232

Table 42. Method RSK-175 Water 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 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

ED_006532_00005078-00233

Table 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

ED_006532_00005078-00234

Table 43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

ED_006532_00005078-00235

Table 43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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
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

ED_006532_00005078-00236

Table 43. Method TO-15 Gas Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
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

ED_006532_00005078-00237

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 an NDA measurement system to radioactive materials present in the waste containers or process components of interest (measurement items). The "calibration confirmation" is a thorough corroboration of the "initial calibration" using traceable working reference materials (WRMs) and representative waste matrix/ process component configurations. The "calibration verification" is a periodic verification of the "initial calibration" to ensure on-going long-term data quality compliance through the period of NDA operations.

Procedural steps for calibration are not specified here. However, those elements that must be considered during the "initial calibration," "calibration confirmation", and "calibration verification" are enumerated. This allows the NDA measurement organization autonomy in devising and implementing techniques and analytical procedures for these three calibration definitions. Through these three mechanisms, the NDA measurement organization shall demonstrate the calibration and associated uncertainty is compliant with applicable client and/or end-user requirements initially and throughout the contract period.

1.1.1 Initial NDA System Calibration

An NDA measurement system "initial calibration" shall be performed to ensure the measurement system response provides valid data of known and documented quality. Calibrations shall be performed using traceable WRMs obtained from suppliers maintaining a nationally recognized reference base and an accredited measurement program. Full documentation of the calibration technique, process, and results is required. For cases where there is an insufficient number and denomination of traceable radioactive material standards to support the "initial calibration", the NDA organization can develop alternate calibration strategies based on available resources. Alternate strategies shall be clearly documented and technically justifiable.

ED_006532_00005078-00238

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

ED_006532_00005078-00239

produce a given matrix configuration shall be carefully specified, procured and the resultant physical properties and configuration documented.

f) If NDA method manuals, national standards, or a mandated NDA calibration methods do not specify the number of traceable WRMs to span the mass/activity and radioactive material compound(s) characteristics of the waste/process component, a minimum number must be determined and technically justified. NDA organization must document this number and their denominations in a calibration SOP or other applicable document. This requirement does not necessarily apply to NDA methods that rely on modeling. However, the method used to assure that the calibration and model appropriately spans and brackets the anticipated analysis space (e.g., provide mechanisms to account for anticipated geometries, radioactive material mass, chemical composition, and matrix characteristics) as per item (b) above must be technically justified and documented. The For NDA methods that do not necessarily require calibration with source material similar in nature to the waste or process items (e.g., neutron counting), those source(s) used are still required to be traceable. However, accounting of the efficiency variation because of the composition of the actual radioactive material shall be assessed and corrected for (e.g., Californium (^{252}Cf) fission neutron spectrum counter efficiency versus uranyl fluoride (UO_2F_2) neutron spectrum efficiency.)

g) The "initial calibration" process shall be clearly documented including the calibration measurement configurations, data acquisition parameters, acquired data, data reduction methods, resultant calibration factors or expressions, statistical analyses and uncertainties. Records containing information pertinent to the calibration process shall be retained including but not limited to:
    1) WRM and/or surrogate waste matrix configurations used to acquire instrument response data, calibration determination techniques,
    2) SOP(s) used,
    3) data acquisition parameters,
    4) NDA system identification,
    5) analytical software used,
    6) traceable standard identifications,
    7) analytical support equipment information,
    8) electronic file storage locations.

    Records shall be sufficient to allow reproduction of the "initial calibration".

h) The initial calibration shall be re-established when repairs or changes are made to the measurement system that are likely to affect one or more calibration parameters. Examples that may require repeating the initial calibration include, but not are limited to:
    1) major NDA system repairs or modifications,
    2) replacement of vital NDA measurement system components (e.g., collimator, multi-channel analyzer (MCA), neutron generator),
    3) change in collimator depth and/or aperture not accounted for in a model, and
    4) significant software modification and/or changes.

ED_006532_00005078-00240

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.

ED_006532_00005078-00241

f) For the case where a corrective action was required and subsequently implemented, the "calibration confirmation" process is to be repeated. Acceptable results must be obtained and documented before the NDA system is considered operational. Where a "calibration confirmation" failure was determined to be due to a minor issue (e.g., wrong constant, wrong efficiency file, or an inappropriate test item), the entire "calibration confirmation" measurement regimen may not need to be repeated. This is acceptable provided it is the true cause of the failure. All corrective actions and their effects, supporting data, results, etc., shall be documented and retained.

g) In the case where the "calibration confirmation" was acceptable for certain types or categories of radioactive material/waste matrix configurations, but unacceptable for other categories with distinctly different source/matrix properties, conditional acceptance of the "calibration confirmation" can be made. The NDA organization, however, must clearly identify which categories of source/matrix configurations are approved for NDA measurement and which are not. The technical basis for accepting certain source/matrix categories shall be documented and available for review. Recalibration or corrective action efforts should be implemented and documented for source/matrix categories not meeting acceptance criteria for "calibration confirmation".

h) The "calibration confirmation" process shall be performed following an initial calibration or when there are indications warranting a re-assessment of the "initial calibration", e.g., the source/matrix configuration of measurement items varies relative to the source/matrix configurations used to develop the "initial calibration". Additional causes for a performing a "calibration confirmation" include:

    1) major NDA system repairs or modifications,
    2) replacement of NDA measurement system components, e.g., detector, neutron generator or supporting electronic components that have the potential to affect data quality,
    3) re-calibration,
    4) significant changes to the NDA system software, and,

i) relocation of the system (applies primarily to fixed stationary systems). 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.

ED_006532_00005078-00242

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 -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 -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 -2.

Table -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 -2. Upper Limits for %RSD vs. Number of Replicates

Number of Replicates	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Max %RSD[a]	1.8	6.6	10.0	12.3	14.0	15.2	16.2	17.1	17.7	18.3	18.8	19.3	19.7	20.0

a – the values listed are derived from the measured standard deviation of the replicate measurements using

$$\frac{s}{\mu} \times 100\% < \sqrt{\frac{(0.292)^2 \times \chi^2_{0.05,n-1}}{n-1}} \times 100\%$$

where **s** is the measured standard deviation, **n** is the number of replicates, **μ** is the known or true value, $\chi^2_{0.05,n-1}$ is the critical value for the upper 5% tail of a one sided chi-squared distribution, with n-1 degrees of freedom, and the **0.292 constant** corresponds to a 95% upper confidence bound on the true system precision limit of 29.2%.

ED_006532_00005078-00243

NDA service providers may develop alternate methods and limits for bias and precision. Such alternate methods and limits must be technically defensible and clearly documented.

Failure to comply with the bias and precision requirements for "calibration confirmation" requires development of a corrective action plan (CAP). The CAP shall include detail on the nature of the failure, its suspected causes, methods to evaluate potential causes, and activities proposed to identify and rectify the deficiency. The CAP results shall be documented and show why the failure occurred and what actions were taken to prevent a re-occurrence. The calibration confirmation shall be performed again after the corrective actions in the CAP have been implemented and the results documented.

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

ED_006532_00005078-00244

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:

a) Transmission Factor - ASTM C1133-89, 'Standard Test Method for NDA of Special Nuclear Material in Low Density Scrap and Waste by Segmented Passive Gamma-Ray Scanning,' ASTM, 1989.
b) Peak ratio - Software such as Multi-Group Analysis for Uranium.

ED_006532_00005078-00245

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, r NBL, etc.). For NDA measurements, traceable materials include radioactive WRMs, certified weights for scale calibrations and thickness measurement methods.

a) The NDA organization shall have a program and procedures for establishing a traceable calibration as well as QC checking of its NDA instrumentation and support equipment. This program shall include a system for selecting, procuring, using, and controlling traceable reference standards for NDA measurement instrumentation and

ED_006532_00005078-00246

support equipment. For cases where traceable working reference materials are not yet available, the NDA organization may propose alternate methods that are technically defensible and clearly documented.

b) Traceable sources used for calibration shall be traceable for all attributes used for the calibration (e.g., a ^{252}Cf source shall be certified in its neutron yield and isotopic composition used to calculated the decay rate, and a mixed nuclide source used to perform an efficiency calibration of a gamma-ray detector shall be certified for the yield of each gamma ray energy used in the calibration and the decay properties of the contributing nuclides).

c) The NDA organization shall have a procedure(s) for the specification, procurement and acceptance of WRMs. The WRM certifications shall be acquired and maintained, and traceable to a nationally recognized reference base (e.g., NIST, NBL).

d) The NDA service provider shall retain records for all WRMs including the manufacturer/vendor, the manufacturer's Certificate of Traceability, the date of receipt, and a certificate expiration date.

e) Traceable standards shall be verified at a minimum of every five years. Standards with an expiration date less than five years shall be verified at a period equal to the time expiration time interval. Verification of a standard is accomplished through an assessment of its usable attribute to the NDA application (e.g., ^{235}U 185.7 keV gamma-ray emission rate and neutron emission rate). The area number of means by which a standard can be deemed verified as acceptable for use.

   1) The standard can be sent to a qualified facility maintaining measurement systems traceable to a certified reference material (CRM) for a determination of the standard attribute of interest. In this case the standard is simply given an updated attribute value and returned to the NDA organization with a revised or new certificate.

   2) Another method is to cross compare the standard with another traceable standard possessing the same attribute in a calibrated and operational measurement system. An evaluation of the results can produce a verification of the standard that is about to or has expired. The NDA organization must determine the acceptable uncertainty in the verified value relative to the NDA characterization process at hand.

   The verification method used and standard verification acceptability criteria shall be documented. The results of the verification are to be documented and maintained as a QA record.

f) WRM Certificates of Traceability shall contain information and data that clearly details traceability to a CRM.

g) Checks needed to maintain confidence in the status of WRMs shall be carried out according to defined procedures and schedules.

h) The NDA service provider shall have procedures for the safe handling, transport, storage and use of WRMs in order to prevent contamination or deterioration and protect their integrity.

ED_006532_00005078-00247

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:
   1) Software development and testing,
   2) Software V&V,
   3) Software configuration control, and
   4) Software operation and maintenance.

c) Computer software developed by the NDA organization shall be documented per applicable software development quality standards. Such standards usually require documentation, including:
   1) Software specification document,
   2) Software design document,
   3) Software test plan, and
   4) Software V&V document (Note: Commercial off-the-shelf software [e.g., word processing, database and statistical programs in general] used within its designed application range are usually considered to be sufficiently validated). However, NDA organization developed software and/or modifications to commercial software must be validated. Installation and operability checks shall also be performed.

d) Software change procedures shall include requirements for the requesting, testing, quality assurance, approving, and implementation of changes.

e) Data including but not limited to, decay constants, branching ratios, material attenuation values, neutron yields, and master gamma libraries used in the reduction of processing of NDA measurement data to a reportable quantity, whether electronic or hardcopy, shall be placed under a control system so only authorized individuals have access.

ED_006532_00005078-00248

f) Working data or source files (e.g., nuclear data libraries, master gamma libraries, geometry files, and efficiency files) shall be controlled by the NDA organization to prevent unauthorized access or inadvertent changes and controlled to document changes by authorized users to allow for re-creatability of the data used.

g) When commercial software is used that has the capability of performing user-defined calculations or macros (e.g., spreadsheet), all user-defined components shall be verified before initial use and after changes. Documentation of such shall be readily available for review. Appropriate protections must be included to preclude inadvertent changes to user-defined equation or macros. Printouts from any spreadsheet should include that information used to calculate the result;

h) Software version control methods must be in place to document the software version currently used as well as data reports with the date and time of generation and the software version used to generate the data report. Software that includes user-defined calculations and/or macros shall also track revisions to the user-defined customization using version information.

i) and confidentiality of data entry or collection, data storage, data transmission and data processing.

j) Computers and automated equipment are to be maintained to ensure proper function and must have appropriate environmental and operating conditions necessary to maintain the integrity of NDA measurement data and information.

k) Procedures are to be established and implemented for the maintenance of security of data, including the prevention of unauthorized access to and the unauthorized amendment of, computer records.

l) An inventory of all applicable software used to generate NDA characterization data shall be maintained that identifies the software name, version, classification and exemption status (DOE 0 414.C or latest version), operating environment, and the person and organization responsible for the software.

m) Maintain a historical file of software, software operating procedures, software changes, and software version numbers.

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

ED_006532_00005078-00249

data and its use with NDA measurement information. The AK ratios or scaling factors must be appropriate to the generation point of the waste, process component, etc.

a) AK Documentation

The use of AK information concerning the radiological composition of a waste type or process component must be documented either in an AK summary report for that waste type/component or other controlled document. Should this information be contained in AK package(s) prepared to meet other general waste characterization requirements, it need not be duplicated in other controlled documents that address the radiological properties of the waste stream. However, all relevant information must be included in the AK record.

All ratios or scaling factors used must be technically sound and based on known, documented relationships or correlations. Uncertainties reported when using ratios and scaling factors are used must include the uncertainty in the ratio or scaling factor.

The type and quantity of supporting documentation may vary by waste stream and shall be compiled in a written record that includes a summary identifying all sources of information used to delineate the waste stream's isotopic distribution or radionuclide scaling factors. The basis and rationale for the delineation shall be clearly summarized in an AK report and traceable to referenced documents. Assumptions made in this rationale shall be identified. The following information should be included as part of the AK written record:

1) Map of the site with the areas and facilities involved in waste generation and process equipment identified,
2) Facility mission description as related to radionuclide-bearing materials and their management (e.g., routine production, fuel research and development, and experimental processes),
3) Description of the specific site locations (such as the area or building) and operations relative to the isotopic composition of the uranium bearing wastes and process components they generated,
4) Waste identification or categorization schemes used at the facility relevant to the waste material's isotopic distribution (e.g., the use of codes that correlate to a specific isotopic distribution and a description of the isotopic/radionuclide composition of each waste stream),
5) Information regarding the waste's physical and chemical composition that could affect the isotopic distribution (e.g., processes used to remove ingrown daughters or alter its expected contribution based solely on radioactive decay kinetics), and
6) Statement of all numerical adjustments applied to derive the material's isotopic distribution (e.g., scaling factors, decay/in-growth corrections and secular equilibrium considerations).

Documentation must be sufficient to enable independent calculation of the scaling factor or ratio of interest.

ED_006532_00005078-00250

b) Supplemental AK Information

Supplemental AK information should be obtained dependent on availability. The amount and type of this information cannot be mandated, but information should be collected as appropriate to support contentions regarding the waste's isotopic distribution. This information is used to compile the waste's AK written record. Supplemental AK documentation that may be used includes but is not limited to information from the following sources:

1) Safeguards and security, materials control and accountability, and other nuclear materials control systems or programs and the data they generated,
2) Reports of nuclear safety or criticality, accidents/excursions involving the use of special nuclear material (SNM), or nuclear material,
3) Waste packaging procedures, waste disposal, building or nuclear material management area logs or inventory records, and site databases that provide information on SNM or nuclear materials,
4) Test plans, research project reports, or laboratory notebooks that describe the radionuclide content of materials used in experiments,
5) Information from site personnel (e.g., documented interviews), and
6) Historical analytical data relevant to the isotopic distribution of the waste stream.

c) AK Discrepancy Resolution

If there is any form of discrepancy between AK information related to isotopic ratios or composition, the NDA organization is responsible for having the sources of the discrepancy evaluated to determine information credibility. Information that is not credible or information that is limited in its applicability to the NDA characterization effort will be identified as such, and the reasons for dismissing it will be justified in writing. Limitations concerning the information will be documented in the AK record and summarized in the AK report. In the event the discrepancy cannot be resolved, the site will perform direct measurements for the impacted population of containers or process items. If discrepancies "result in a change to the original determinations, the AK summary will be updated.

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.

ED_006532_00005078-00251

1) Title and contact information, including:
   i. Report title (e.g., "NDA Measurement Item Report"),
   ii. Name of NDA organization,
   iii. 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
   iv. Identification of project name, site, or facility NDA measurement items are associated with.
2) Measurement item identification and QC information:
   i. Measurement item identification/designator and other identifiers/designations as applicable (e.g., the clients own identifier),
   ii. Date(s) of NDA data acquisition,
   iii. Analysis, background, and QC file names,
   iv. Measurement item description,
   v. NDA field worksheet file name, log name, or other identifier,
   vi. Gross/net weight, if applicable,
   vii. NDA measurement live time, and
   viii. Location of NDA measurement system, site name, facility name, building name, and other identifying information.

3) Primary radionuclide measurement results:
   i. Primary NDA measurement quantitation method (e.g., gamma, neutron),
   ii. Primary radioisotopes and their associated TMU s in appropriate units, (for example, gram, activity, activity concentration, MDA, and % uncertainty),
   iii. Total radionuclide mass, activity, concentration, and associated TMU,
   iv. ^{235}U fissile gram equivalent and associated TMU (gram), and
   v. Other primary quantities such as uranium enrichment weight percent (wt%) and associated wt% TMU.
4) NDA acquisition and analysis information:
   i. NDA detector or system identification,
   ii. 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),
   iii. Identification of real time radiography examination files, if applicable,
   iv. The acquisition software identification and version, and
   v. Analysis software identification and version.
5) Comment/Narrative section:
   i. Name or reference to procedures used to acquire the NDA measurement data analyze the data, and acquire supporting data/information used in analysis,

ED_006532_00005078-00252

ii. Name or reference to QC procedures utilized in the acquisition and processing of the data,

iii. Identification or reference to WRM and check source(s) used for calibration and/or QC activities,

iv. Identification of or reference to calibration procedures and records and/or location, and,

v. If not specified elsewhere, definition of the quoted uncertainties (i.e., one σ, two σ). When TMU is reported differently on the batch cover sheet of the IMS, the method of expressing TMU shall be specified on the NDA measurement item report sheet or the applicable procedures referenced.

The NDA measurement item report is to have the analyst signature and date and the independent technical reviewer signature and date.

b) NDA Data Review

All NDA measurement data must be reviewed and approved by qualified personnel prior to being reported. At a minimum, the data and analysis must be reviewed by an independent technical reviewer (a second qualified person). This reviewer shall be an individual other than the data generator (analyst) who is qualified to have performed the initial work. The independent technical reviewer shall verify, at a minimum, the following information:

1) 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;
2) "calibration verification" measurements were performed and reviewed as acceptable;
3) NDA system data acquisition and reduction were conducted in a technically correct manner in accordance with current methods (verification of procedure and revision);
4) 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;
5) Proper constants such as half-lives, branching ratios, attenuation values, neutron yields, gamma libraries were used;
6) Data were reported in the proper units and correct number of significant figures;
7) Values that are not verifiable to within rounding or significant difference discrepancies must be rectified prior to completion of independent technical review;
8) The data have been reviewed for transcription errors;
9) Calibrations have been documented;
10) Standards used are traceable to nationally recognized certificates.

ED_006532_00005078-00253

c) NDA Data Verification

Data verification is a systematic confirmation by examination and provision of evidence that specified requirements have been met data to ensure that the required data quality characteristics have been obtained. The verification process ensures that applicable quality controls have been properly implemented and data validity per program requirements has been met. Verification activities are usually performed at the batch level where all QA elements ranging from NDA measurement reports to compliance with applicable regulations are collected, collated, and prepared for submittal. NDA measurement data reports are to be provided to the client on a batch basis as determined with and agreed to by the client.

1) Batch data reports are to be prepared for each measurement batch on standard form (hard copy or electronic equivalent). Batch data reports shall at a minimum include the following:
   i. NDA organization name, NDA measurement system identification, batch number, NDA measurement item identifications included in the batch, date and signature release by authorized personnel;
   ii. Table of contents
   iii. QC data, backgrounds, replicate data, and control charts, etc., for the relevant batch time period;
   iv. Data verification per the NDA service provider QA Plan, and as per applicable procedures.
2) Batch reports must be reviewed and approved by qualified personnel before being submitted. Only appropriately trained and qualified personnel shall be allowed to perform data verification/review. Verification reviews shall ensure:
   a) The QC documentation for the batch report is complete and includes as applicable a list of containers in the set or batch and applicable set or batch QC results.
   b) Data were collected as described in the planning documents and are complete and correct. All batch data reports must be approved by the project manager or designee. The project manager shall verify at a minimum the following information:
      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.

ED_006532_00005078-00254

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 its' the term of operation. The testing will be single-blind and representative of the matrix types and configurations, and analytes (^{235}U, ^{238}U, etc.) to be characterized;

b) Unacceptable NDA results for PE test sample(s), as determined per PE program criteria, will require the NDA organization to implement corrective action procedures and submit a corrective action plan to the PE program or applicable oversight agency. Results of the corrective action plan shall be documented and available for review.

c) Documentation of successful capability demonstration such as a Certification Statement or letter of concurrence from the qualifying agency must be acquired .and retained by the NDA organization. All associated supporting data necessary to reproduce the PE measurement results as contained in the Certification Statement or equivalent document must be retained by the NDA organization.

d) Once the initial capability demonstration is successfully completed, continuing demonstration of method performance is to be accomplished through the periodic "calibration verification" measurements as well as all applicable QC requirements.

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.

ED_006532_00005078-00255

In addition to the replicate requirement are pre- and post-batch QC checks (e.g., background and energy calibration checks). A batch can be fewer than 20 items as for the case where there are fewer than 20 similar measurement items available for analysis or other driving circumstances, such as throughput requirements.

For each measurement item batch, QC measures are to be performed before commencement of a batch and at the end of the batch. An analytical batch may span a period of more than one day, but the requirement to perform QC checks per day is not superseded. The replicate QC measure does not have to be performed twice per batch, but rather once. Performance checks shall bracket the NDA measurements which comprise the batch. Out of control performance checks for a given NDA instrument shall cause the batch data to be considered suspect. Corrective actions shall be in place to evaluate the measurement item results for the affected batch.

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.

ED_006532_00005078-00256

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

ED_006532_00005078-00257

measurement is acquired by randomly selecting one measurement item that has been processed through the NDA system for the batch. This measurement item is then to be re-measured using the same NDA system, software, and acquisition/reduction parameters. Data analysis is to be performed independently for the two measurements. The second measurement of the item is to be performed any time before the start of the next data set or batch. This repeat measurement is then the replicate for that batch. A minimum of one replicate measurement is required for each batch. For a randomly selected replicate measurement item that corresponds to a measurement below the lower limit of quantitation (LLQ), the 95% uncertainty ranges of the pair of measurements must overlap.

When two replicates are utilized to assess repeatability, the data should be evaluated using the Relative Percent Difference (RPD) as follows:

$$\frac{|D-S|}{S} \times 100\% \leq 25\%$$

Where:

S = initial randomly selected measurement item

D = duplicate result for measurement item S

An acceptable RPD shall be less than or equal to 25% or other criteria specifically requested by the client. A control chart of the RPD shall be maintained for trending analysis. Procedures shall be established for the collection, processing and periodic evaluation of replicate data. Alternate methods for determining repeatability and assessing its acceptability may be implemented by the NDA organization provided they are technically justifiable, documented and available for review. The replicate data is to be monitored using control charts or tolerance charts to ensure the instrument reproducibility is within statistical control.

Check sources used for QC checks should be traceable, long-lived and provide adequate counting statistics for a relatively short count time. If the check source is not traceable, it should be correlated with a traceable source or well known, characterized and documented.

All performance data shall be monitored on an as-recorded basis and over time using control charts and trending techniques. Most monitoring techniques assume that measurement data are distributed normally and that observations are independent. The assumption of normality should be assessed prior to implementation of a control regimen. The NDA organization is responsible for determining acceptance criteria for as-recorded and long term data trending. Recommended control chart limits and actions levels are contained in Table -2. 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

ED_006532_00005078-00258

range(s), the required responses in Table -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 -3 Range of Applicability.

Table -3 Range of Applicability

Category	Acceptability Range[a]	Required Response
Acceptable Range	$\lvert Data \rvert$[b] $\leq 2\sigma$[c]	No action required
Warning Range	2σ[c] $< \lvert Data \rvert \leq 3\sigma$[c]	The performance check shall be rerun no more than two times. If the rerun performance result is within 2σ, then the additional performance checks shall be documented and work may continue. If the system does not fall within the ± 2σ after two rerun performance checks, then the required response for Action Range shall be followed.
Action Range	$\lvert Data \rvert > 3\sigma$[c]	Work shall stop and the occurrence shall be documented and appropriately dispositioned (e.g., initiating a nonconformance report). The NDA system shall be removed from service pending successful resolution of the failure cause. All assays performed since the last acceptable performance check, are suspect, pending satisfactory resolution. At a minimum, a "calibration verification" is required prior to returning the system back to service.

a - American National Standards Institute. *Nondestructive Assay Measurement Control and Assurance*, ANSI N15.36.

b - absolute value

c - the standard deviation is only based on the reproducibility of the data check measurements themselves. This is not TMU.

ED_006532_00005078-00259