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**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**



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**Department of Defense (DoD)**  
**Quality Systems Manual (QSM) for Environmental  
Laboratories**  
**Version 5.0**

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## **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**

  
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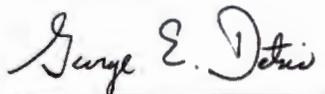
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## Preface

The Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the Department of Energy (DOE) Consolidated Audit Program (DOECAP) 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.

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## 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 |

|  |           |
|--|-----------|
| 5.10 REPORTING THE RESULTS .....   | 32        |
| <b>6.0 HAZARDOUS AND RADIOACTIVE MATERIALS MANAGEMENT AND HEALTH AND SAFETY PRACTICES.....</b> | <b>35</b> |
| 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 |

|  |     |
|--|-----|
| 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**

|   |            |
|---|------------|
| <b>1.0 INTRODUCTION.....</b>                        | <b>173</b> |
| <b>2.0 LCS LIMIT TABLES .....</b>                   | <b>173</b> |
| 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        |

|   |     |
|---|-----|
| 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 REQUIREMENTS .....                          | 247 |
| 3.0 QC ACTION LEVELS AND RESPONSE.....                 | 248 |

# Volume 1, Module 1: Proficiency Testing (PT)

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## 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

(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.

## **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

provided by an approved supplier. The PT suppliers must be approved by the PEO/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:

- 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.

# Volume 1, Module 2: Quality Systems General Requirements

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## 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.

## 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.

**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

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.

**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.

**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;

- 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.

#### 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;

- 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.

## **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.

## **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)**

## **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.

#### **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.

#### **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)**

## 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

- “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 it was not
  - iv) Performing improper manual integrations, including peak shaving, peak enhancing, or baseline manipulation to meet QC criteria or to avoid corrective actions
- f) Concealing a known analytical or sample problem
- g) Concealing a known improper or unethical behavior or action
- h) Failing to report the occurrence of a prohibited practice or known improper or unethical act to the appropriate laboratory or contract representative, or to an appropriate government official.

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

### **5.3.3 DoD/DOE (Requirement)**

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

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

- b) A Storage Blank must be stored with all volatile organic samples, regardless of suspected concentration levels. Storage Blanks shall be used to determine if cross-contamination may have occurred. Laboratories shall have written procedures and criteria for evaluating Storage Blanks, appropriate to the types of samples being stored. The Storage Blanks shall be stored in the same manner as the customer samples. The Storage Blanks shall be analyzed 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 ( $\pm$ ) around the

reported analytical results at a specified confidence level. A laboratory may report the in-house, statistically-derived LCS control limits based on historical LCS recovery data as an estimate of the minimum laboratory contribution to analytical uncertainty at a 99% confidence level. For testing laboratories, the laboratory shall ensure that the equipment used can provide the analytical portion of measurement uncertainty needed by the customer.

#### **5.4.7.1 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.

- 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;

- 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:

| Performance Check  | Frequency   | Acceptance Criteria   |
|--|---|---|
| Balance calibration check<br>[Using two standard weights that bracket the expected mass]   | Daily prior to use  | Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$ , whichever is greater<br>Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{mg}$ , whichever is greater |
| Verification of standard mass<br>[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)<br>[use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily] | Refrigerators: $0^{\circ}\text{C}$ to $6^{\circ}\text{C}$<br>Freezers: $\leq -10^{\circ}\text{C}$   |
| Thermometer verification check<br>[Using a thermometer traceable to the SI through an NMI]<br>[Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.]<br>[If only a single temperature is used, at the temperature of use] | Liquid in glass: Before first use and annually<br>Electronic: Before first use and quarterly  | Apply correction factors or replace thermometer   |
| Volumetric labware   | Class B: By lot before first use<br>Class A and B: Upon evidence of deterioration   | Bias: Mean within $\pm 2\%$ of nominal volume<br>Precision: RSD $\leq 1\%$ of nominal volume (based on 10 replicate measurements)                           |
| Non-volumetric labware<br>[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 $\pm 3\%$ of nominal volume<br>Precision: RSD $\leq 3\%$ of nominal volume (based on 10 replicate measurements)                           |

| Performance Check             | Frequency  | Acceptance Criteria  |
|-------------------------------|--|--|
| Mechanical volumetric pipette | Daily before use   | Bias: Mean within $\pm 2\%$ of nominal volume<br>Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements)<br>[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<br>Replace if deterioration is evident  |
| Drying oven temperature check | Daily prior to and after use   | Within $\pm 5\%$ of set temperature  |
| Water purification system     | Daily prior to use   | Per Laboratory SOP   |
| Radiological Survey Equipment | Daily prior to use<br>[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.

#### **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.,  $1.00 \pm 0.01$  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  $1.00\text{g} \pm 0.01\text{g}$ , 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

- 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:

- 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*):

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.

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.

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 identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

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

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

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

### **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:

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.

### **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.

### **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

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

Waste brokering and TSD 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 TSD 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 TSDs. 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 TSDs.

#### **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

# Volume 1 Module 3: Quality Systems for Asbestos Testing

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## 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.

#### **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.

# Volume 1 Module 4: Quality Systems for Chemical Testing

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## 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

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.

### **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.

### **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.

### 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;

- 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  $\pm 3$  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.

### **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

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 1/10<sup>th</sup> the amount measured in any associated sample, or 1/0<sup>th</sup> 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.

#### **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.

# **Volume 1, Module 5: Quality Systems for Microbiological Testing**

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

# VOLUME 1, MODULE 6: Quality Systems for Radiochemical Testing

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## 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  $b$  of non-detection (Type II error), while accepting a probability  $a$  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  $a$  and  $b$  probabilities are assumed to be 0.05. MARLAP utilizes the Minimum Detectable Concentration (MDC) term instead of MDA.
- b) MDA Factors and Conditions - MDAs are determined based on factors and conditions such as instrument settings and matrix type, which influence the measurement. The MDA is used to evaluate the capability of a method relative to the required detection reporting limit (RL). Sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency shall be optimized to result in sample MDAs less than or equal to the

RLs. If RLs are not achieved, then the cause shall be addressed comprehensively in the Case narrative.

- c) MDA Calculation - The basic MDA calculation shall be based on the concepts developed by L. A. Currie from his paper "Limits for Qualitative Detection and Quantitative Determination, *Analytical Chemistry*, March, 1968, Vol. 40, or from the MARLAP Manual Chapter 20. The following general equations derived from the work of L. A. Currie can be used to calculate the MDA.

i) With a Blank Population:

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

K = efficiency \* e<sup>-λt</sup> \* aliquot fraction \* tracer recovery \* Yield

T<sub>s</sub> = count time of the sample in minutes

s<sub>b</sub> = standard deviation of the blank population where the blank population is in net blank counts in count time T<sub>s</sub>

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.

- 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<sup>-t</sup> \* aliquot fraction \* tracer Recovery \* Yield

T<sub>S</sub> = count time of the sample in minutes

T<sub>B</sub> = 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 probabilities assumed for the decision level shall also be used for the calculation of the MDA.

### 1.5.2.1.2 DoD/DOE (Requirement)

- a) Decision Level (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 ( $L_c$ ) 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:

$$DL = \frac{(t \cdot S_B) + \overline{R_B}}{E \cdot R \cdot IDF \cdot W}$$

Where:

DL = the decision level in disintegrations per minute per unit volume or weight (dpm/unit);

$S_B$  = the standard deviation of a set of appropriate blank net count rate after background subtraction for blanks counted for the same length of time as the sample;

$R_B$  = the average blank count rate in counts per minute (cpm);

t = the student t factor for appropriate degrees of freedom and confidence level;

E = the fractional detector efficiency (c/d) for the sample;

R = the fractional chemical yield for the sample;

IDF = the ingrowth or decay factor for the sample; and

W = the weight or volume of the sample.

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

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

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).

## 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:  $|Z_{\text{Blank}}| \leq 3$  (MARLAP 18.4.1) or a laboratory shall use Method Blank in-house control limits of  $\pm 3 \sigma$  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).

### 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  $LCS \pm 3 \sigma$  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

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.

### 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

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  $\pm 3$  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:

- 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.

- 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  $\pm 40$  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  $\pm 40$  keV of the expected peak energy.

h) Background Requirements:

- i) The background total counts (or counts per unit time) for each target analyte and tracer isotope ROI shall be analyzed on each detector and documented.
- ii) The background for each ROI shall be sufficiently low to ensure that required detection limits are met.
- iii) The limits of acceptability for each background ROI shall be documented. These shall be set such that RLs can be obtained for backgrounds at the limit of acceptability.
- iv) Background count times shall be equal to or longer than sample count times.

i) Detector Response Determination Requirements

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  $\leq 100$  keV.
- iii) The tracer peak energy for each sample and QC sample shall be within  $\pm 50$  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.

### 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

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.

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

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.

- 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.

- 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.

# Volume 1, Module 7: Quality Systems for Toxicity Testing

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

# Appendix A: Reporting Requirements

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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.

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;

- 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;

- 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.

## Appendix B: Quality Control Requirements

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**Table – 1. Organic Analysis by Gas Chromatography**

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action                              | Flagging Criteria            | Comments  |
|---|--|--|--|------------------------------|---|
| <b>Breakdown check (Endrin/DDT Method 8081 only)</b>                      | Before sample analysis and at the beginning of each 12-hour shift.           | Degradation of DDT and Endrin must each be $\leq 15\%$ .   | Correct problem, then repeat breakdown checks. | Flagging is not appropriate. | No samples shall be run until degradation of DDT and Endrin is each $\leq 15\%$ .   |
| <b>Initial Calibration (ICAL) for all analytes (including surrogates)</b> | At instrument set-up and after ICV or CCV failure, prior to sample analysis. | ICAL must meet one of the three options below:<br><br>Option 1: RSD for each analyte $\leq 20\%$ ;<br><br>Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$ ;<br><br>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.<br><br>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.<br><br>No samples shall be analyzed until ICAL has passed. |

**Table – 1. Organic Analysis by Gas Chromatography**

| <b>QC Check</b>                                     | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>                                | <b>Flagging Criteria</b>     | <b>Comments</b>  |
|---|--|--|---|------------------------------|--|
| <b>Retention Time window position establishment</b> | 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.   |
| <b>Retention Time (RT) window width</b>             | At method set-up and after major maintenance (e.g., column change).                  | RT width is $\pm 3$ times standard deviation for each analyte RT from the 72-hour study.   | NA.   | NA.                          | Calculated for each analyte and surrogate.   |
| <b>Initial Calibration Verification (ICV)</b>       | Once after each ICAL, analysis of a second source standard prior to sample analysis. | All reported analytes within established RT windows.<br><br>All reported analytes within $\pm 20\%$ of true value.                                       | Correct problem, rerun ICV. If that fails, repeat ICAL. | Flagging is not appropriate. | No samples shall be analyzed until calibration has been verified with a second source. |

**Table – 1. Organic Analysis by Gas Chromatography**

| QC Check   | Minimum Frequency   | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments  |
|--|---|--|--|---|---|
| <b>Continuing Calibration Verification (CCV)</b> | 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.<br><br>All reported analytes and surrogates within $\pm 20\%$ of true value. | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br><br>or<br><br>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.          |
| <b>Method Blank (MB)</b>                         | 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. |

**Table – 1. Organic Analysis by Gas Chromatography**

| <b>QC Check</b>  | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>  | <b>Corrective Action</b>  | <b>Flagging Criteria</b>  | <b>Comments</b>  |
|--|----------------------------|---|---|---|--|
| <b>Laboratory Control Sample (LCS)</b>                       | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.<br><br>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.                       |
| <b>Matrix Spike (MS)</b>                                     | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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. |
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>RPD $\leq$ 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.   |

**Table – 1. Organic Analysis by Gas Chromatography**

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action   | Flagging Criteria   | Comments  |
|---|--|--|---|---|---|
| <b>Surrogate Spike</b>                                  | 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.  |
| <b>Confirmation of positive results (second column)</b> | 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.<br>Results between primary and secondary column RPD ≤ 40%. | NA.   | Apply J-flag if RPD > 40%. Discuss in the case narrative.   | Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column. |

**Table – 2. Organic Analysis by High-Performance Liquid Chromatography (HPLC)**

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action                                       | Flagging Criteria            | Comments   |
|---|--|--|---|------------------------------|--|
| <b>Initial Calibration (ICAL) for all analytes (including surrogates)</b> | At instrument set-up and after ICV or CCV failure, prior to sample analysis.         | ICAL must meet one of the three options below:<br><br>Option 1: RSD for each analyte $\leq 20\%$ ;<br><br>Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$ ;<br><br>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.<br><br>No samples shall be analyzed until ICAL has passed. |
| <b>Retention Time window position establishment</b>                       | 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.   |
| <b>Retention Time (RT) window width</b>                                   | At method set-up and after major maintenance (e.g., column change).                  | RT width is $\pm 3$ times standard deviation for each analyte RT from the 72-hour study.   | NA.   | NA.                          | Calculated for each analyte and surrogate.   |
| <b>Initial Calibration Verification (ICV)</b>                             | Once after each ICAL, analysis of a second source standard prior to sample analysis. | All reported analytes within established RT windows.<br><br>All reported analytes within $\pm 15\%$ 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.                             |

**Table – 2. Organic Analysis by High-Performance Liquid Chromatography (HPLC)**

| QC Check   | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments   |
|--|--|--|--|---|--|
| <b>Continuing Calibration Verification (CCV)</b> | 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.<br><br>All reported analytes and surrogates within $\pm 15\%$ true value.  | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br><br>or<br><br>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.<br><br>Retention time windows are updated per the method. |
| <b>Method Blank (MB)</b>                         | 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.  |
| <b>Laboratory Control Sample (LCS)</b>           | One per preparatory batch.   | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.<br><br>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.                             | Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.   |

**Table – 2. Organic Analysis by High-Performance Liquid Chromatography (HPLC)**

| QC Check   | Minimum Frequency          | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments   |
|--|----------------------------|--|---|--|--|
| <b>Matrix Spike (MS)</b>                                     | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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. |
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>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.   |
| <b>Surrogate Spike</b>                                       | 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.   |

**Table – 2. Organic Analysis by High-Performance Liquid Chromatography (HPLC)**

| QC Check  | Minimum Frequency                       | Acceptance Criteria   | Corrective Action | Flagging Criteria   | Comments   |
|---|---|---|-------------------|---|--|
| <b>Confirmation of positive results (second column)</b> | All positive results must be confirmed. | Calibration and QC criteria for second column are the same as for initial or primary column analysis.<br><br>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.<br><br>Use project-specific reporting requirements if available; otherwise, use method requirements, if available; otherwise, report the result from the primary column. |

**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  |
|-------------------------------|-------------------------------------|--|-------------------|------------------------------|---|
| <b>Soil drying procedure</b>  | 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. |
| <b>Soil sieving procedure</b> | 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. |   |

**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  |
|---------------------------------|---|--|--|--|---|
| <b>Soil grinding procedure</b>  | 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.   |   |
| <b>Soil grinding blank</b>      | 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. |
| <b>Soil subsampling process</b> | 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.   |   |

**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   |
|---|--|--|---|--|--|
| <b>Soil sample triplicate</b>   | At the subsampling step, one sample per batch.<br><br>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.<br><br>The RSD for results above the LOQ must not exceed 20%.  | Examine the project-specific requirements.<br>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. |  |
| <b>Aqueous sample preparation</b>   | 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.  |
| <b>Initial Calibration (ICAL) for all analytes (including surrogates)</b> | At instrument setup and after ICV or CCV failure, prior to sample analysis.  | ICAL must meet one of the three options below:<br><br>Option 1: RSD for each analyte $\leq 15\%$ ;<br><br>Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$ ;<br><br>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.<br><br>No samples shall be analyzed until ICAL has passed. |
| <b>Initial Calibration Verification (ICV)</b>                             | Once after each ICAL, analysis of a second source standard prior to sample analysis.   | All reported analyte(s) and surrogates within $\pm 20\%$ of true value.  | Correct problem. Rerun ICV. If that fails, repeat ICAL.   | Flagging is not appropriate.   | No samples shall be analyzed until calibration has been verified with a second source.                             |

**Table – 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   |
|--|--|--|--|---|--|
| <b>Continuing Calibration Verification (CCV)</b> | Before sample analysis, after every 10 field samples, and at the end of the analysis sequence. | All reported analytes and surrogates within $\pm 20\%$ of the true value.  | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br>or<br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.          |
| <b>Method Blank (MB)</b>                         | 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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

**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   |
|--|----------------------------|---|---|--|--|
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch. | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>Use LCS Tables 8330B for HPLC analysis.</p> <p>Use LCS Tables 8321 for LC/MS or LC/MS/MS analysis.</p> | 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. | <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.</p> | <p>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.</p> <p>Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> |
| <b>Matrix Spike (MS)</b>               | One per preparatory batch. | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p>   | 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.  |

**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   |
|--|----------------------------|---|---|--|--|
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD). | Examine the project-specific requirements.<br>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.<br><br>The data shall be evaluated to determine the source of difference. |
| <b>Surrogate Spike</b>                                       | All field and QC samples.  | QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or<br><br>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.   |

**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   |
|---|---|--|---------------------------|---|--|
| <b>Confirmation of positive results (second column)</b> | All positive results must be confirmed. | <p>Calibration and QC criteria are the same for the confirmation analysis as for initial or primary column analysis.</p> <p>Results between primary and second column RPD <math>\leq</math> 40%.</p> | Report from both columns. | Apply J-flag if RPD > 40%. Discuss in the case narrative. | <p>Use of a UV detector with a UV diode array detector or vice versa is not considered a valid confirmation technique.</p> <p>Confirmation analysis is not needed if LC/MS or LC/MS/MS was used for the primary analysis.</p> <p>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.</p> <p>Use project specific reporting requirements if available; otherwise, report from the primary column.</p> |

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

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action                                | Flagging Criteria            | Comments  |
|---|--|--|--|------------------------------|---|
| <b>Tune Check</b>   | 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.  |
| <b>Performance Check ( Method 8270 only)</b>                              | At the beginning of each 12-hour period, prior to analysis of samples. | Degradation ≤ 20% for DDT.<br>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.<br>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.     |
| <b>Initial calibration (ICAL) for all analytes (including surrogates)</b> | At instrument set-up, prior to sample analysis                         | Each analyte must meet one of the three options below:<br><u>Option 1:</u> RSD for each analyte ≤ 15%;<br><br><u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$ ;<br><br><u>Option 3:</u> 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.<br>No samples shall be analyzed until ICAL has passed.<br>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. |

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

| <b>QC Check</b>                                     | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>  | <b>Corrective Action</b>                                | <b>Flagging Criteria</b>     | <b>Comments</b>  |
|---|--|---|---|------------------------------|--|
| <b>Retention Time window position establishment</b> | 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.<br><br>On days when ICAL is not performed, the initial CCV is used. | NA.   | NA.                          | Required for each analyte and surrogate.   |
| <b>Evaluation of Relative Retention Times (RRT)</b> | With each sample.  | RRT of each reported analyte within $\pm 0.06$ RRT units.   | Correct problem, then rerun ICAL.                       | NA                           | RRTs may be updated based on the daily CCV.<br><br>RRTs shall be compared with the most recently updated RRTs. |
| <b>Initial Calibration Verification (ICV)</b>       | Once after each ICAL, analysis of a second source standard prior to sample analysis. | All reported analytes within $\pm 20\%$ of true value.  | Correct problem. Rerun ICV. If that fails, repeat ICAL. | Flagging is not appropriate. | No samples shall be analyzed until calibration has been verified with a second source.                         |

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

| QC Check   | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments   |
|--|--|--|--|--|--|
| <b>Continuing Calibration Verification (CCV)</b> | 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 $\pm 20\%$ of true value.<br><br>All reported analytes and surrogates within $\pm 50\%$ for end of analytical batch CCV. | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br><br>or<br><br>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.<br><br>If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met. |
| <b>Internal standards (IS)</b>                   | Every field sample, standard and QC sample.  | Retention time within $\pm 10$ 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.<br><br>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.<br><br>Flagging is not appropriate for failed standards. |  |

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

| <b>QC Check</b>                        | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>  | <b>Flagging Criteria</b>  | <b>Comments</b>  |
|--|----------------------------|--|---|---|--|
| <b>Method Blank (MB)</b>               | 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.<br><br>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.<br><br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.   |
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.<br><br>Results may not be reported without a valid LCS.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.                |
| <b>Matrix Spike (MS)</b>               | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. | Examine the project-specific requirements.<br>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.<br><br>If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error. |

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

| QC Check   | Minimum Frequency          | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments   |
|--|----------------------------|--|---|--|--|
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch. | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).</p> | 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. | <p>MSD: Must contain all surrogates and all analytes to be reported.</p> <p>The data shall be evaluated to determine the source of difference.</p> |
| <b>Surrogate Spike</b>                                       | 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.   |

| <b>Table - 5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)</b> |   |  |  |                              |  |
|--|---|--|--|------------------------------|--|
| <b>QC Check</b>  | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>   | <b>Flagging Criteria</b>     | <b>Comments</b>  |
| <b>Tune Check</b>  | 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.           |
| <b>Retention Time window defining mix</b>  | 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. |  |
| <b>GC column performance check (for SP-2331 column or equivalent)</b>  | At the beginning and end of each 12-hr period during which samples or calibration solutions are analyzed. | <p><u>Peak separation between 2,3,7,8-TCDD and other TCDD isomers:</u> Resolved with a valley of <math>\leq 25\%</math>.</p> <p><u>For calibration verification standard only:</u> 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 <math>\leq 50\%</math>, per method.</p> | Correct problem, then repeat column performance checks.          | Flagging is not appropriate. | Needed only if using a column other than DB-5 or equivalent. |

| <b>QC Check</b>   | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>                                | <b>Flagging Criteria</b>     | <b>Comments</b>  |
|---|---|--|---|------------------------------|--|
| <b>GC Column performance check (for DB-5 column or equivalent)</b>      | At the beginning and end of each 12-hr period during which samples or calibration solutions are analyzed.<br><br>Included with the ICAL standard (CC3) and the calibration verification standard. | <u>Peak separation of standard CC3:</u> Peak between the <sup>13</sup> C-2,3,7,8-TCDD and <sup>13</sup> C-1,2,3,4-TCDD must be resolved with a valley of ≤ 25%;<br><br><u>For calibration verification standard only:</u> 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. |
| <b>Initial calibration (ICAL) for all analytes identified in method</b> | At instrument set-up and after ICV or CCV failure, prior to sample analysis.  | Ion abundance ratios must be in accordance with the method.<br><br>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.                                |

| <b>Table - 5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)</b> |   |   |   |  |   |
|--|---|---|---|--|---|
| <b>QC Check</b>  | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>  | <b>Corrective Action</b>  | <b>Flagging Criteria</b>   | <b>Comments</b>   |
| <b>Initial Calibration Verification (ICV)</b>  | Once after each ICAL, analysis of a second source standard prior to sample analysis.                    | <p>Ion abundance specified in the method must be met for all PCDD/PCDF peaks, including labeled internal and recovery standards.</p> <p>Sensitivity criteria of an S/N ratio &gt; 2.5 for unlabeled PCDD/PCDF ions and &gt; 10 for labeled internal and recovery standards.</p> <p>All reported analytes and IS within <math>\pm 20\%</math> of true value.</p> | 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.  |
| <b>Calibration Verification (CCV)</b>  | At the beginning of each 12-hr period of sample analysis, after successful GC and MS resolution checks. | <p>Ion abundance specified in the method must be met for all PCDD/PCDF peaks, including labeled internal and recovery standards.</p> <p>Sensitivity criteria of an S/N ratio &gt; 2.5 for unlabeled PCDD/PCDF ions and &gt; 10 for labeled internal and recovery standards.</p> <p>All reported analytes and IS within <math>\pm 20\%</math> of true value.</p> | <p>Recalibrate, and reanalyze all affected samples since the last acceptable CCV;</p> <p>or</p> <p>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.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.</p> | <p>Results may not be reported without valid calibration verification.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> |

| <b>Table - 5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)</b> |   |  |   |  |   |
|--|---|--|---|--|---|
| <b>QC Check</b>  | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>  | <b>Flagging Criteria</b>   | <b>Comments</b>   |
| <b>Internal standards(IS)</b>  | 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<br><br>Flagging is not appropriate for failed standards. |   |
| <b>Sensitivity Check</b>   | At the end of 12-hr sample analysis period or at the end of analysis (whichever comes first) .<br><br>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.   |   |
| <b>Method Blank (MB)</b>   | 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.<br><br>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. |

| <b>Table - 5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)</b> |                            |  |   |  |   |
|--|----------------------------|--|---|--|---|
| <b>QC Check</b>  | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>  | <b>Flagging Criteria</b>   | <b>Comments</b>   |
| <b>Laboratory Control Sample (LCS)</b>   | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.   | Correct problem, then re-prepare 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.  |
| <b>Matrix Spike (MS)</b>   | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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. |
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b>   | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>MSD or MD: RPD of all analytes $\leq$ 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.  |

| <b>Table - 5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)</b> |                           |   |   |   |                 |
|--|---------------------------|---|---|---|-----------------|
| <b>QC Check</b>  | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>  | <b>Corrective Action</b>  | <b>Flagging Criteria</b>  | <b>Comments</b> |
| <b>Surrogate Spike</b>   | All field and QC samples. | QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits<br>or<br>in-house LCS limits if analyte(s) are not listed. | Correct problem, then re-prepare 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. |                 |

| <b>QC Check</b>                        | <b>Minimum Frequency</b>                            | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>   | <b>Flagging Criteria</b>     | <b>Comments</b>   |
|--|---|--|--|------------------------------|---|
| <b>Sample PCDD/PCDF Identification</b> | Identify all positive sample detections per method. | <p>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.</p> <p>Absolute RTs of the recovery standards must be within <math>\pm 10</math> seconds of those in the calibration verification standard.</p> <p>All ions listed in Table 8 of the method must be present in the SICP, must maximize simultaneously (<math>\pm 2</math> sec.), and must have not saturated the detector.</p> <p>S/N ratio of ISs <math>\geq 10</math> times background noise.</p> <p>Remaining ions in Table 8 of the method must have an S/N ratio <math>\geq 2.5</math> times the background noise.</p> | Correct problem, then re-prepare 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). |

| <b>Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)</b> |   |  |   |                              |   |
|---|---|--|---|------------------------------|---|
| <b>QC Check</b>   | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>                              | <b>Flagging Criteria</b>     | <b>Comments</b>   |
| <b>Resolving Power</b>  | Prior to ICAL and at the beginning and the end of each 12-hour period of analysis.  | Static resolving power $\geq$ 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.  |
| <b>Performance Check</b>  | Prior to ICAL or calibration verification. At the beginning of each 12-hr period during which samples or calibration solutions are analyzed.  | <p><u>Peak separation between 2,3,7,8-TCDD and other TCDD isomers: Resolved with a valley of <math>\leq</math> 25%.</u></p> <p>Identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram.</p> <p>Absolute retention times for switching from one homologous series to the next <math>\geq</math> 10 sec. for all components of the mixture.</p> | Correct problem then repeat column performance check. | Flagging is not appropriate. | <p>Use GC column performance check solution</p> <p>If the laboratory operates during consecutive 12-hr periods.</p> <p>No samples shall be analyzed until performance check is within criteria.</p> |
| <b>Initial calibration (ICAL) for all analytes identified in method</b>   | 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. | <p>Ion abundance ratios in accordance with the method.</p> <p>S/N ratio <math>\geq</math> 10 for all reported analyte ions. RSD <math>\leq</math> 20% for the response factors (RF) for all 17 unlabeled standards. RSD <math>\leq</math> 20% for the RFs for the 9 labeled IS.</p>  | Correct problem, then repeat ICAL.                    | Flagging is not appropriate. | <p>No samples shall be analyzed run until ICAL has passed.</p> <p>Calibration may not be forced through origin.</p>   |

**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  |
|---|--|--|---|--|---|
| <b>Initial Calibration Verification (ICV)</b> | 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 $\pm 20\%$ D of RF established in ICAL; <u>and</u> For labeled standards, RF within $\pm 30\%$ 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.  |
| <b>Calibration Verification (CCV)</b>         | 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 $\pm 20\%$ D of RF established in ICAL; <u>and</u> For labeled standards, RF within $\pm 30\%$ 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.<br><br><u>End-of-run CCV:</u> If the RF for unlabeled standards $\leq 25\%$ RPD and the RF for labeled standards $\leq 35\%$ 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. |

| <b>Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)</b> |  |  |   |  |   |
|---|--|--|---|--|---|
| <b>QC Check</b>   | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>  | <b>Flagging Criteria</b>   | <b>Comments</b>   |
| <b>Calibration Verification (CCV)</b>   |  |  | 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.<br><br>Otherwise analyze samples with positive detections, if necessary. |  |   |
| <b>Internal Standards (IS)</b>  | 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-prepare and reanalyze the samples with failed IS.  | Apply Q-flag to results of all affected samples and explain in the case narrative.   |   |
| <b>Method Blank (MB)</b>  | 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, reprepare 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. |

| <b>Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)</b> |                            |  |   |   |   |
|---|----------------------------|--|---|---|---|
| <b>QC Check</b>   | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>  | <b>Flagging Criteria</b>  | <b>Comments</b>   |
| <b>Laboratory Control Sample (LCS)</b>  | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.   | Correct problem, then re-prepare 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.<br>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.<br>Results may not be reported without a valid LCS.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.   |
| <b>Matrix Spike (MS)</b>  | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.   | Examine the project-specific requirements.<br>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.<br>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. |
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b>  | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br>MSD or MD: RPD of all analytes $\leq$ 20% (between MS and MSD or sample and MD). | Examine the project-specific requirements.<br>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.  |

| <b>Table - 6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)</b> |   |  |  |  |   |
|---|---|--|--|--|---|
| <b>QC Check</b>   | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>   | <b>Flagging Criteria</b>                         | <b>Comments</b>   |
| <b>Internal Standards (IS)</b>  | 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-prepare and reanalyze the samples with failed IS. | Apply Q-flag to results of all affected samples. |   |
| <b>Sample Estimated Maximum Possible Concentration (EMPC)</b>   | Every sample with a response S/N $\geq$ 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 $\geq$ 2.5. | NA.  | Flagging is not appropriate.                     |   |
| <b>Sample 2,3,7,8-TCDD toxicity equivalents (TEQ) concentration</b>   | All positive detections.  | Per method.  | NA.  | Flagging is not appropriate.                     | Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD. |

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

| QC Check   | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments   |
|--|--|--|--|---|--|
| <b>Initial Calibration (ICAL) for all analytes</b> | 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.<br>CVAA/Mercury: minimum 5 standards and a calibration blank.<br><br>No samples shall be analyzed until ICAL has passed. |
| <b>Initial Calibration Verification (ICV)</b>      | Once after each ICAL, analysis of a second source standard prior to sample analysis. | All reported analytes within $\pm 10\%$ 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.   |
| <b>Continuing Calibration Verification (CCV)</b>   | After every 10 field samples and at the end of the analysis sequence.                | All reported analytes within $\pm 10\%$ of the true value. | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br><br>or<br>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.<br><br>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.   |

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

| QC Check  | Minimum Frequency   | Acceptance Criteria  | Corrective Action   | Flagging Criteria   | Comments   |
|---|---|--|---|---|--|
| <b>Method Blank (MB)</b>                                  | 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.<br>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.<br>Flagging is only appropriate in cases where the samples cannot be reprepared or reanalyzed. |
| <b>Initial and Continuing Calibration Blank (ICB/CCB)</b> | 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.<br>For CCB, failures due to carryover may not require an ICAL.                            |
| <b>Laboratory Control Sample (LCS)</b>                    | One per preparatory batch.  | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>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.<br>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.                           |
| <b>Matrix Spike (MS)</b>                                  | One per preparatory batch.  | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>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.                  |

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

| QC Check  | Minimum Frequency   | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments   |
|---|---|--|--|--|--|
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b>        | One per preparatory batch.  | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br>MSD or MD: RPD of all analytes $\leq$ 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.   |
| <b>Dilution Test (Flame AA and GFAA only)</b>                       | One per preparatory batch if MS or MSD fails.                               | Five-fold dilution must agree within $\pm$ 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. |
| <b>Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)</b> | 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.   |
| <b>Method of Standard Additions (MSA)</b>                           | When dilution or post digestion spike fails and if the required by project. | NA.  | NA.  | NA.  | Document use of MSA in the case narrative.   |

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

| QC Check   | Minimum Frequency  | Acceptance Criteria  | Corrective Action   | Flagging Criteria            | Comments   |
|--|--|--|---|------------------------------|--|
| <b>Linear Dynamic Range (LDR) or high-level check standard</b> | At initial set up and checked every 6 months with a high standard at the upper limit of the range. | Within $\pm 10\%$ 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. |
| <b>Initial Calibration (ICAL) for all analytes</b>             | 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.<br>No samples shall be analyzed until ICAL has passed.          |
| <b>Initial Calibration Verification (ICV)</b>                  | Once after each ICAL, analysis of a second source standard prior to sample analysis.               | All reported analytes within $\pm 10\%$ of true value.           | Correct problem. Rerun ICV. If that fails, repeat ICAL.                       | Flagging is not appropriate. | No samples shall be analyzed until calibration has been verified with a second source.                             |

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

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments   |
|---|--|--|--|--|--|
| <b>Continuing Calibration Verification (CCV)</b>            | After every 10 field samples, and at the end of the analysis sequence. | All reported analytes within $\pm 10\%$ of the true value.   | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br>or<br>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.<br>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.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.  |
| <b>Low-level Calibration Check Standard (Low-level ICV)</b> | Daily.   | All reported analytes within $\pm 20\%$ 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. |
| <b>Method Blank (MB)</b>                                    | 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.<br>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.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.                               |

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

| QC Check   | Minimum Frequency   | Acceptance Criteria   | Corrective Action   | Flagging Criteria   | Comments  |
|--|---|---|---|---|---|
| <b>Initial and Continuing Calibration Blank (ICB/CCB)</b>                            | 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.<br><br>For CCB, failures due to carryover may not require an ICAL.   |
| <b>Interference Check Solutions (ICS) (also called Spectral Interference Checks)</b> | After ICAL and prior to sample analysis.  | <u>ICS-A:</u> Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes);<br><u>ICS-AB:</u> Within $\pm 20\%$ 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.  |
| <b>Laboratory Control Sample (LCS)</b>   | One per preparatory batch.  | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.<br><br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |
| <b>Matrix Spike (MS)</b>   | One per preparatory batch.  | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.                                |

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

| <b>QC Check</b>  | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>  | <b>Corrective Action</b>   | <b>Flagging Criteria</b>   | <b>Comments</b>   |
|--|--|---|--|--|---|
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch.   | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br>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.  |
| <b>Dilution Test</b>   | 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. |
| <b>Post-Digestion Spike (PDS) Addition (ICP only)</b>        | 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.   |
| <b>Method of Standard Additions (MSA)</b>                    | When dilution test or post digestion spike fails and if required by project.                                   | NA.   | NA.  | NA.  | Document use of MSA in the case narrative.  |

**Table – 9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)**

| <b>QC Check</b>  | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>  | <b>Corrective Action</b>   | <b>Flagging Criteria</b>     | <b>Comments</b>   |
|--|--|---|--|------------------------------|---|
| <b>Linear Dynamic Range (LDR) or High-level Check Standard</b> | At initial set-up and checked every 6 months with a high standard at the upper limit of the range. | Within $\pm 10\%$ 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. |
| <b>Tuning</b>  | Prior to ICAL.   | Mass calibration $\leq 0.1$ amu from the true value;<br>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.  |
| <b>Initial Calibration (ICAL) for All Analytes</b>             | 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.<br>No samples shall be analyzed until ICAL has passed.     |
| <b>Initial Calibration Verification (ICV)</b>                  | Once after each ICAL, analysis of a second source standard prior to sample analysis.               | All reported analytes, within $\pm 10\%$ of true value.   | Correct problem. Rerun ICV. If that fails, repeat ICAL.                      | Flagging is not appropriate. | No samples shall be analyzed until calibration has been verified with a second source.                        |

**Table – 9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)**

| QC Check  | Minimum Frequency   | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments  |
|---|---|--|--|---|---|
| <b>Continuing Calibration Verification (CCV)</b>            | After every 10 field samples and at the end of the analysis sequence. | All reported analytes within $\pm 10\%$ of the true value. | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br>or<br>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.<br>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.  |
| <b>Low-level Calibration Check Standard (Low Level ICV)</b> | Daily.  | All reported analytes within $\pm 20\%$ 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.<br><br>Low-level calibration check standard should be less than or equal to the LOQ. |

**Table – 9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)**

| QC Check  | Minimum Frequency   | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments   |
|---|---|--|--|---|--|
| <b>Internal Standards (IS)</b>                            | 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.<br><br>Reanalyze sample at 5-fold dilutions until criteria is met.<br><br>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.                          |
| <b>Method Blank (MB)</b>                                  | 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.<br><br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |
| <b>Initial and Continuing Calibration Blank (ICB/CCB)</b> | 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.<br><br>For CCB, failures due to carryover may not require an ICAL.              |

**Table – 9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)**

| QC Check   | Minimum Frequency                        | Acceptance Criteria  | Corrective Action   | Flagging Criteria   | Comments   |
|--|--|--|---|---|--|
| <b>Interference Check Solutions (ICS) (also called Spectral Interference Checks)</b> | After ICAL and prior to sample analysis. | <u>ICS-A</u> : Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes);<br><u>ICS-AB</u> : 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.<br>ICS-AB is not needed if instrument can read negative responses.  |
| <b>Laboratory Control Sample (LCS)</b>   | One per preparatory batch.               | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.             | Correct problem, then re-prepare 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.<br>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. |
| <b>Matrix Spike (MS)</b>   | One per preparatory batch.               | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>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.               |

**Table – 9. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)**

| QC Check   | Minimum Frequency  | Acceptance Criteria   | Corrective Action  | Flagging Criteria  | Comments   |
|--|--|---|--|--|--|
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch.   | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br>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.   |
| <b>Dilution Test</b>   | 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. |
| <b>Post Digestion Spike (PDS) Addition</b>                   | 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.   |
| <b>Method of Standard Additions (MSA)</b>                    | When dilution or post digestion spike fails and if the required by project.                              | NA.   | NA.  | NA.  | Document use of MSA in the case narrative.   |

**Table – 10. Inorganic Analysis by Colorimetric Hexavalent Chromium**

| <b>QC Check</b>                                  | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>                             | <b>Corrective Action</b>   | <b>Flagging Criteria</b>   | <b>Comments</b>  |
|--|---|--|--|--|--|
| <b>Initial Calibration (ICAL)</b>                | 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.<br>No samples shall be analyzed until ICAL has passed.                            |
| <b>Initial Calibration Verification (ICV)</b>    | Once after each ICAL, analysis of a second source standard prior to sample analysis.                | All reported analytes within $\pm 10\%$ 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.   |
| <b>Continuing Calibration Verification (CCV)</b> | Daily before sample analysis, after every 15 field samples and at the end of the analysis sequence. | All reported analytes within $\pm 10\%$ of true value. | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br>or<br>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.<br>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. |

**Table – 10. Inorganic Analysis by Colorimetric Hexavalent Chromium**

| <b>QC Check</b>                        | <b>Minimum Frequency</b>    | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>   | <b>Flagging Criteria</b>  | <b>Comments</b>   |
|--|-----------------------------|--|--|---|---|
| <b>Method Blank (MB)</b>               | 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.<br><br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.  |
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch.  | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.<br><br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.   |
| <b>Matrix Spike (MS)</b>               | Once per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.<br><br>Verification check ensures lack of reducing conditions or interference from matrix. |

**Table – 10. Inorganic Analysis by Colorimetric Hexavalent Chromium**

| QC Check   | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments   |
|--|--|--|--|--|--|
| <b>Matrix spike Duplicate (MSD) or Matrix Duplicate (MD)</b>                         | <p><u>Aqueous matrix:</u> One per every 10 project samples.</p> <p><u>Solid matrix:</u> One per preparatory batch.</p> | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).</p> | <p>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.</p> | <p>Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the case narrative.</p> | <p>The data shall be evaluated to determine the source of difference. Results may not be reported without a valid pair.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> |
| <b>Soluble and Insoluble Pre-Digestion Matrix Spikes (solid matrix samples only)</b> | <p>One soluble and insoluble pre-digestion MS analyzed per preparatory batch prior to analysis.</p>                    | <p>MS recoveries within 75 – 125%.</p>   | <p>Correct problem and re-homogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results.</p>  | <p>Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the case narrative.</p> |  |
| <b>Post-digestion Matrix Spike (solid matrix samples)</b>                            | <p>One per preparatory batch.</p>  | <p>Recovery within 85 - 115%.</p>  | <p>No specific corrective action, unless required by the project.</p>  | <p>Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the case narrative.</p> | <p>Criteria apply for samples with concentrations &gt; 50 X LOQ prior to dilution.</p>   |

**Table – 11. Cyanide Analysis**

| <b>QC Check</b>                               | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>                    | <b>Corrective Action</b>                                   | <b>Flagging Criteria</b>     | <b>Comments</b>   |
|---|---|---|--|------------------------------|---|
| <b>Initial Calibration (ICAL)</b>             | 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.<br>No samples shall be analyzed until ICAL has passed.                           |
| <b>Distillation Verification</b>              | Once after each ICAL, with two distilled ICAL standards; prior to sample analysis.<br>Not required if all ICAL standards are distilled. | Within $\pm 10\%$ 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.<br>No samples shall be analyzed until distillation technique has been verified. |
| <b>Initial Calibration Verification (ICV)</b> | Once after each ICAL, analysis of a second source standard prior to sample analysis.  | Within $\pm 10\%$ of true value.              | Correct problem. Rerun ICV. If that fails, repeat ICAL.    | Flagging is not appropriate. | No samples shall be analyzed until calibration has been verified.   |

**Table – 11. Cyanide Analysis**

| QC Check   | Minimum Frequency   | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments  |
|--|---|--|--|--|---|
| <b>Continuing Calibration Verification (CCV)</b> | After every 10 field samples and at the end of the analysis sequence. | Within $\pm 10\%$ of true value.   | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br>or<br>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.<br><br>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.          |
| <b>Method Blank (MB)</b>                         | 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.<br><br>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. |

**Table – 11. Cyanide Analysis**

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action   | Flagging Criteria   | Comments  |
|---|--|--|---|---|---|
| <b>Initial and Continuing Calibration Blank (ICB/CCB)</b> | Before beginning a sample run;<br>after every 10 field samples;<br>and at end of the analysis sequence.<br>(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.  |
| <b>Laboratory Control Sample (LCS)</b>                    | One per preparatory batch.   | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.<br><br>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.              |
| <b>Matrix Spike (MS)</b>                                  | One per preparatory batch.   | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. | Examine the project-specific requirements.<br>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. |

**Table – 11. Cyanide Analysis**

| QC Check  | Minimum Frequency   | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments   |
|---|---|--|--|--|--|
| <b>Matrix Spike Duplicate (MSD) and Matrix Duplicate (MD)</b> | One per preparatory batch.  | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>MSD or MD: RPD of all analytes <math>\leq</math> 20% (between MS and MSD or sample and MD).</p> | 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. |
| <b>Method of Standard Additions (MSA)</b>                     | When dilution or post digestion spike fails and if the required by project. | NA.  | NA.  | NA.  | Document use of MSA in the case narrative.                         |

**Table – 12. Common Anions Analysis by IC**

| <b>QC Check</b>                                     | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>                                | <b>Flagging Criteria</b>               | <b>Comments</b>   |
|---|--|--|---|--|---|
| <b>Initial Calibration (ICAL) for all analytes</b>  | 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.<br>No samples shall be analyzed until ICAL has passed. |
| <b>Retention Time window position establishment</b> | 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.   |
| <b>Retention Time (RT) window width</b>             | At method set-up and after major maintenance (e.g., column change).                  | RT width is $\pm 3$ times standard deviation for each analyte RT over a 24-hour period.  | NA.   | NA.                                    | Calculated for each analyte.  |
| <b>Initial Calibration Verification (ICV)</b>       | Once after each ICAL, analysis of a second source standard prior to sample analysis. | All reported analytes within established RT windows.<br>All reported analytes within $\pm 10\%$ of true value.   | Correct problem. Rerun ICV. If that fails, repeat ICAL. | Flagging criteria are not appropriate. | Freshly prepared ICV.<br>No samples shall be analyzed until calibration has been verified.          |

**Table – 12. Common Anions Analysis by IC**

| QC Check   | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments   |
|--|--|--|--|--|--|
| <b>Continuing Calibration Verification (CCV)</b> | Before sample analysis; after every 10 field samples; and at the end of the analysis sequence. | All reported analytes within established retention time windows.<br><br>All reported analytes within $\pm 10\%$ of true value. | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br><br>or<br>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.<br><br>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.<br><br>Retention time windows are updated per the method. |
| <b>Method Blank (MB)</b>                         | 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.<br><br>Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.                | Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  |

**Table – 12. Common Anions Analysis by IC**

| QC Check   | Minimum Frequency          | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments  |
|--|----------------------------|--|--|---|---|
| <b>Laboratory Control Sample (LCS)</b>                       | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.   | Correct problem, then re-<br>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.<br><br>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | Must contain all reported analytes.<br><br>Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.                         |
| <b>Matrix Spike (MS)</b>                                     | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>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.<br><br>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.) |
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>MSD or MD: RPD of all analytes $\leq$ 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.<br><br>The data shall be evaluated to determine the source of difference.   |

**Table – 13. Perchlorate by Mass Spectrometry Methods**

| QC Check                            | Minimum Frequency   | Acceptance Criteria  | Corrective Action   | Flagging Criteria                      | Comments  |
|-------------------------------------|---|--|---|--|---|
| <b>Interference Threshold Study</b> | 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).  | <p>Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate.</p> <p>The threshold is the concentration of the common suppressors where perchlorate recovery falls outside an 80-120% window.</p>  | NA.   | Flagging criteria are not appropriate. | This study and site history will determine the concentration at which the ICS suppressors should be set.  |
| <b>Mass Calibration</b>             | <p>Instrument must have a valid mass calibration prior to any sample analysis.</p> <p>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).</p> | <p>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.</p> <p>Mass calibration must be verified by acquiring a full scan continuum mass spectrum of a perchlorate stock standard.</p> | If the mass calibration fails, recalibrate. If it still fails, consult manufacturer instructions on corrective maintenance. | Flagging criteria are not appropriate. | <p>Problem must be corrected. No samples may be analyzed under a failing mass calibration.</p> <p>Perchlorate ions should be within <math>\pm 0.3</math> <i>m/z</i> of mass 99, 101, and 107 or their respective daughter ion masses (83, 85, and 89), depending on which ions are quantitated.</p> |

**Table – 13. Perchlorate by Mass Spectrometry Methods**

| <b>QC Check</b>                               | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>   | <b>Flagging Criteria</b>               | <b>Comments</b>  |
|---|--|--|--|--|--|
| <b>Tune Check</b>                             | 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.   |
| <b>Initial Calibration (ICAL)</b>             | At instrument setup or after ICV or CCV failure, prior to sample analysis. | ICAL must meet one of the two options below:<br><br>Option 1: RSD for each analyte $\leq 15\%$ ;<br><br>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.<br><br>No samples shall be analyzed until ICAL has passed.   |
| <b>Initial Calibration Verification (ICV)</b> | Once after each ICAL.  | Perchlorate concentration must be within $\pm 15\%$ 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.<br><br>No samples shall be analyzed until calibration has been verified with a second source. |

**Table – 13. Perchlorate by Mass Spectrometry Methods**

| QC Check  | Minimum Frequency   | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments  |
|---|---|--|---|--|---|
| <b>Continuing Calibration Verification (CCV)</b>                | <p>On days an ICAL is performed, after every 10 field samples and at the end of the analytical sequence.</p> <p>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.</p> | <p>Perchlorate concentration must be within <math>\pm 15\%</math> of its true value.</p>   | <p>Recalibrate, and reanalyze all affected samples since the last acceptable CCV;</p> <p>or</p> <p>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.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.</p>  | <p>Results may not be reported without a valid CCV.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>  |
| <b>Isotope Ratio <math>^{35}\text{Cl}/^{37}\text{Cl}</math></b> | <p>Every sample, batch QC sample, and standard.</p>   | <p>Monitor for either the parent ion at masses 99/101 or the daughter ion at masses 83/85 depending on which ions are quantitated.</p> <p>Must fall within 2.3 to 3.8.</p> | <p>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.</p> <p>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.</p>               | <p>If reanalysis after cleanup fails to meet acceptance criteria, data must be qualified with a Q-flag and explained in the case narrative.</p> <p>The disposition of results of alternate techniques used to confirm presence of perchlorate must be discussed in the case narrative.</p> | <p>Decision to report data failing ratio check should be thoroughly documented in case narrative.</p> <p>The use of cleanup procedures, post spike samples, and dilutions must be identified in the case narrative.</p> |

**Table – 13. Perchlorate by Mass Spectrometry Methods**

| QC Check                               | Minimum Frequency  | Acceptance Criteria  | Corrective Action   | Flagging Criteria   | Comments   |
|--|--|--|---|---|--|
| <b>Internal Standard (IS)</b>          | Addition of <sup>18</sup> O-labeled perchlorate to every sample, batch QC sample, standard, instrument blank, and method blank.  | Measured <sup>18</sup> O IS area within ± 50% of the value from the average of the IS area counts of the ICAL.<br><br>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.   |
| <b>Interference Check Sample (ICS)</b> | 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).<br><br>At least one ICS must be analyzed daily.<br><br>The ICS shall be prepared at the LOQ. | Perchlorate concentration must be within ± 20% of its true value.  | Correct problem.<br>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.<br><br>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.<br><br>No samples may be reported that are associated with a failing ICS. |

**Table – 13. Perchlorate by Mass Spectrometry Methods**

| QC Check                              | Minimum Frequency   | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments   |
|---------------------------------------|---|--|--|---|--|
| <b>Laboratory Reagent Blank (LRB)</b> | 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.<br><br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.<br><br>Additional LRBs may be needed to ensure that there was no carryover from over range samples. |
| <b>Method Blank (MB)</b>              | 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.<br><br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.   |

**Table – 13. Perchlorate by Mass Spectrometry Methods**

| QC Check                               | Minimum Frequency                     | Acceptance Criteria   | Corrective Action   | Flagging Criteria  | Comments   |
|--|---------------------------------------|---|---|--|--|
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch.            | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> | <p>Correct problem. Reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.</p> | <p>LCS must be spiked at the LOQ.</p> <p>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.</p> <p>LCS must undergo the same preparation and pretreatment steps as the samples in the batch.</p>                     |
| <b>Matrix Spike (MS)</b>               | One per preparatory batch per matrix. | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> | <p>Examine the project specific requirements. Contact the client as to additional measures to be taken.</p>   | <p>For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.</p>  | <p>The MS must be spiked at the LOQ.</p> <p>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.</p> <p>MS must undergo the same preparation and pretreatment steps as the samples in the batch.</p> |

**Table – 13. Perchlorate by Mass Spectrometry Methods**

| QC Check   | Minimum Frequency                     | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments   |
|--|---------------------------------------|--|---|--|--|
| <b>Matrix Spike Duplicate (MSD) or Laboratory Duplicate (LD)</b> | One per preparatory batch per matrix. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>MSD or MD: RPD of all analytes $\leq$ 15% (between MS and MSD or sample and MD). | Examine the project specific requirements.<br>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.<br><br>The data shall be evaluated to determine the source of difference. |

**Table – 14. Chemical Warfare Agents by GC/MS**

| QC Check  | Minimum Frequency  | Acceptance Criteria   | Corrective Action                                       | Flagging Criteria            | Comments   |
|---|--|---|---|------------------------------|--|
| <b>Tune Check</b>   | 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.   |
| <b>Initial Calibration (ICAL) for all analytes and surrogates</b> | At instrument set-up and after ICV or CCV failure, prior to sample analysis.         | Each analyte must meet one of the three options below:<br><u>Option 1:</u> RSD for each analyte $\leq 15\%$ ;<br><u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$ ;<br><u>Option 3:</u> 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.<br>No samples shall be analyzed until ICAL has passed.<br>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. |
| <b>Initial Calibration Verification (ICV)</b>                     | Once after each ICAL, analysis of a second source standard prior to sample analysis. | All reported analytes and surrogates within $\pm 25\%$ 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.   |
| <b>Retention Time window position establishment</b>               | 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.   |

**Table – 14. Chemical Warfare Agents by GC/MS**

| QC Check   | Minimum Frequency   | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments  |
|--|---|--|--|--|---|
| <b>Retention Time (RT) window width</b>          | At method set-up and after major maintenance (e.g., column change).                     | RT width is $\pm 3$ times standard deviation for each analyte RT from the 72-hour study.   | NA.  | NA.  | Calculated for each analyte and surrogate.  |
| <b>Continuing Calibration Verification (CCV)</b> | Before sample analysis, after every 10 field samples, and at the end of the prep batch. | All reported analytes within established RT windows.<br><br>All reported analytes and surrogates within $\pm 25\%$ of true value.<br><br>All reported analytes and surrogates within $\pm 50\%$ for end of prep batch CCV. | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br><br>or<br><br>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.<br><br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |
| <b>Internal Standards (IS)</b>                   | Every field sample, Standard and QC sample.   | Retention time within $\pm 10$ 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.<br><br>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.<br><br>Flagging is not appropriate for failed standards. |   |

**Table – 14. Chemical Warfare Agents by GC/MS**

| QC Check                               | Minimum Frequency          | Acceptance Criteria   | Corrective Action   | Flagging Criteria   | Comments  |
|--|----------------------------|---|---|---|---|
| <b>Method Blank (MB)</b>               | 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.<br><br>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.<br>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.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.  |
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>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.<br>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.<br><br>Results may not be reported without a valid LCS.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

**Table – 14. Chemical Warfare Agents by GC/MS**

| QC Check   | Minimum Frequency          | Acceptance Criteria   | Corrective Action  | Flagging Criteria  | Comments  |
|--|----------------------------|---|--|--|---|
| <b>Matrix Spike (MS)</b>                                     | One per preparatory batch. | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>Limits may be set at 50-150% until sufficient data has been generated to establish in-house control limits.</p>  | 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. | <p>Must contain all surrogates and all analytes to be reported.</p> <p>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.</p> |
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch. | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>Limits may be set at 50-150% until sufficient data has been generated to establish in-house control limits.</p> <p>MSD or MD: RPD of all analytes <math>\leq</math> 20% (between MS and MSD or sample and MD).</p> | 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. | <p>MSD: Must contain all surrogates and all analytes to be reported.</p> <p>The data shall be evaluated to determine the source of difference.</p>  |

**Table – 14. Chemical Warfare Agents by GC/MS**

| QC Check               | Minimum Frequency         | Acceptance Criteria   | Corrective Action   | Flagging Criteria   | Comments   |
|------------------------|---------------------------|---|---|---|--|
| <b>Surrogate Spike</b> | All field and QC samples. | QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits<br><br>or in-house LCS limits if analyte(s) are not listed.<br><br>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. |

**Table - 15. Perfluorinated Compounds by Liquid Chromatography/Mass Spectrometry**

| <b>QC Check</b>   | <b>Minimum Frequency</b>   | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>   | <b>Flagging Criteria</b>   | <b>Comments</b>   |
|---|--|--|--|--|---|
| <b>Initial Calibration (ICAL) for all analytes and surrogates</b> | 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.<br><br>Calibration can be linear (5 standards) or quadratic (6 standards); weighting is allowed. |
| <b>Initial Calibration Verification (ICV)</b>                     | Once after each ICAL, analysis of a second source standard prior to sample analysis.   | All reported analytes and surrogates within $\pm 25\%$ 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.  |
| <b>Continuing Calibration Verification (CCV)</b>                  | 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 $\pm 25\%$ of true value.  | Recalibrate, and reanalyze all affected samples since the last acceptable CCV;<br>or<br>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.<br><br>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.   |

**Table - 15. Perfluorinated Compounds by Liquid Chromatography/Mass Spectrometry**

| <b>QC Check</b>               | <b>Minimum Frequency</b>  | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>  | <b>Flagging Criteria</b>  | <b>Comments</b>   |
|-------------------------------|---|--|---|---|---|
| <b>Internal Standard (IS)</b> | 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.<br><br>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.  |
| <b>Tune Check</b>             | Prior to ICAL and after any mass calibration or maintenance is performed.   | Tuning standard must contain analytes of interest or appropriate substitute.<br><br>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.   |
| <b>Method Blank (MB)</b>      | 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.<br><br>Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. | Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

**Table - 15. Perfluorinated Compounds by Liquid Chromatography/Mass Spectrometry**

| <b>QC Check</b>                        | <b>Minimum Frequency</b>              | <b>Acceptance Criteria</b>   | <b>Corrective Action</b>   | <b>Flagging Criteria</b>  | <b>Comments</b>  |
|--|---------------------------------------|--|--|---|--|
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch.            | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>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.<br><br>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.                       |
| <b>Matrix Spike (MS)</b>               | One per preparatory batch per matrix. | A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.<br><br>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.<br><br>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. |

**Table - 15. Perfluorinated Compounds by Liquid Chromatography/Mass Spectrometry**

| QC Check   | Minimum Frequency                     | Acceptance Criteria   | Corrective Action   | Flagging Criteria  | Comments   |
|--|---------------------------------------|---|---|--|--|
| <b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b> | One per preparatory batch per matrix. | <p>A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>If in-house LCS limits do not exist, use 70-130% until limits are established.</p> <p>MSD or MD: RPD of all analytes <math>\leq</math> 30% (between MS and MSD or sample and MD)</p> | 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. | <p>The data shall be evaluated to determine the source of error.</p> <p>Analyze MS/MSD for low concentration samples and Sample/MD for high concentration samples.</p> |
| <b>Surrogate Spike</b>                                       | All field and QC samples.             | <p>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.</p> <p>Limits may be set at 70-130% until sufficient data has been generated to establish in-house control limits.</p>   | <p>If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect.</p> <p>For failed QC samples, correct problem, and rerun all failed samples.</p> | Apply Q-flag and discuss in the case narrative.  | Alternative surrogates are recommended when there is obvious chromatographic interference.   |

**Table – 16. Alpha Spectrometry**

| QC Check  | Minimum Frequency  | Acceptance Criteria   | Corrective Action                         | Flagging Criteria                             | Comments  |
|---|--|---|---|---|---|
| <p><b>Initial Calibration (ICAL)</b><br/><b>(Energy, efficiency and FWHM peak resolution)</b></p> | <p>Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.3)</p> | <p>Verify manufacturer's specifications for point source efficiency (MARLAP);<br/>and<br/>Two calibration peaks that are: 1) <math>\geq 700</math> keV apart; or 2) that bracket all peaks to be determined.<br/><br/>Energy vs. channel slope equation <math>&lt; 15</math> keV per channel.<br/><br/>Full Width –Half Maximum (FWHM) <math>\leq 100</math> keV for each peak used for calibration.<br/><br/>Minimum of 3,000 net counts in each peak.</p> | <p>Correct problem, then repeat ICAL.</p> | <p>Flagging criteria are not appropriate.</p> | <p>Use traceable calibration source (CS) that matches sample test source (STS) configuration (type, size and position relative to the detector).<br/><br/>May use same count for initial efficiency calibration.<br/><br/>No samples may be run until energy and FWHM calibration criteria are met.</p> |

**Table – 16. Alpha Spectrometry**

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action   | Flagging Criteria                      | Comments  |
|---|--|--|---|--|---|
| <b>Initial Calibration Verification (ICV)</b>                   | 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).<br>or<br>Observed peak centroid falls within $\pm 20$ keV from reference energy for each peak used in the initial energy calibration.<br><br>FWHM $\leq 100$ keV and within $\pm 20$ keV of corresponding calibration peaks in initial energy calibration. | Repeat ICV to check for error.<br><br>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.<br><br>Bracketing peaks may also be used that are $>1000$ keV apart.<br><br>No samples may be run until calibration has been verified with a second source. |
| <b>Continuing Calibration Verification (CCV) (Pulsar check)</b> | Pulsar energy verification weekly, prior to analysis of samples.<br><br>Use either Pulsar check or Check source. | Energy response check shall have a tolerance limit set at $\pm 3\%$ or control chart set at $\pm 3\sigma$ (MARLAP 18.5.6.3).<br>or<br>Observed peak centroid falls $\leq 20$ keV from reference energy.  | Recount and check control chart for trends.<br><br>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.<br><br>No samples may be run until calibration has been verified.  |

**Table – 16. Alpha Spectrometry**

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments  |
|---|--|--|--|---|---|
| <b>Continuing Calibration Verification (CCV) (Check source)</b> | Weekly source check verification prior to analysis of samples.<br>Use either Pulsar check or Check source. | Response checks shall have a tolerance limit or control chart set at $\pm 3\%$ or $3\sigma$ . (MARLAP 18.5.6.3)<br>or<br>Observed peak centroid falls within 20 keV from reference energy for each peak used in the initial energy calibration.<br>FWHM $\leq 100$ keV and within 30 keV of corresponding calibration peaks in initial energy calibration. | Recount and check control chart for trends.<br>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.<br>No samples may be run until calibration has been verified.           |
| <b>Background Subtraction Count (BSC) Measurement</b>           | Prior to initial use or after initial calibration and monthly. (MARLAP 18.5.6.3)                           | Within $\pm 3\sigma$ 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.<br>Determine cause, correct problem, re-establish BSC.<br>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).<br>Activity must meet project objectives. |

**Table – 16. Alpha Spectrometry**

| QC Check                                    | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments   |
|---|--|--|--|---|--|
| <b>Instrument Contamination Check (ICC)</b> | Performed weekly, at minimum, and after counting high activity samples.<br><br>Count duration ≥ longest STS count. | $Z_{\text{Blank}} \leq 3$ for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1)   | Check control chart for trends and recount. Determine cause and correct problem.<br><br>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.<br><br>Explain in the case narrative.      |  |
| <b>Method Blank (MB)</b>                    | One per preparatory batch. (MARLAP 18.4.1)   | $ Z_{\text{Blank}}  \leq 3$ . Investigate recurrent results with $ Z_{\text{Blank}}  \geq 2$ . (MARLAP 18.4.1)<br><br>or<br><br>In-house control limits of $\pm 3 \sigma$ of the mean. | Recount the blank to confirm results. Inspect MB control chart for indication of significant bias<br><br>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.<br><br>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.<br><br>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.<br><br>With project approval and appropriate qualification and narration, report results with a count rate >5 times that of the affected ROI in the MB. |

**Table – 16. Alpha Spectrometry**

| QC Check                               | Minimum Frequency  | Acceptance Criteria  | Corrective Action   | Flagging Criteria   | Comments  |
|--|--|--|---|---|---|
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch.   | $ Z_{LCS}  \leq 3$ . Investigate recurrent results with $ Z_{LCS}  \geq 2$ . (MARLAP 18.4.3)<br>or<br>Use in-house control limits of $LCS \pm 3\sigma$ 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.<br><br>Reprep and reanalyze the LCS and all associated samples. | If reanalysis cannot be performed, data must be qualified and explained in the case narrative.<br><br>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.<br><br>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.<br><br>Acceptance criteria for LCS recovery may be specified by the project. |
| <b>Matrix Spike (MS)</b>               | One per preparatory batch.<br><br><i>(MS not required when chemical yield tracers or carriers are employed).</i> | If activity of the MS > 5 times the unspiked sample, then $ Z_{MS}  \leq 3$ . (MARLAP 18.4.3)<br>or<br>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.  |

**Table – 16. Alpha Spectrometry**

| QC Check                  | Minimum Frequency                               | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments  |
|---------------------------|---|--|---|--|---|
| <b>Sample Duplicate</b>   | One per preparatory batch per matrix.           | $ Z_{Dup}  \leq 3$ . Investigate recurrent results with $ Z_{Dup}  \geq 2$ . (MARLAP 18.4.1)<br>or<br>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.<br>Examine the project-specific requirements.<br>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.  |
| <b>Tracers (if used)</b>  | Added to each sample as isotopic yield monitor. | Isotopic yield within 30-110%.<br>FWHM $<100$ keV and peak energy within $\pm 40$ 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.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |
| <b>Carriers (if used)</b> | 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.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

**Table -17. Gamma Spectrometry**

| QC Check   | Minimum Frequency  | Acceptance Criteria   | Corrective Action                         | Flagging Criteria                             | Comments   |
|--|--|---|---|---|--|
| <p><b>Initial Calibration (ICAL)</b><br/> <b>(Energy, efficiency and FWHM peak resolution)</b></p> | <p>Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.2)</p> | <p>Verify manufacturer's specifications for gamma peak resolution. (MARLAP 18.5.6.2)</p> <p>Efficiency vs. energy for each geometry/matrix. 95% confidence limit of the fitted function: <math>\leq 8\%</math> over energy range. (MARLAP 18.5.6.2)</p> <p>or</p> <p>Peak energy difference is within 0.1 keV of reference energy for all points.</p> <p>Peak Full Width at Half Maximum (FWHM) &lt; 2.5 keV at 1332 keV.</p> <p>Energy vs. channel slope equation shall be linear and accurate to 0.5 keV.</p> | <p>Correct problem, then repeat ICAL.</p> | <p>Flagging criteria are not appropriate.</p> | <p>Traceable calibration source (CS) matches sample test source (STS) configuration (type, size, geometry and position relative to the detector).</p> <p>Minimum of 10,000 net counts in each peak in at least six calibration peaks that bracket the range of use.</p> <p>No samples may be run until all calibration criteria are met.</p> |

**Table -17. Gamma Spectrometry**

| QC Check                                      | Minimum Frequency  | Acceptance Criteria  | Corrective Action   | Flagging Criteria                      | Comments  |
|---|--|--|---|--|---|
| <b>Initial Calibration Verification (ICV)</b> | After ICAL for energy/efficiency and prior to analysis of samples. | Observed peaks of second source standard fall within $\pm 10\%$ of initial calibration value relative to energy, FWHM, and efficiency. | Verify second source standard and repeat ICV to check for errors.<br>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).<br>Minimum of 10,000 net counts in each peak in at least six calibration verification peaks that bracket the range of use.<br>No samples may be run until calibration has been verified. |

**Table -17. Gamma Spectrometry**

| QC Check  | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments   |
|---|--|--|--|---|--|
| <p><b>Continuing Calibration Verification (CCV) (Daily Check)</b></p> | <p>Daily or prior to use.<br/>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.</p> | <p>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 <math>\pm 3\%</math> or <math>3\sigma</math> of the mean. (MARLAP 18.5.6.2)<br/>or<br/><u>Peak Energy/Efficiency:</u> low, mid, and high energies within 10% of the initial calibration value;<br/><u>FWHM:</u> low, mid, and high energies within 10% of initial FWHM value.</p> | <p>Correct problem, rerun CCV. If that fails, then repeat ICAL.<br/><br/>Reanalyze all samples since the last successful calibration verification.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.<br/><br/>Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.</p> | <p>Results may not be reported without a valid CCV.<br/><br/>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> |

**Table -17. Gamma Spectrometry**

| QC Check   | Minimum Frequency   | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments   |
|--|---|--|---|--|--|
| <p><b>Background Subtraction Count Measurement (BSC)</b><br/>(Long count for subtracting background from blanks or test sources)</p> | <p>Immediately after ICAL and then performed on at least a monthly basis.<br/>(MARLAP 18.5.6.2)</p>   | <p>Statistical test of successive counts and count rates for identified background peaks show no significant difference.<br/>(MARLAP 18.5.6.2)</p>   | <p>Recount and check control chart for trends. Determine cause, correct problem, re-establish BSC.<br/><br/>If background activity has changed, re-establish BSC and reanalyze or qualify all impacted samples since last acceptable BSC.</p> | <p>Apply B-flag to all results for specific nuclide(s) in all samples associated with the blank.</p>                           | <p>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.<br/><br/>The counting interval for the long count shall be between one and four times the nominal counting interval of the test sources.<br/><br/>(MARLAP 18.5.6.2)</p> |
| <p><b>Instrument Contamination Check (ICC)</b><br/>(Short count for controlling gross contamination)</p>                             | <p>Daily or when working with long count times before and after each analytical batch.<br/><br/>Check after counting high activity samples.</p> | <p>No extraneous peaks identified (i.e., no new peaks in the short background spectrum compared to previous spectra); The tolerance limit or control chart: <math>\pm 3\%</math> or <math>3\sigma</math> of the mean activity.<br/>(MARLAP 18.5.6.2)</p> | <p>Recount the background. If still out of control, locate and correct problem; reanalyze or qualify all impacted samples since last acceptable ICC.<br/><br/>If background activity has changed, re-establish BSC and reanalyze samples.</p> | <p>If corrective action fails, apply Q-flag to all results for specific nuclide(s) in all samples associated with the BSC.</p> | <p>Integrate spectrum from ~50 - 2,000 keV to check for gross contamination.<br/>(MARLAP 18.5.6.2)</p>   |

**Table -17. Gamma Spectrometry**

| QC Check                 | Minimum Frequency          | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments   |
|--------------------------|----------------------------|--|---|--|--|
| <b>Method Blank (MB)</b> | One per preparatory batch. | $ Z_{\text{Blank}}  \leq 3$ for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1)<br>or<br>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.<br>Inspect MB control chart for indication of significant bias.<br>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)<br>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.)<br>Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

**Table -17. Gamma Spectrometry**

| QC Check                               | Minimum Frequency                     | Acceptance Criteria   | Corrective Action  | Flagging Criteria   | Comments   |
|--|---------------------------------------|---|--|---|--|
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch.            | $ Z_{LCS}  \leq 3$ . Investigate recurrent results with $ Z_{LCS}  \geq 2$ . (MARLAP 18.4.3)<br>or<br>Use in-house control chart limits of $\pm 3\sigma$ of the mean. In-house control limits may not fall more than 25% from the known LCS value.<br>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.<br>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.<br>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.<br>LCS must be counted for a sufficient time to meet the required project minimum activity.<br>Results may not be reported without a valid LCS. Qualification is only appropriate in cases where the samples cannot be reanalyzed. |
| <b>Sample Duplicate</b>                | One per preparatory batch per matrix. | $ Z_{Dup}  \leq 3$ . Investigate recurrent results with $ Z_{Dup}  \geq 2$ . (MARLAP 18.4.1)<br>or<br>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.<br>Examine the project-specific requirements.<br>Contact the client as to additional measures to be taken.  | For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.  | The data shall be evaluated to determine the source of difference.   |

**Table – 18. Gas Flow Proportional Counting**

| QC Check   | Minimum Frequency   | Acceptance Criteria   | Corrective Action                          | Flagging Criteria                             | Comments  |
|--|---|---|--|---|---|
| <p><b>Initial Calibration - Voltage Plateau (ICALV)</b><br/>(separate plateaus determined for alpha and beta activity)</p> | <p>Prior to initial use and after loss of control. (MARLAP 18.5.6.1)</p>  | <p>Verify manufacturer's specifications. (MARLAP 18.5.6.1)</p> <p>Plot voltage vs. count rate to determine proper operating voltages. (MARLAP 18.5.6.1)</p> <p>or</p> <p>Slope of the plateau less than 5% over a range of 100V.</p>  | <p>Correct problem, then repeat ICALV.</p> | <p>Flagging criteria are not appropriate.</p> | <p>Series of 1-minute counts in &lt;50V steps from ~300V to ~1500V.</p> <p>No samples may be run until plateau calibration criteria are met.</p>  |
| <p><b>Initial Calibration - Efficiency (ICALE)</b></p>   | <p>Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1)</p> | <p>Verify manufacturer's specifications for detector efficiency for both alpha and beta counting modes using electroplated sources. (MARLAP 18.5.6.1)</p> <p>A 1<math>\sigma</math> counting uncertainty of <math>\leq 1\%</math> shall be achieved for all detector efficiency determinations. (MARLAP 18.5.6.1)</p> | <p>Correct problem, then repeat ICALE.</p> | <p>Flagging criteria are not appropriate.</p> | <p>Detector's counting efficiency, using traceable calibration sources, shall be determined for each radionuclide used to analyze test sources.</p> <p>No samples may be run until efficiency calibration criteria are met.</p> |

**Table – 18. Gas Flow Proportional Counting**

| QC Check   | Minimum Frequency  | Acceptance Criteria   | Corrective Action                    | Flagging Criteria                      | Comments   |
|--|--|---|--------------------------------------|--|--|
| <b>Initial Calibration –<br/><u>Cross-talk Factors</u><br/>(ICALCT)</b>    | 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.<br><br>For mass loaded test sources, determine crosstalk factors for the nuclide as a function of test source mass.<br><br>No samples may be run until cross talk calibration criteria are met. |
| <b>Initial Calibration –<br/><u>Self-Absorption Curve</u><br/>(ICALSA)</b> | 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 $\leq 10\%$ and $\leq 5\%$ uncertainty for alpha and beta, respectively. (MARLAP 18.5.6.1)<br><br>or<br><br>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.<br><br>No samples may be run until mass attenuation calibration criteria are met.  |

**Table – 18. Gas Flow Proportional Counting**

| QC Check  | Minimum Frequency   | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments  |
|---|---|--|---|--|---|
| <b>Efficiency Calibration Verification (IECV)</b> | 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 $\pm 3\%$ or $3\sigma$ of the mean. (MARLAP 18.5.6.1)<br>or<br>Value of second source calibration for each isotope within $\pm 10\%$ of initial calibration value. | Correct problem and verify second source standard. Rerun IECV.<br><br>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).<br><br>No samples may be run until calibration has been verified. |
| <b>Continuing Calibration Verification (CCV)</b>  | After a counting gas change and daily for short test-source counting intervals.<br><br>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 $\pm 3\%$ or $3\sigma$ of the mean.   | Correct problem, rerun calibration verification.<br><br>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.<br><br>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.<br><br>Results may not be reported without a valid CCV.<br><br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.           |

**Table – 18. Gas Flow Proportional Counting**

| QC Check  | Minimum Frequency   | Acceptance Criteria   | Corrective Action   | Flagging Criteria   | Comments   |
|---|---|---|---|---|--|
| <b>Method Blank (MB)</b>  | One per preparatory batch.  | Z <sub>Blank</sub>   ≤ 3 for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1)<br>or<br>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.<br>Inspect MB control chart for indication of significant bias.<br>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.<br>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.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed.<br>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). |
| <b>Background Subtraction Count (BSC) Measurement (Long count for subtracting background from blanks or test sources)</b> | Performed at least on a monthly basis.<br>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)<br>or<br>Within ±3σ of mean activity of recent BSCs (minimum of 3 BSCs).   | Check control chart for trend and recount.<br>Determine cause and correct problem.<br>If background activity has changed, re-establish BSC.<br>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.<br>Activity must meet project requirements.  |

**Table – 18. Gas Flow Proportional Counting**

| QC Check  | Minimum Frequency   | Acceptance Criteria   | Corrective Action  | Flagging Criteria   | Comments   |
|---|---|---|--|---|--|
| <b>Instrument Contamination Check (ICC)</b><br><b>(Short count for controlling gross contamination)</b> | Daily or when working with long count times, before and after each analytical batch.<br>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)<br>or<br>Within $\pm 3\sigma$ 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.<br>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.   |
| <b>Laboratory Control Sample (LCS)</b>  | One per preparatory batch.  | $ Z_{LCS}  \leq 3$ . Investigate recurrent results with $ Z_{LCS}  \geq 2$ . (MARLAP 18.4.3)<br>or<br>Use in-house control chart limits of $\pm 3\sigma$ of the mean. In-house control limits may not fall more than 25% from the known LCS value.<br>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.<br>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.<br>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.<br>LCS must be counted for a sufficient time to meet the required project minimum activity.<br>Results may not be reported without a valid LCS.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

**Table – 18. Gas Flow Proportional Counting**

| QC Check                 | Minimum Frequency  | Acceptance Criteria   | Corrective Action   | Flagging Criteria  | Comments   |
|--------------------------|--|---|---|--|--|
| <b>Matrix Spike (MS)</b> | One per preparatory batch.<br><i>(MS not required when yield tracers are employed)</i> | If activity of the MS > 5 times the unspiked sample, $ Z_{MS}  \leq 3$ . (MARLAP 18.4.3)<br>or<br>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.   |
| <b>Sample Duplicate</b>  | One per preparatory batch per matrix.  | $ Z_{Dup}  \leq 3$ . Investigate recurrent results with $ Z_{Dup}  \geq 2$ . (MARLAP 18.4.1)<br>or<br>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.   |
| <b>Tracers (if used)</b> | 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. |

**Table – 18. Gas Flow Proportional Counting**

| QC Check                  | Minimum Frequency                               | Acceptance Criteria            | Corrective Action                                   | Flagging Criteria  | Comments   |
|---------------------------|---|--------------------------------|---|--|--|
| <b>Carriers (if used)</b> | Added to each sample as chemical yield monitor. | Chemical yield within 30-110%. | Reanalysis of sample, including sample preparation. | For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met. | The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

**Table - 19. Liquid Scintillation Counter Analysis**

| QC Check  | Minimum Frequency   | Acceptance Criteria   | Corrective Action                  | Flagging Criteria                      | Comments   |
|---|---|---|------------------------------------|--|--|
| <b>Initial Calibration (ICAL)</b><br><b>(Efficiency, ROI)</b> | 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)<br>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 <sup>3</sup> H and/or <sup>14</sup> C.<br>No samples may be run until efficiency calibration criteria are met and energy ROIs are established for radionuclides of interest. |
| <b>Method Calibration (QCAL)</b><br><b>(Quench curve)</b>     | 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.<br>Individual calibration sources shall be counted to achieve ROI measurement uncertainty of ≤1%. (MARLAP 18.5.6.4)<br>or<br>Minimum 10,000 counts for each data point.<br>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.<br>No samples may be run until calibration criteria are passed.   |

**Table - 19. Liquid Scintillation Counter Analysis**

| QC Check   | Minimum Frequency     | Acceptance Criteria   | Corrective Action  | Flagging Criteria                      | Comments   |
|--|-----------------------|---|--|--|--|
| <b>Method Calibration (QCAL)</b><br><b>(Standard Addition)</b> | Once after each ICAL. | Statistically evaluate replicate test-source analyses. (MARLAP 18.5.6.4)<br>or<br>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.<br><br>No samples may be run until calibration (Standard Addition) has been verified. |
| <b>Initial Calibration Verification (ICV)</b>                  | Once after each ICAL. | Value of each second source nuclide $\pm 10\%$ of initial calibration value.  | Correct problem and verify second source standard. Rerun ICV.<br><br>If that fails, correct problem and repeat ICAL. | Flagging criteria are not appropriate. | Use a second source standard for each nuclide.<br><br>No samples may be run until calibration has been verified.   |

**Table - 19. Liquid Scintillation Counter Analysis**

| QC Check   | Minimum Frequency  | Acceptance Criteria  | Corrective Action  | Flagging Criteria   | Comments  |
|--|--|--|--|---|---|
| <b>Continuing Calibration Verification (CCV)</b> | <p>Counting efficiency performance check performed on day-of-use basis.</p> <p>Prior to use for short counting intervals.</p> <p>Before and after a test source batch for longer counting intervals. (MARLAP 18.5.6.4)</p> <p>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.</p> | <p>Response checks should have a tolerance limit or control chart set at <math>\pm 3\%</math> or <math>3\sigma</math> of the mean. (MARLAP 18.5.6.4)</p>   | <p>Correct problem, rerun calibration verification. If that fails, then repeat ICAL.</p> <p>Reanalyze all samples since the last successful calibration verification.</p>  | <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.</p> | <p>ROI for unquenched reference standards (typically 3H and/or 14C).</p> <p>Results may not be reported without a valid CCV.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>   |
| <b>Method Blank (MB)</b>                         | <p>One per preparatory batch.</p>  | <p><math> Z_{\text{Blank}}  \leq 3</math>. Investigate recurrent results with <math> Z_{\text{Blank}}  \geq 2</math>. (MARLAP 18.4.1)</p> <p>or</p> <p>In-house control limits of <math>\pm 3 \sigma</math> of the mean.</p> <p>With project approval and appropriate qualification and narration, report results with a count rate <math>&gt;5</math> times that of the affected ROI in the MB.</p> | <p>Recount the blank to confirm results, unless all sample results are <math>&gt;5</math> times the blank activity.</p> <p>If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.</p> <p>All samples following the last acceptable background measurement must be reanalyzed.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.</p>                | <p>Results may not be reported without a valid method blank.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> <p>Blank matrices must be the same as the associated samples (i.e. radon free distilled or deionized water, or representative of the material media).</p> |

**Table - 19. Liquid Scintillation Counter Analysis**

| QC Check  | Minimum Frequency                                   | Acceptance Criteria  | Corrective Action   | Flagging Criteria  | Comments   |
|---|---|--|---|--|--|
| <b>Background Subtraction Count (BSC) Measurement (Unquenched blank; applicable when MSA is used)</b> | 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)<br>or<br>Within $\pm 3\sigma$ 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.<br>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.<br>Activity must meet project objectives. |
| <b>Method Background Measurement (MBM) (Quenched blank)</b>   | 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)<br>or<br>Within $\pm 3\sigma$ 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.<br>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).                    |

**Table - 19. Liquid Scintillation Counter Analysis**

| QC Check                               | Minimum Frequency  | Acceptance Criteria   | Corrective Action  | Flagging Criteria   | Comments   |
|--|--|---|--|---|--|
| <b>Laboratory Control Sample (LCS)</b> | One per preparatory batch.   | $ Z_{LCS}  \leq 3$ . Investigate recurrent results with $ Z_{LCS}  \geq 2$ . (MARLAP 18.4.3)<br>or<br>Use in-house control chart limits of $\pm 3\sigma$ of the mean. In-house control limits may not fall more than 25% from the known LCS value.<br>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.<br>Reprep and reanalyze method LCS and all associated samples. | If reanalysis cannot be performed, data must be qualified and explained in the case narrative.<br>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.<br>LCS must be counted for a sufficient time to meet the required project minimum activity.<br>Results may not be reported without a valid LCS.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |
| <b>Matrix Spike (MS)</b>               | One per preparatory batch.<br><i>(MS not required when yield tracers or carriers are employed)</i> | If activity of the MS > 5 times the unspiked sample, $ Z_{MS}  \leq 3$ . (MARLAP 18.4.3)<br>or<br>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.   |

**Table - 19. Liquid Scintillation Counter Analysis**

| QC Check                | Minimum Frequency                      | Acceptance Criteria  | Corrective Action  | Flagging Criteria  | Comments  |
|-------------------------|--|--|--|--|---|
| <b>Sample Duplicate</b> | One per preparatory batch per matrix.  | $ Z_{Dup}  \leq 3$ . Investigate recurrent results with $ Z_{Dup}  \geq 2$ . (MARLAP 18.4.1)<br>or<br>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.  |
| <b>Tracers</b>          | 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.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |
| <b>Carriers</b>         | 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.<br>Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

# Appendix C: Laboratory Control Sample (LCS) Control Limits and Requirements

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## 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  $\pm$  3 sample standard deviations.

## 2.0 LCS Limit Tables

| <b>Table 1. Method 1668 Solid Matrix</b> |                |                  |             |                           |                            |                            |
|--|----------------|------------------|-------------|---------------------------|----------------------------|----------------------------|
| <b>CAS ID</b>                            | <b>Analyte</b> | <b>N Records</b> | <b>Mean</b> | <b>Standard Deviation</b> | <b>Lower Control Limit</b> | <b>Upper Control Limit</b> |
| 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                        |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

|           |                   |      |      |      |    |     |
|-----------|-------------------|------|------|------|----|-----|
| 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 |

| 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<br>[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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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-Chloroaniline]            | 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                 |

**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                 |

**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-Nitrosopiperidine             | 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                 |

**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                 |

**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                 |

**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-Chloroaniline]            | 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                 |

**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                 |

**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-Nitrosopiperidine             | 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                  |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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                 |

**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<br>[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                 |

**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                 |

**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<br>[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                 |

# APPENDIX D: Non-Destructive Assay (NDA)

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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.

The development and establishment of an “initial calibration” shall address the following as applicable:

- a) SOPs shall be in place to specify steps/activities necessary to develop and determine the “initial calibration” including but not limited to, specification of traceable radioactive sources or their alternates, geometrical positioning of sources, traceable source/matrix media configurations, acquisition of NDA system response data, computational methods, analysis of response data to determine a robust calibration, calibration acceptance criteria, calibration applicability and qualifiers and calibration uncertainty.
- b) The “initial calibration” shall be performed through the use of traceable working reference materials, unless exceptions have been stipulated and documented. For mass calibrations (i.e., calibrations that use a direct measurement of the same isotopes, matrices, and containers that will subsequently be measured in unknown items), the radioactive material mass and matrix characteristics must span and bracket the range of anticipated values for the measurement items. For calibrations based on instrument response modeling, sufficient information shall be provided in the method description and calibration regimen to assure that the calibration measurements and model appropriately spans and brackets the anticipated analysis space (e.g., provide mechanisms to account for anticipated geometries, radioactive material mass, chemical composition, and matrix characteristics). For enrichment determinations using the enrichment meter technique, the initial calibration must span the range of enrichments in anticipated unknown item measurements.
- c) The measurement uncertainty associated with the application of the “initial calibration” shall be established using a sound and technically defensible technique. Methods for the estimation of total measurement uncertainty (TMU) shall be developed and documented. Where applicable, the calibration uncertainty shall include terms for mass, matrix characteristics and configurations and radioactive material properties. These methods shall consider, at a minimum, uncertainty components, the calibration uncertainty model (method of uncertainty component propagation), estimates of uncertainty introduced by differences between item characteristics and calibration modeling assumptions. For example, if the model assumes a homogeneous distribution of the isotope of interest, the uncertainty introduced if items are not homogeneous using a worst case distribution as determined through a documented engineering judgment including supporting data must be determined.
- d) The NDA measurement method capability related to each initial calibration must be defined and documented. As applicable, this capability includes waste matrix types, process equipment types, geometries, configurations, radioactive material types, matrix density range, hydrogenous material range, radioactive material mass range, radioactive material compound, and other parameters affecting instrument response. The intent of defining the capability is to delineate those source/matrix configurations where the calibration is applicable and where it is not.
- e) Where surrogate materials are used to simulate waste matrices, their configuration(s) must be nominally representative of the actual waste item population. The design of surrogate matrix configurations must be documented. Surrogate materials used to

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

- f) If NDA method manuals, national standards, or a mandated NDA calibration methods do not specify the number of traceable WRMs to span the mass/activity and radioactive material compound(s) characteristics of the waste/process component, a minimum number must be determined and technically justified. NDA organization must document this number and their denominations in a calibration SOP or other applicable document. This requirement does not necessarily apply to NDA methods that rely on modeling. However, the method used to assure that the calibration and model appropriately spans and brackets the anticipated analysis space (e.g., provide mechanisms to account for anticipated geometries, radioactive material mass, chemical composition, and matrix characteristics) as per item (b) above must be technically justified and documented. The For NDA methods that do not necessarily require calibration with source material similar in nature to the waste or process items (e.g., neutron counting), those source(s) used are still required to be traceable. However, accounting of the efficiency variation because of the composition of the actual radioactive material shall be assessed and corrected for (e.g., Californium ( $^{252}\text{Cf}$ ) fission neutron spectrum counter efficiency versus uranyl fluoride ( $\text{UO}_2\text{F}_2$ ) 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.

### 1.1.2 Calibration Confirmation

A confirmation of the “initial” NDA measurement system calibration shall be performed. In this context, confirmation means the “initial calibration” shall be assessed and determined to be correct and true by the objective collection of evidence supporting the calibration was properly established.

- a) The “calibration confirmation” process is to produce objective evidence demonstrating the applicability and correctness of the “initial calibration” relative to the waste forms and process components of interest. The recommended method is to assemble test item(s) consisting of traceable source/matrix configuration(s) nominally representative of the waste form and/or process components to be characterized. They cannot be the same configurations used to establish the “initial calibration”. They must contain a known and traceable radioactive element/isotope, mass/activity and/or enrichment in a known and representative matrix configuration. The confirmation test item(s) are then measured using the “initial calibration” of the NDA system. The number of differing test item configurations used to confirm the calibration is to be determined by the NDA organization and documented. The reported “calibration confirmation” measurement result must agree, with criteria as established by the NDA organization, with the known element/isotope, mass/activity and/or enrichment of the confirmation test item(s). The NDA organization acceptance criteria shall not exceed the criteria as presented in Section 1.1.3 unless technically justified and documented.
- b) The radioactive sources used for “calibration confirmation” purposes shall, to the extent practicable, be representative of the actual radioactive material compositions and chemical compounds as found in the measurement item inventory of interest.
- c) Radioactive material standards used for “calibration confirmation” are to be traceable to a nationally recognized reference base (e.g., National Institute of Standards and Technology [NIST] or New Brunswick Laboratory [NBL]). The traceable standards used for “calibration confirmation” shall not be related to (from the same feedstock or lineage) those used to perform the “initial calibration”. Noncompliance with this requirement, due to lack of a sufficient variety of traceable standards, can be temporarily waived provided an adequate alternate confirmation strategy is devised.
- d) Calibration confirmation acceptance is assessed through the degree of agreement between the known “calibration confirmation” test item value and that as per the NDA confirmation measurement result. The NDA organization is to determine and document representative “calibration confirmation” source/matrix surrogate configuration(s). The NDA organization may also develop “calibration confirmation” bias and precision acceptance criteria specific to the NDA system and measurement items under consideration. Recommended “calibration confirmation” acceptance criteria are delineated in Section 1.1.3.
- e) Calibration confirmation results outside NDA organization defined acceptance criteria require implementation of corrective action(s) as applicable. Calibration confirmation results are not to exceed the maximum allowable acceptance criteria of Section 1.1.3 unless the NDA organization has specifically determined and documented greater limits with the requisite technical justification.

- f) For the case where a corrective action was required and subsequently implemented, the “calibration confirmation” process is to be repeated. Acceptable results must be obtained and documented before the NDA system is considered operational. Where a “calibration confirmation” failure was determined to be due to a minor issue (e.g., wrong constant, wrong efficiency file, or an inappropriate test item), the entire “calibration confirmation” measurement regimen may not need to be repeated. This is acceptable provided it is the true cause of the failure. All corrective actions and their effects, supporting data, results, etc., shall be documented and retained.
- g) In the case where the “calibration confirmation” was acceptable for certain types or categories of radioactive material/waste matrix configurations, but unacceptable for other categories with distinctly different source/matrix properties, conditional acceptance of the “calibration confirmation” can be made. The NDA organization, however, must clearly identify which categories of source/matrix configurations are approved for NDA measurement and which are not. The technical basis for accepting certain source/matrix categories shall be documented and available for review. Recalibration or corrective action efforts should be implemented and documented for source/matrix categories not meeting acceptance criteria for “calibration confirmation”.
- h) The “calibration confirmation” process shall be performed following an initial calibration or when there are indications warranting a re-assessment of the “initial calibration”, e.g., the source/matrix configuration of measurement items varies relative to the source/matrix configurations used to develop the “initial calibration”. Additional causes for a performing a “calibration confirmation” include:
  - 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.

- 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 <sup>a</sup> | 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

$$\frac{s}{m} \cdot 100\% < \sqrt{\frac{(0.292)^2 \cdot c_{0.05, n-1}^2}{n-1}} \cdot 100\%$$

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

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

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

#### **1.1.4 Calibration Verification**

“Calibration verification” is a measure designed to provide continual and long-term information on the stability of the “initial calibration” while minimizing the impact on NDA operational schedules and resources. The “calibration verification” test item(s) must meet the bias acceptance criteria delineated in Section 1.1.3. A “calibration verification” shall be performed at least once every five operational days for each measurement system and calibration in use. A five day operational period is defined as a rolling tally of five days where NDA operations were in effect, not necessarily consecutive. The start point for the five day operational period is from the start of approved operations or the first operational day after the previous rolling five day tally was completed. The five day operational “calibration verification” requirement may be extended to a maximum of thirty operational days provided the NDA organization can demonstrate and technically justify the long term stability of the NDA system per established acceptance criteria.

Calibration verification test items are typically selected from or assembled from the traceable standards and matrix containers or process component mock-ups used in the “calibration confirmation” process. The “calibration verification” test item is to be submitted to NDA operations in a “blind” manner, where applicable, and processed through the measurement routine as though it were an actual measurement item. The “calibration verification” test items are to be selected and/or configured and submitted such that during a 12-month period the operational space of the NDA system “initial calibration” is spanned. The “calibration verification” is a point check in the calibration realm. It is not required that each waste matrix type comprising the operational space of the NDA system be tested. However, it is expected that the “calibration verification” configurations vary over the operational space. The NDA organization is responsible for specification, assembly and selection of “calibration verification” test items and meeting the applicable rolling operational day period, (i.e., minimum five days, maximum thirty days).

Acceptable performance for a “calibration verification” measurement result in terms of bias, trending measures and so forth shall be determined and documented by the NDA organization. It is recommended that the “calibration confirmation” acceptability requirements of Section 1.1.3 be considered in this process. A CAP for out-of-control “calibration verification” results is to be prepared by the NDA organization. The CAP shall include a provision requiring the evaluation

of measurement item data potentially affected by the failed “calibration verification” measure. The “calibration verification” protocol, monitoring, acceptance criteria, action levels, etc., are to be clearly documented and readily available for review. The calibration verification data is to be control charted and monitored for trends over time.

The NDA organization can utilize other methods of “calibration verification” provided they are technically justifiable and documented.

## 1.2 NDA Method Detection Limit

A methodology shall be in place to determine NDA measurement system detection limit for those radionuclides specified per the client/end-user requirements. It shall be re-determined each time there is a significant change in the measurement method or matrix configuration. Instruments performing low-level waste discrimination measurements must have a minimum detectable activity (MDA)/lower limit of detection (LLD) sufficient to meet the acceptance criteria. The methodology for determination of the MDA/LLD is to be documented by the NDA organization.

The LLD is that level of radioactivity which, if present, yields a measured value greater than the critical level ( $L_c$ ) with a 95% probability, where the  $L_c$  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.,  $^{99}\text{Tc}$ . 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  $\beta$  of non-detection (Type II error) while accepting a probability  $\alpha$  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 ( $\alpha$ ) and beta ( $\beta$ ) 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.

## 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

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  $^{252}\text{Cf}$  source shall be certified in its neutron yield and isotopic composition used to calculate the decay rate, and a mixed nuclide source used to perform an efficiency calibration of a gamma-ray detector shall be certified for the yield of each gamma ray energy used in the calibration and the decay properties of the contributing nuclides).
- c) The NDA organization shall have a procedure(s) for the specification, procurement and acceptance of WRMs. The WRM certifications shall be acquired and maintained, and traceable to a nationally recognized reference base (e.g., NIST, NBL).
- d) The NDA service provider shall retain records for all WRMs including the manufacturer/vendor, the manufacturer's Certificate of Traceability, the date of receipt, and a certificate expiration date.
- e) Traceable standards shall be verified at a minimum of every five years. Standards with an expiration date less than five years shall be verified at a period equal to the time expiration time interval. Verification of a standard is accomplished through an assessment of its usable attribute to the NDA application (e.g.,  $^{235}\text{U}$  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.

## 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.

- 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 measurable by NDA methods be quantified and/or the minimum detectable activity determined and reported.

For radionuclides to be reported per contractual requirements, but not quantifiable through existing NDA techniques, isotopic ratios or radionuclide scaling factors based on acceptable knowledge (AK) of the facility process are commonly employed. The radionuclides and isotopes that are quantifiable through the NDA methods are used in conjunction with AK derived ratios and scaling factors to quantify the radionuclides not directly quantifiable. To use AK to determine such ratios and scaling factors, the NDA organization must technically justify the AK

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

a) AK Documentation

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

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

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

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

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

b) Supplemental AK Information

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

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

c) AK Discrepancy Resolution

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

## 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.

- 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. <sup>235</sup>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,

- 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  $\sigma$ , two  $\sigma$ ). 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.

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.

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 ( $^{235}\text{U}$ ,  $^{238}\text{U}$ , etc.) to be characterized;
- b) Unacceptable NDA results for PE test sample(s), as determined per PE program criteria, will require the NDA organization to implement corrective action procedures and submit a corrective action plan to the PE program or applicable oversight agency. Results of the corrective action plan shall be documented and available for review.
- c) Documentation of successful capability demonstration such as a Certification Statement or letter of concurrence from the qualifying agency must be acquired .and retained by the NDA organization. All associated supporting data necessary to reproduce the PE measurement results as contained in the Certification Statement or equivalent document must be retained by the NDA organization.
- d) Once the initial capability demonstration is successfully completed, continuing demonstration of method performance is to be accomplished through the periodic "calibration verification" measurements as well as all applicable QC requirements.

## 2.0 NDA Quality Control

The purpose of a measurement control program is to test and ensure the stability of the measurement process and to gain additional information on measurement uncertainties where practicable. The measurement control program provides for the administration, evaluation, and control of measurement processes. The design of the measurement control program is to ensure the NDA measurement process provides data of sufficient quality (i.e., the measurement system is in control per defined criteria). The NDA organization can then make and document qualifying statements about the suitability and validity of measurement data as generated for the client and/or end-user.

QC measurements are to be performed in conjunction with and related to a batch of NDA measurement items. A batch is a grouping of similar measurement items to which a set of QC criteria is applied to demonstrate acceptability of the results. The batch size is specified to be 20 items such that when one replicate is performed per batch, a 5% check of the data is achieved.

In addition to the replicate requirement are pre- and post-batch QC checks (e.g., background and energy calibration checks). A batch can be fewer than 20 items as 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.

- g) Written procedures shall be developed and documented to address out-of-control conditions and the subsequent re-qualification of the instrument.

## 2.2 NDA QC Requirements

Procedures cited in various ASTM, ANSI standards, NRC standard practices, and guidelines as referenced in Appendix A are recommended for use at all NDA measurement facilities. QC requirements must at a minimum include the following:

- a) Background Measurements must be performed and recorded for neutron and gamma systems for each system in use at least once per day and twice for each batch. The once per day background measurement can serve as the beginning or ending background measurement required for the batch. The two background measurements for each batch shall bracket the start and end of the batch, one at the beginning of the batch and one at the end of the batch, unless technical justification to do otherwise is developed and documented. The count time for neutron and gamma background checks shall be at least as long as the measurement count time unless otherwise specified and documented by an appropriately qualified individual. The background measurement shall be evaluated before daily NDA measurements commence. Depending on environmental conditions, the background frequency may need to be increased to ensure data quality. Increases in the frequency of background measurements shall be determined and documented by an appropriately qualified individual (Note: Enrichment measurement systems that employ an infinite-thickness analysis technique do not require a background performance check). The recorded background data is to be monitored using control charts or tolerance charts to ensure the background environment is within statistical control. Contributions to background because of radiation from nearby radiation producing equipment, standards, or wastes must be controlled to the extent practicable or more frequent background checks must be performed.
- b) Instrument Performance Measurement checks must be acquired for each NDA measurement system in use at least once per day and twice for each data batch. For each performance check two measurements shall be used to bracket the batch, one before and one after the batch measurements are completed. Performance checks include detection efficiency checks, matrix correction checks, and for spectrometric instruments, energy calibration and energy resolution checks. The NDA organization is to establish acceptable performance check ranges or limits as applicable. An out-of control energy calibration check may cause measurement item results to be suspect since the last successful energy calibration check. Energy calibration checks can be performed at a greater frequency than once per day. Performance checks, as applicable, shall also be acquired on support equipment. The recorded performance measurement checks are to be monitored using control charts or tolerance charts to ensure the instrument performance is within statistical control.
- c) Replicate Measurements are used to determine the repeatability of a measurement system that represents the intrinsic instrument variability. Repeatability variance is a short-term variance usually dominated by counting statistics. The replicate

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

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

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

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

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 <sup>a</sup>    | Required Response   |
|------------------|-------------------------------------|---|
| Acceptable Range | $ Data ^b \leq 2\sigma^c$           | No action required  |
| Warning Range    | $2\sigma^c <  Data  \leq 3\sigma^c$ | The performance check shall be rerun no more than two times. If the rerun performance result is within $2\sigma$ , then the additional performance checks shall be documented and work may continue. If the system does not fall within the $\pm 2\sigma$ after two rerun performance checks, then the required response for Action Range shall be followed.  |
| Action Range     | $ Data  > 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.