# DEPARTMENT OF DEFENSE
# QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES

**Prepared By**

**DoD Environmental Data Quality Workgroup**

**Final Version 3**
**January 2006**

# Quality Systems Manual for Environmental Laboratories

**VERSION 3 FINAL**

**Based On**
**National Environmental Laboratory Accreditation Conference (NELAC)**
**Chapter 5 (Quality Systems)**
**NELAC Voted Version – 5 June 2003**

**Prepared By**

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## PREFACE TO THE DOD QUALITY SYSTEMS MANUAL

### *Purpose*

This document provides requirements and guidance on the establishment and management of quality systems for environmental testing laboratories that intend to perform work for DoD. This document is based on the National Environmental Laboratory Accreditation Conference's (NELAC) Chapter 5 Quality Systems standard and provides implementation clarification and expectations for DoD environmental programs. It is designed to serve as a standard reference for DoD representatives from all Components who design, implement, and oversee contracts with environmental testing laboratories. While the standard is based on NELAC requirements, laboratories providing services to the Department of Defense are not required to be approved by a National Environmental Laboratory Accreditation Program accrediting authority.

### *Background*

To be accredited under the National Environmental Laboratory Accreditation Program (NELAP), laboratories shall have a comprehensive quality system in place, the requirements for which are outlined in NELAC Chapter 5 (Quality Systems). Using NELAC Chapter 5 as its textual base, the *DoD Quality Systems Manual for Environmental Laboratories* is designed to replace common components of the following documents, previously issued by individual Components of DoD:

- United States Navy – Enclosure 1 to Appendix A of the Navy *Installation Restoration Chemical Data Quality Manual (IR CDQM)*, September 1999.
- Air Force Center for Environmental Excellence – *Quality Assurance Project Plan, Version 3.*1, July 2000.
- U.S. Army Corps of Engineers (USACE) – Appendix I of Engineer Manual (EM) 200-1-3, February 2001.

In combining the common components of these three documents, this manual allows laboratories to design quality systems to meet basic requirements for laboratory accreditation under NELAP, as well as meets the implementation needs of all DoD Components. The document achieves this by summarizing and elaborating on DoD's expectations of the laboratory with respect to the implementation of specific components of the NELAC Quality System.

The DoD Environmental Data Quality Workgroup is currently developing policy and procedures for the implementation of this manual. As such, until such time as further standardization by DoD occurs, this document may be supplemented by Component-specific requirements.

### *Project-Specific Requirements*

Requirements contained in this manual may be supplemented by **project-specific requirements or regulations**. The laboratory bears the responsibility for meeting all **State requirements**. Nothing in this document relieves any laboratory from complying with contract requirements, with host-nation final governing standards (as applicable), or with **Federal, State, and/or local regulations**.

### Results and Benefits

The side-by-side integration of NELAC requirements with clarifications by DoD regarding implementation creates several benefits for the laboratory, DoD, and the regulatory communities.

- **Standardization of Processes –** Because this manual provides laboratories with a comprehensive set of requirements that meet the needs of all DoD clients, as well as NELAP, the laboratory may use it to create a standardized quality system. Ultimately, this standardization will save laboratory resources by establishing one set of consistent requirements for all DoD environmental work. The standardized guidance will also serve to "level the playing field" for laboratories competing for DoD contracts, because the expectations will be identical across all DoD Components. An assessment that satisfies the needs of one Component will satisfy comparable needs of the other Components as well. As such, this manual will facilitate the standardization of audits, of assessment, which are consistent and transferable between Components. The result will be saved resources for both the Government and private sector.

- **Deterrence of Improper, Unethical, or Illegal Actions** – Improper, unethical, or illegal activities by only a few laboratories have implications throughout the industry, with negative impacts on all laboratories. This manual addresses this issue by establishing a minimum threshold program for all laboratories to use to deter and detect improper, unethical, or illegal actions.

- **Specification of Compliance Requirements** – This manual applies to all laboratories involved in compliance testing.

- **Foundations for the Future** – A standardized approach to quality systems that is shared by laboratories, NELAP, and DoD will pave the way for the standardization of other processes in the future. For example, this manual might serve as a platform for a standardized strategy for implementing nonstandard methods.

***Audience***

This manual is designed to meet the needs of the following audiences:

- Public (i.e., Government) and private laboratories, contracted with DoD either directly or through a prime contractor or subcontractor;
- DoD contracting personnel, who will use this document to ensure consistency with NELAP when drafting contracts; and
- DoD oversight personnel and assessors, who will use this document to uniformly and consistently evaluate the laboratory's implementation of NELAP and DoD program requirements.

***Document Format***

Because the *DoD Quality Systems Manual for Environmental Laboratories* is designed to complement and implement NELAC Chapter 5 (Quality Systems), that document serves as the primary text for this implementation manual. NELAC revised Chapter 5 in 2002 to follow ISO/IEC 17025:1999, which supersedes ISO/IEC Guide 25. As a result, the version of this manual has been similarly revised. The section numbering has been slightly changed from that of NELAC Chapter 5, as the manual is meant to be a stand-alone document. The number 5 has been eliminated from all section and subsection headings. However, second-level numbering has been retained to maintain an organization parallel to the NELAC Quality Systems requirements. For instance, Section 5.4.2 in NELAC Chapter 5 is equivalent to Section 4.2 in this manual. DoD clarifications that elaborate on specific NELAC requirements are presented in gray text boxes in the applicable section of the document. This allows laboratories preparing for NELAP accreditation to implement their quality systems in a way that fulfills the needs of DoD as well as NELAP. For ease of reference, each gray clarification box in the document is numbered. In addition, there are two sets of appendices to this DoD manual. The first set is the NELAC appendices, modified with DoD clarification boxes. The second set is DoD appendices. The DoD appendices include specific focus on areas of standardization that will be implemented across all DoD Components for laboratory services.

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## ACRONYM LIST

**°C:** Degrees Celsius
**ANSI/ASQC:** American National Standards Institute/American Society for Quality Control
**ASTM:** American Society for Testing and Materials
**CAS:** Chemical Abstract Service
**CCV:** Continuing calibration verification
**CFR:** Code of Federal Regulations
**CLP:** Contract Laboratory Program
**COC:** Chain of custody
**CV:** Coefficient of variation
**DO:** Dissolved oxygen
**DOC:** Demonstration of capability
**DoD:** Department of Defense
**DQOs:** Data quality objectives
**EC:** Exposure concentration
**EPA:** Environmental Protection Agency
**g/L:** Grams per liter
**GC/MS:** Gas chromatography/mass spectrometry
**ICP-MS:** Inductively coupled plasma-mass spectrometer
**ICV:** Initial calibration verification
**ID:** Identifier
**ISO/IEC:** International Organization for Standardization /International Electrotechnical Commission
**LC50:** Lethal concentration at 50%
**LCS:** Laboratory control sample
**MDL:** Method detection limit
**mg/kg:** Milligrams per kilogram
**MQO:** Measurement quality objective
**MS:** Matrix spike
**MSD:** Matrix spike duplicate
**NELAC:** National Environmental Laboratory Accreditation Conference
**NELAP:** National Environmental Laboratory Accreditation Program
**NIST:** National Institute of Standards and Technology
**NOEC:** No-observable-effects concentration
**OSHA:** Occupational Safety and Health Administration
**PC:** Personal computer
**PCBs:** Polychlorinated biphenyls
**PT:** Proficiency testing
**PTOB/PTPA:** Proficiency Testing Oversight Body/Proficiency Testing Provider Accreditor
**QA:** Quality assurance
**QAD:** Quality Assurance Division (EPA)
**QAMS:** Quality Assurance Management Section
**QAPP:** Quality Assurance Project Plan
**QC:** Quality control
**RL:** Reporting limit
**RPD:** Relative percent difference
**RSD:** Relative standard deviation
**SD:** Serial dilutions
**SMSD:** Statistical minimum significant difference
**SOP:** Standard operating procedure
**TAC:** Test acceptability criteria
**TSS:** Total suspended solids
**UV:** Ultraviolet
**VOC:** Volatile organic compound
**WET:** Whole effluent toxicity

## QUALITY SYSTEMS

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

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

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

**ISO 9002:** ISO 9001:2000 replaced the 1994 versions of ISO 9001 and ISO 9002, therefore ISO 9002 no longer exists.

**1**

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

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

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

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

### 1.0 SCOPE

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

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

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

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

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

**Scope of DoD Document:**

- These standards are applicable to any laboratory providing sample analysis to support environmental programs for DoD installations and facilities within the United States, its possessions, and internationally.
- These standards are intended to apply to laboratories that produce definitive data, regardless of the methods being applied (i.e., scientifically valid and legally admissible data).
- These standards may be supplemented by project-specific requirements, as agreed upon by the DoD, regulators, laboratories, and other involved parties.
- The laboratory bears the responsibility for meeting all regulatory agency requirements. Nothing in this document relieves any laboratory from complying with contract requirements, with host-nation final governing standards, or with Federal, State, and/or local regulations.

**2**

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

**DoD Clarification Boxes:** Section 1.3 refers to notes in plain text of the manual. Gray DoD clarification boxes (such as this one) do contain requirements and are an integral part of this manual.

**3**

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

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

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

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

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

| | |
|---|---|
| Management Responsibilities | 4.2.6, 4.2.6.1, and 4.2.6.2 |
| Training | 5.2.7 |
| Control and Documentation | 4.15 |

## 2.0 REFERENCES

See Appendix A.

## 3.0 TERMS AND DEFINITIONS

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

See Appendix A, Glossary, of NELAC Chapter 1.

**Definitions:** For reference purposes, applicable terms from the NELAC Glossary are included as Appendix B in this DoD manual. Furthermore, additional terms not currently included in the NELAC Glossary are defined by DoD to aid the laboratory in implementing this standard appropriately. These terms are also in Appendix B.

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## 4.0 MANAGEMENT REQUIREMENTS

### 4.1 Organization

**4.1.1** The laboratory or the organization of which it is part shall be an entity that can be held legally responsible.

**4.1.2** It is the responsibility of the laboratory to carry out its environmental testing activities in such a way as to meet the requirements of this Standard and to satisfy the needs of the client, the regulatory authorities or organizations providing recognition.

**4.1.3** The laboratory management system shall cover work carried out in the laboratory's permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

**4.1.4** If the laboratory is part of an organization performing activities other than environmental testing, the responsibilities of key personnel in the organization that have an involvement or influence on the environmental testing activities of the laboratory shall be defined in order to identify potential conflicts of interest.

a) Where a laboratory is part of a larger organization, the organizational arrangements shall be such that departments having conflicting interests, such as production, commercial marketing or financing do not adversely influence the laboratory's compliance with the requirements of this Standard.

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

**4.1.5** The laboratory shall:

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

b) have processes to ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work;

c) have policies and procedures to ensure the protection of its clients' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results;

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

d) have policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment or operational integrity;

e) define the organization and management structure of the laboratory, its place in any parent organization, and the relationships between quality management, technical operations and support services;

f) specify the responsibility, authority and interrelationships of all personnel who manage, perform or verify work affecting the quality of the environmental tests;

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

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

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

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

The technical director(s) shall meet the requirements specified in the Accreditation Process (see 4.1.1.1 of NELAC).

i) appoint a member of staff as quality manager (however named) who, irrespective of other duties and responsibilities, shall have defined responsibility and authority for ensuring that the quality system is implemented and followed at all times; the quality manager shall have direct access to the highest level of management at which decisions are made on laboratory policy or resources;

Where staffing is limited, the quality manager may also be the technical director or deputy technical director.

The quality manager (and/or his/her designees) shall:

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

2) have functions independent from laboratory operations for which they have quality assurance oversight;

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

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

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

6) arrange for or conduct internal audits as per 4.13 annually; and,

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

**Quality Assurance – Duty of Quality Manager:** The quality manager shall also be responsible for ensuring continuous improvement at the laboratory through the use of control charts and other method performance indicators (for example, proficiency testing (PT) samples and internal and external audits). **5**

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

k) for purposes of qualifying for and maintaining accreditation, each laboratory shall participate in a proficiency test program as outlined in Chapter 2 of NELAC.

**Proficiency Testing Program – Corrective Actions:** Laboratory management is responsible for following through with proficiency testing programs and for ensuring that corrective actions are implemented after testing and evaluating the effectiveness of the corrective actions. **6**

## 4.2 Quality System

**4.2.1** The laboratory shall establish, implement and maintain a quality system based on the required elements contained in this chapter and appropriate to the type, range and volume of environmental testing activities it undertakes. The laboratory shall document its policies, systems, programs, procedures and instructions to the extent necessary to assure the quality of the environmental test results. The system's documentation shall be communicated to, understood by, available to, and implemented by the appropriate personnel.

**Quality System Documentation:** This documentation includes the quality manual, standard operating procedure (SOP) documents, and other appropriate reference documents and texts. Copies of all quality system documentation provided to DoD for review must be available in English. **7**

**4.2.2** The laboratory's quality system policies and objectives shall be defined in a quality manual (however named). The overall objectives shall be documented in a quality policy statement. The quality

policy statement shall be issued under the authority of the chief executive. It shall include at least the following:

a) the laboratory management's commitment to good professional practice and to the quality of its environmental testing in servicing its clients; The laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

b) the management's statement of the laboratory's standard of service;

c) the objectives of the quality system;

The laboratory management shall ensure that these policies and objectives are documented in a quality manual.

d) a requirement that all personnel concerned with environmental testing activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work; and

e) the laboratory management's commitment to compliance with this Standard.

**4.2.3** The quality manual shall include or make reference to the supporting procedures including technical procedures. It shall outline the structure of the documentation used in the quality system.

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

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

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

**Quality Manual Updating:** The following list reflects topic areas that shall be included in the quality manual. Additional details about each topic area are provided in the sections that follow. The manual shall be reviewed at least annually for accuracy and adequacy, and updated as appropriate. All such reviews shall be documented and available for assessment.

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The quality manual and related quality documentation shall also contain:

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

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

**Corporations – Laboratory Relationships with Corporations:** This includes the laboratory's relationship(s) to corporate affiliations and networks.

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

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

**Document Control – Distribution:** Consistent with the definition of "Document Control" provided in NELAC Appendix B, this control system shall ensure that all analysts implementing the task(s) or procedure(s) described in that SOP shall be made individually aware that changes to an SOP have occurred. A copy (paper or electronic) of the updated SOP shall be available in close proximity to the workstation (i.e., within the same work area).

**10**

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

**Personnel To Be Included in Quality Manual:** At a minimum, the following managerial and supervisory staff (however named) shall be considered key staff, and their job descriptions shall be included in the quality manual and other related documents:

(1) Executive Staff (for example, Chief Executive Officer, Chief Operating Officer, laboratory director, technical director);
(2) Technical directors/supervisors (for example, section supervisors for organics and inorganics);
(3) Quality systems directors/supervisors (for example, quality manager, quality auditors); and
(4) Support systems directors/supervisors (for example, information systems supervisor, purchasing director, and project managers).

In addition, the quality manual shall include job descriptions for key staff in each of these four areas, as appropriate to the laboratory.

If the size and organization of the laboratory precludes separate managers and/or supervisors in each of these key areas, the functions covered in the four areas shall be addressed in the job descriptions provided for the key staff.

The quality manual shall describe the relationship of the key staff listed above to other technical and support staff. Any changes in key personnel for the laboratory must be documented to all laboratory users.

Other technical staff includes those individuals who conduct the work of the laboratory (for example, sample receipt) and documentation staff, the chemists who perform sample preparation and analysis. Support staff administers the business practices of the laboratory, as well as information management, purchasing, and contractual systems. Quality staff oversees the implementation of the quality system and reports to the quality manager or his/her designee.

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

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

**Traceability of Measurements:** Standards addressing this issue are included in Section 5.6 (Measurement Traceability), Section 5.6.4 (Documentation and Labeling of Standards, Reagents, and Reference Materials), and Section 4.12 (Control of Records).

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

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

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

k) procedures for handling submitted samples;

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

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

n) reference to verification practices which may include interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

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

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

q) procedures for dealing with complaints;

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

s) procedures for audits and data review;

**Audits – Quality Manual Specification:** The quality manual or a referenced standard operating procedure shall also specify which records are considered necessary to conduct an adequate review. **13**

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

u) reference to procedures for reporting analytical results; and

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

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

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

**4.2.6** The laboratory shall establish and maintain data integrity procedures. These procedures shall be defined in detail within the quality manual. There are four required elements within a data integrity system. These are 1) data integrity training, 2) signed data integrity documentation for all laboratory employees, 3) in-depth, periodic monitoring of data integrity, and 4) data integrity procedure documentation. The data integrity procedures shall be signed and dated by senior management. These procedures and the associated implementation records shall be properly maintained and made available for assessor review. The data integrity procedures shall be annually reviewed and updated by management.

**4.2.6.1** Laboratory management shall provide a mechanism for confidential reporting of data integrity issues in their laboratory. A primary element of the mechanism is to assure confidentiality and a receptive

environment in which all employees may privately discuss ethical issues or report items of ethical concern.

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

**Program to Detect and Prevent Improper, Unethical, or Illegal Actions:** Additional descriptions related to this requirement are included in Section 5.2.7 and DoD clarification box 29.
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## 4.3 Document Control

### 4.3.1 General

The laboratory shall establish and maintain procedures to control all documents that form part of its quality system (internally generated or from external sources). Documents include policy statements, procedures, specifications, calibration tables, charts, textbooks, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic or written.

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

### 4.3.2 Document Approval and Issue

**4.3.2.1** All documents issued to personnel in the laboratory as part of the quality system shall be reviewed and approved for use by authorized personnel prior to issue. A master list or an equivalent document control procedure identifying the current revision status and distribution of documents in the quality system shall be established and be readily available to preclude the use of invalid and/or obsolete documents.

**4.3.2.2** The procedure(s) adopted shall ensure that:

a) authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed;

b) documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements;

c) invalid or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use; and

d) obsolete documents retained for either legal or knowledge preservation purposes are suitably marked.

**4.3.2.3** Quality system documents generated by the laboratory shall be uniquely identified. Such identification shall include the date of issue and/or revision identification, page numbering, the total number of pages or a mark to signify the end of the document, and the issuing authority(ies).

### 4.3.3 Document Changes

**4.3.3.1** Changes to documents shall be reviewed and approved by the same function that performed the original review unless specifically designated otherwise. The designated personnel shall have access to pertinent background information upon which to base their review and approval.

**4.3.3.2** Where practicable, the altered or new text shall be identified in the document or the appropriate attachments.

**4.3.3.3** If the laboratory's documentation control system allows for the amendment of documents by hand, pending the re-issue of the documents, the procedures and authorities for such amendments shall be defined. Amendments shall be clearly marked, initialed and dated. A revised document shall be formally re-issued as soon as practicable.

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

### 4.4 Review of Requests, Tenders and Contracts

**4.4.1** The laboratory shall establish and maintain procedures for the review of requests, tenders and contracts. The policies and procedures for these reviews leading to a contract for environmental testing shall ensure that:

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

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

The purpose of this review of capability is to establish that the laboratory possesses the necessary physical, personnel and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the environmental tests in question. The review may encompass results of earlier participation in interlaboratory comparisons or proficiency testing and/or the running of trial environmental test programs using samples or items of known value in order to determine uncertainties of measurement, detection limits, confidence limits, or other essential quality control requirements. The current accreditation status of the laboratory must also be reviewed. The laboratory must inform the client of the results of this review if it indicates any potential conflict, deficiency, lack of appropriate accredtation status, or inability on the laboratory's part to complete the client's work.

c) the appropriate environmental test method is selected and capable of meeting the clients' requirements (see 5.4.2).

Any differences between the request or tender and the contract shall be resolved before any work commences. Each contract shall be acceptable both to the laboratory and the client.

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

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

For review of routine and other simple tasks, the date and the identification (e.g., the initials) of the person in the laboratory responsible for carrying out the contracted work are considered adequate. For repetitive routine tasks, the review need be made only at the initial inquiry stage or on granting of the contract for on-going routine work performed under a general agreement with the client, provided that the client's requirements remain unchanged. For new, complex or advanced environmental testing tasks, a more comprehensive record should be maintained.

**4.4.3** The review shall also cover any work that is subcontracted by the laboratory.

**4.4.4** The client shall be informed of any deviation from the contract.

**4.4.5** If a contract needs to be amended after work has commenced, the same contract review process shall be repeated and any amendments shall be communicated to all affected personnel. Suspension of accreditation, revocation of accreditation, or voluntary withdrawal of accreditation must be reported to the client.

### 4.5 Subcontracting of Environmental Tests

**4.5.1** When a laboratory subcontracts work whether because of unforeseen reasons (e.g., workload, need for further expertise or temporary incapacity) or on a continuing basis (e.g., through permanent subcontracting, agency or franchising arrangements), this work shall be placed with a laboratory accredited under NELAP for the tests to be performed or with a laboratory that meets applicable statutory and regulatory requirements for performing the tests and submitting the results of tests performed. The laboratory performing the subcontracted work shall be indicated in the final report and non-NELAP accredited work shall be clearly identified.

**Subcontractor Laboratories:**

- Subcontractor laboratories must have an established and documented laboratory quality system that complies with this Manual.
- Subcontractor laboratories must be approved by the specific DoD Component laboratory approval process.
- Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of proficiency testing (PT) samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.
- Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
- Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

These requirements apply to the use of any laboratory under the same corporate umbrella, but at a different physical facility.

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

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

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

### 4.6 Purchasing Services and Supplies

**4.6.1** The laboratory shall have a policy and procedure(s) for the selection and purchasing of services and supplies it uses that affect the quality of the environmental tests. Procedures shall exist for the purchase, reception and storage of reagents and laboratory consumable materials relevant for the environmental tests.

**4.6.2** The laboratory shall ensure that purchased supplies and reagents and consumable materials that affect the quality of environmental tests are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the environmental tests concerned. These services and supplies used shall comply with specified requirements. Records of actions taken to check compliance shall be maintained.

**4.6.3** Purchasing documents for items affecting the quality of laboratory output shall contain data describing the services and supplies ordered. These purchasing documents shall be reviewed and approved for technical content prior to release.

**Purchasing Documents:** These documents shall include date of receipt, expiration date (where applicable), source (i.e., provider or supplier), lot number, and calibration and verification records and certifications for whatever services and supplies may affect the quality of associated test results. Examples of these services and supplies that may have an impact on the quality of data include balance calibration, solvents, standards, Class A glassware, and sample containers. Furthermore, all of these supplies shall be maintained according to the applicable requirements specified in Sections 5.6.3 and 5.6.4.

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

### 4.7 Service to the Client

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

**Client Notification:** Service to the client means proactive engagement, as highlighted in clarifications throughout this manual. Examples of situations that may require client notification include:

- Incorrect, obsolete, or improper methods
- The need to optimize methods to ensure achievement of Quality Assurance Project Plan (QAPP) objectives (e.g., for difficult matrix, poor performing analyte)
- Lack of project guidance documents, such as the QAPP, or the need for clarification of requirements in the document (e.g., action levels, detection and quantitation capabilities)
- Problems with sampling or analysis that may impact results (e.g., improper preservation of sample)

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### 4.8 Complaints

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

**Complaints/Problems Response System:** The laboratory's quality system shall contain a process for responding to complaints and/or problems. At a minimum, this will include tracking of quality checks, internal audits, and quality control trending. Documentation of this response and resolution of the problem, as applicable to DoD, shall be maintained. In addition, the laboratory shall use this information as part of its quality system to identify patterns of problems and to correct them. These logs shall be available for DoD review, to help DoD assess the effectiveness of the laboratory's corrective action process. This information will be considered confidential but will, nonetheless, be used by DoD to assess the effectiveness of the laboratory's quality system.

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## 4.9 Control of Nonconforming Environmental Testing Work

**4.9.1** The laboratory shall have a policy and procedures that shall be implemented when any aspect of its environmental testing work, or the results of this work, do not conform to its own procedures or the agreed requirements of the client. The policy and procedures shall ensure that:

a) the responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding of test reports, as necessary) are defined and taken when nonconforming work is identified;

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

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

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

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

**4.9.2** Where the evaluation indicates that the nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the corrective action procedures given in 4.10 shall be promptly followed.

## 4.10 Corrective Action

### 4.10.1 General

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

### 4.10.2 Cause Analysis

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

### 4.10.3 Selection and Implementation of Corrective Actions

Where corrective action is needed, the laboratory shall identify potential corrective actions. It shall select and implement the action(s) most likely to eliminate the problem and to prevent recurrence.

Corrective actions shall be to a degree appropriate to the magnitude and the risk of the problem.

The laboratory shall document and implement any required changes resulting from corrective action investigations.

### 4.10.4 Monitoring of Corrective Actions

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

### 4.10.5 Additional Audits

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

### 4.10.6 Technical Corrective Action

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

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

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

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

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

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

**Audits – Corrective Action:** Managers, including the quality manager, are also responsible for acting upon corrective action report reviews. Furthermore, managers are ultimately accountable for the follow-through, verification, and evaluation of these corrective actions.

**Monitoring of Corrective Actions:** Historical corrective action reports should be periodically reviewed to identify long-term trends or recurring problems.

**Regular Review:** All operations shall be systematically and thoroughly reviewed at regular intervals (at least annually) to:

- Obtain input on the laboratory's operations:
- Determine what considerations need to be given to input (from reviews); and
- Determine how corrective action(s), if necessary, shall be carried out.

**Reference:** American Society for Quality Control. 1991. Q2 – Quality Management and Quality System Elements for Laboratories – Guidelines.

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

**Data Qualifiers:** Some of the standard data qualifiers, to be used by laboratories only, are listed below. Additional data qualifiers may be used by data validators when evaluating data usability (for example, an "R" flag for rejected data).

U – Undetected at the limit of detection: The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J – Estimated: The analyte was positively identified; the quantitation is an estimation (for example, matrix interference, outside the calibration range).
B – Blank contamination: The analyte was detected above one-half the reporting limit in an associated blank (see DoD clarification box D-5).
N – Nontarget analyte: The analyte is a tentatively identified compound (using mass spectroscopy).
Q – One or more quality control criteria (for example, LCS recovery, surrogate spike recovery) failed. Data usability should be carefully assessed by the project team. Assessment by DoD may result in rejection of data and potential contractual nonpayment based on unacceptable performance.

These flags are a minimum. If a laboratory has more and they are consistent with DoD and properly defined, the laboratory may use them. When other flags are required contractually, those shall be used. In addition, data qualifiers may be combined when appropriate.

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### 4.11 Preventive Action

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

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

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

### 4.12 Control of Records

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

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

#### 4.12.1 General

**4.12.1.1** The laboratory shall establish and maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records shall include reports from internal audits and management reviews as well as records of corrective and preventive actions. Records may be in any media, such as hard copy or electronic media.

**4.12.1.2** All records shall be legible and shall be stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss. Retention times of records shall be established.

**4.12.1.3** All records shall be held secure and in confidence.

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

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

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

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

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

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

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

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

g) Refer to 5.4.7.2 for Computer and Electronic Data.

### 4.12.2 Technical Records

**4.12.2.1** The laboratory shall retain records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each test report issued, for a defined period. The records for each environmental test shall contain sufficient information to facilitate identification of factors affecting the uncertainty and to enable the environmental test to be repeated under conditions as close as possible to the original. The records shall include the identity of personnel responsible for the sampling, performance of each environmental test and checking of results.

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

**4.12.2.3** When mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records shall be signed or initialed by the person making the correction. In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data.

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

#### 4.12.2.4 Records Management and Storage

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

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

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

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

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

f) The laboratory shall have a plan to ensure that the records are maintained or transferred according to the clients' instructions (see 4.1.8.e of NELAC) in the event that a laboratory transfers ownership or goes out of business. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

#### 4.12.2.5 Laboratory Sample Tracking

##### 4.12.2.5.1 Sample Handling

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

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

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

c) sample storage and tracking including shipping receipts, sample transmittal forms (chain of custody form); and

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

##### 4.12.2.5.2 Laboratory Support Activities

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

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

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

c) copies of final reports;

d) archived SOPs;

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

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

g) proficiency test results and raw data; and

h) results of data review, verification, and crosschecking procedures.

#### 4.12.2.5.3 Analytical Records

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

a) laboratory sample ID code;

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

**Analytical Records:** For DoD work, laboratory identification and both date and time of analysis are considered to be essential information, and shall be included as part of the analytical record. **21**

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

d) analysis type;

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

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

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

h) sample analysis;

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

j) calibration criteria, frequency and acceptance criteria;

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

l) quality control protocols and assessment;

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

n) method performance criteria including expected quality control requirements.

#### 4.12.2.5.4 Administrative Records

The following shall be maintained:

a) personnel qualifications, experience and training records;

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

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

### 4.13 Internal Audits

**Audits and Reviews**: The following two sections refer to internal assessment tools to be used by the laboratory. Section 4.13 discusses systems audits and technical audits, both of which shall be conducted annually to evaluate whether the quality system is being implemented at the operational level of the laboratory. Section 4.14 addresses higher-level management reviews, designed to evaluate whether the quality system itself is effective. These can be done in conjunction with each other or separately, at the discretion of the laboratory.

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**4.13.1** The laboratory shall periodically, in accordance with a predetermined schedule and procedure, and at least annually, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the quality system and this Standard. The internal audit program shall address all elements of the quality system, including the environmental testing activities. It is the responsibility of the quality manager to plan and organize audits as required by the schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out.

**Audits – Internal:** Internal audits shall include both technical audits and systems audits. They may be scheduled or unannounced. Technical audits verify compliance with method-specific requirements, as well as operations related to the test method (for example, sample preparation). (These operations include all actions related to data generation and the assurance of its quality.) Systems audits verify compliance with the laboratory's quality system, based on the NELAC Quality System, and are documented in the laboratory's quality manual. Methods for responding to complaints, sample acceptance policies, and sample tracking are examples of procedures that would be reviewed as part of a systems audit. Data audits are considered a subset of technical audits.

An audit schedule shall be established such that all elements/areas of the laboratory are reviewed over the course of 1 year.

Personnel performing an internal audit shall complete the audit under the direction of the quality manager, however named. To be considered "trained and qualified," the internal auditor shall be trained and qualified in conducting the type of audit under review.

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

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

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

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

**Audits – Time Frame:** The time frame for these actions shall be based on the magnitude of the problem and its impact on the defensibility and use of data.
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**4.13.4** Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken.

### 4.14 Management Reviews

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

a) the suitability of policies and procedures;

b) reports from managerial and supervisory personnel;

c) the outcome of recent internal audits;

d) corrective and preventive actions;

e) assessments by external bodies;

f) the results of interlaboratory comparisons or proficiency tests;

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

h) client feedback;

i) complaints; and

j) other relevant factors, such as quality control activities, resources and staff training.

**Management Review:** This is a separate review from the internal audit discussed in Section 4.13.1 and shall be completed by laboratory managerial personnel. As noted in clarification box 22, however, internal audits and management reviews may be conducted in conjunction with each other.
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**4.14.2** Findings from management reviews and the actions that arise from them shall be recorded. The management shall ensure that those actions are carried out within an appropriate and agreed timescale.

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

**4.15** The laboratory, as part of their overall internal auditing program, shall ensure that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery of potential issues shall be handled in a confidential manner until such time as a follow up evaluation, full investigation, or other appropriate actions have been completed and the issues clarified.

All investigations that result in finding of inappropriate activity shall be documented and shall include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. All documentation of these investigation and actions taken shall be maintained for at least five years.

## 5.0 TECHNICAL REQUIREMENTS

### 5.1 General

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

a) human factors (5.2);

b) accommodation and environmental conditions (5.3);

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

d) equipment (5.5);

e) measurement traceability (5.6);

f) sampling (5.7); and

g) the handling of samples (5.8).

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

### 5.2 Personnel

**5.2.1** The laboratory management shall ensure the competence of all who operate specific equipment, perform environmental tests, evaluate results, and sign test reports. When using staff who are undergoing training, appropriate supervision shall be provided. Personnel performing specific tasks shall be qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

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

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

**Technical Directors – Qualifications:** Required qualifications for the technical director(s) are addressed below. DoD stresses that a director or designee meeting the qualifications below shall be present in each area of analytical service. Laboratory management, as addressed in Section 5.2.6, is defined as designees (for example, laboratory manager, technical director, supervisors, and quality managers, however named) having oversight authority and responsibility for laboratory output.

The following requirements are direct excerpts from **NELAC Chapter 4 (Accreditation Process), June 5, 2003**.

**4.1.1 Personnel Qualifications**

Persons who do not meet the education credential requirements but possess the requisite experience of Section 4.1.1.1 of the NELAC standards shall qualify as technical director(s) subject to the following conditions.

a) The person must be a technical director of the laboratory on the date the laboratory applies for NELAP accreditation and/or becomes subject to NELAP accreditation, and must have been a technical director in that laboratory continuously for the previous 12 months or more.

b) The person will be approved as technical director for only those fields of accreditation for which he/she has been technical director in that laboratory for the previous 12 months or more.

c) A person who is admitted as a technical director under these conditions, and leaves the laboratory, will be admitted as technical director for the same fields of accreditation in another NELAP laboratory.

d) A person may initially be admitted as a technical director under the provisions of this section during the first twelve months that the primary Accrediting Authority offers the NELAP fields of accreditation for which the person seeks to be technical director or during the first twelve months that the program is required by the state in which the laboratory is located.

**4.1.1.1 Definition, Technical Director(s)**

The technical director(s) means a full-time member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results. The title of such person may include but is not limited to laboratory director, technical director, laboratory supervisor or laboratory manager. A laboratory may appoint one or more technical directors for the appropriate fields of accreditation for which they are seeking accreditation. His/her name must appear in the national database. This person's duties shall include, but not be limited to, monitoring standards of performance in quality control and quality assurance; monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data. An individual shall not be the technical director(s) of more than one accredited environmental laboratory without authorization from the primary accrediting authority. Circumstances to be considered in the decision to grant such authorization shall include, but not be limited to, the extent to which operating hours of the laboratories to be directed overlap, adequacy of supervision in each laboratory, and the availability of environmental laboratory services in the area served. The technical director(s) who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director(s) to temporarily perform this function. If this absence exceeds 65 consecutive calendar days, the primary accrediting authority shall be notified in writing.

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Qualification of the technical director(s):

a) Any technical director of an accredited environmental laboratory engaged in chemical analysis shall be a person with a bachelor's degree in the chemical, environmental, biological sciences, physical sciences, or engineering, with at least 24 college semester credit hours in chemistry and at least two years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

b) Any technical director of an accredited environmental laboratory limited to inorganic chemical analysis, other than metals analysis, shall be a person with at least an earned associate's degree in the chemical, physical, or environmental sciences, or two years of equivalent and successful college education, with a minimum of 16 college semester credit hours in chemistry. In addition, such a person shall have at least two years of experience performing such analysis.

c) Any technical director of an accredited environmental laboratory engaged in microbiological or biological analysis shall be a person with a bachelor's degree in microbiology, biology, chemistry, environmental sciences, physical sciences, or engineering with a minimum of 16 college semester credit hours in general microbiology and biology and at least two years of experience in the environmental analysis of representative analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

   A person with an associate's degree in an appropriate field of the sciences or applied sciences, with a minimum of four college semester credit hours in general microbiology, may be the technical director(s) of a laboratory engaged in microbiological analysis limited to fecal coliform, total coliform, and standard plate count. Two years of equivalent and successful college education, including the microbiology requirement, may be substituted for the associate's degree. In addition, each person shall have one year of experience in environmental analysis.

d) Any technical director of an accredited environmental laboratory engaged in radiological analysis shall be a person with a bachelor's degree in chemistry, physics, or engineering with 24 college semester credit hours of chemistry with two or more years of experience in the radiological analysis of environmental samples. A master's or doctoral degree in one of the above disciplines may be substituted for one year experience.

e) The technical director(s) of an accredited environmental laboratory engaged in microscopic examination of asbestos and/or airborne fibers shall meet the following requirements:

   i) For procedures requiring the use of a transmission electron microscope, a bachelor's degree, successful completion of courses in the use of the instrument, and one year of experience, under supervision, in the use of the instrument. Such experience shall include the identification of minerals.

   ii) For procedures requiring the use of a polarized light microscope, an associate's degree or two years of college study, successful completion of formal coursework in polarized light microscopy, and one year of experience, under supervision, in the use of the instrument. Such experience shall include the identification of minerals.

   iii) For procedures requiring the use of a phase contrast microscope, as in the determination of airborne fibers, an associate's degree or two years of college study, documentation of successful completion of formal coursework in phase contrast microscopy, and one year of experience, under supervision, in the use of the instrument.

f) Any technical director of an accredited environmental laboratory engaged in the examination of radon in air shall have at least an associate's degree or two years of college and one year of experience in radiation measurements, including at least one year of experience in the measurement of radon and/or radon progeny.

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**5.2.2** The management of the laboratory shall formulate the goals with respect to the education, training and skills of the laboratory personnel. The laboratory shall have a policy and procedures for identifying training needs and providing training of personnel. The training program shall be relevant to the present and anticipated tasks of the laboratory.

**5.2.3** The laboratory shall use personnel who are employed by, or under contract to, the laboratory. Where contracted and additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's quality system.

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

**5.2.5** The management shall authorize specific personnel to perform particular types of sampling, environmental testing, to issue test reports, to give opinions and interpretations and to operate particular types of equipment. The laboratory shall maintain records of the relevant authorization(s), competence, educational and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel. This information shall be readily available and shall include the date on which authorization and/or competence is confirmed.

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

**5.2.6** The laboratory management shall be responsible for:

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

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

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

**Work Cell – Definition of Work Cell:** Additional guidance on this issue is provided in Section 5.4.2.2.f, g, and h and DoD clarification box C-2 (Section C.1). A “work cell” is considered to be all those individuals who see a sample through the complete process of preparation, extraction, and analysis. To ensure that the entire preparation, extraction, and analysis process is completed by a group of capable individuals, the laboratory shall ensure that each member of the work cell (including a new member entering an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a “team,” the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance.

A work cell may NOT be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

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

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

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

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

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

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

   iii. at least four consecutive laboratory control samples with acceptable levels of precision and accuracy. The laboratory must determine the acceptable limits for precision and accuracy prior to analysis; or

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

**Continued Proficiency:** For DoD, a documented, ongoing process of analyst review using QC samples can serve as the annual demonstration of continued proficiency. QC samples can be reviewed to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary.

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

e) supervising all personnel employed by the laboratory;

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

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

**5.2.7** Data integrity training shall be provided as a formal part of new employee orientation and must also be provided on an annual basis for all current employees. Topics covered shall be documented in writing and provided to all trainees. Key topics covered during training must include organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues, and record keeping. Training shall include discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation. Employees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment or civil/criminal prosecution. The initial data integrity training and the annual refresher training shall have a signature attendance sheet or other form of documentation that demonstrates all staff have participated and understand their obligations related to data integrity. Senior managers acknowledge their support of these procedures by 1) upholding the spirit and intent of the organization's data integrity procedures and 2) effectively implementing the specific requirements of the procedures.

Specific examples of breaches of ethical behavior should be discussed including improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards. Data integrity training requires emphasis on the importance of proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially deficient. The data integrity procedures may also include written ethics agreements, examples of improper practices, examples of improper chromatographic manipulations, requirements for external ethics program training, and any external resources available to employees.

**A Program To Detect and Prevent Improper, Unethical, or Illegal Actions:** In order to perform work for DoD under this manual, the laboratory shall have a documented program to prevent improper, unethical, or illegal actions. To facilitate the implementation of this required program, DoD has compiled the following text to (1) clearly define improper, unethical, or illegal actions; (2) outline elements of prevention and detection programs for improper, unethical, or illegal actions; and (3) identify examples of inappropriate (i.e., potentially fraudulent) laboratory practices. Data shall be produced according to the project-specific requirements as specified in the final approved project documents, such as the approved QAPP. The laboratory shall be aware of these requirements and be able to show that these requirements were followed.

**Definitions:** Improper actions are defined as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional. Unethical or illegal actions are defined as the deliberate falsification of analytical or quality assurance results, where failed method or contractual requirements are made to appear acceptable.

**Prevention and Detection Program for Improper, Unethical, or Illegal Actions:** Prevention of laboratory improper, unethical, or illegal actions begins with a zero-tolerance philosophy established by management. Improper, unethical, or illegal actions are detected through the implementation of oversight protocols. Laboratory management shall implement a variety of proactive measures to promote prevention and detection of improper, unethical, or illegal activities. The following components constitute the baseline and minimum requirements for an improper, unethical, or illegal actions prevention program and shall be included as part of the laboratory's comprehensive quality program:

- An ethics policy that is read and signed by all personnel;
- Initial and annual ethics training, as described in Section 5.2.7;
- Internal audits, as described in Section 4.13;
- Inclusion of antifraud language in subcontracts;
- Analyst notation and sign-off on manual integration changes to data (see also DoD clarification boxes 50 and 57);
- Active use of electronic audit functions when they are available in the instrument software (see also Section 5.4.7.2 ); and
- A "no-fault" policy that encourages laboratory personnel to come forward and report inappropriate activities.

A proactive, "beyond the basics" approach to the prevention of improper, unethical, or illegal actions, is a necessary part of laboratory management. As such, in addition to the mandatory requirements above, the laboratory shall institute other actions to deter and detect improper, unethical, or illegal actions (i.e., designate an ombudsman, such as a data integrity officer, to whom laboratory personnel can report improper, unethical, or illegal practices, or provide routine communication of training, lectures, and changes in policy intended to reduce improper, unethical, or illegal actions).

**29 (continued on next page)**

**Examples of Improper, Unethical, or Illegal Practices:** Documentation that clearly shows how all analytical values were obtained shall be maintained by the laboratory and supplied to the data user when necessary. To avoid miscommunication, a laboratory shall clearly document all errors, mistakes, and basis for manual integrations within the case narrative. Notification should also be made to the appropriate people such that appropriate corrective actions can be initiated. Gross deviations from specified procedures should be investigated for potential improper, unethical, or illegal actions, and findings of fraud should be prosecuted to the fullest extent of the law. Examples of improper, unethical, or illegal practices are identified below:

- Improper use of manual integrations to meet calibration or method QC criteria (for example, peak shaving or peak enhancement are considered improper, unethical, or illegal actions if performed solely to meet QC requirements);
- Intentional misrepresentation of the date or time of analysis (for example, intentionally resetting a computer system's or instrument's date and/or time to make it appear that a time/date requirement was met);
- Falsification of results to meet method requirements;
- Reporting of results without analyses to support (i.e., dry-labbing);
- Selective exclusion of data to meet QC criteria (for example, initial calibration points dropped without technical or statistical justification);
- Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported, or QC control limits presented within the laboratory Quality Assurance Manual or SOPs that are not indicative of historical laboratory performance or used for batch control;
- Notation of matrix interference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (for example, method blanks or laboratory control samples);
- Unwarranted manipulation of computer software (for example, improper background subtraction to meet ion abundance criteria for GC/MS tuning, chromatographic baseline manipulations);
- Improper alteration of analytical conditions (for example, modifying EM voltage, changing GC temperature program to shorter analytical run time) from standard analysis to sample analysis;
- Misrepresentation of QC samples (for example, adding surrogates after sample extraction, omitting sample preparation steps for QC samples, over- or underspiking); and
- Reporting of results from the analysis of one sample for those of another.

***References:***

California Military Environmental Coordination Committee (EPA, CAL EPA, and DoD). March 1997. "Best Practices for the Detection and Deterrence of Laboratory Fraud."
U.S. Army Corps of Engineers – Interim Chemical Data Quality Management (CDQM) Policy for USACE HTRW Projects. USACE – HTRW. 8 December 1998.

**29**

### 5.3 Accommodation and Environmental Conditions

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

**Environmental Conditions:** Environmental conditions include heating, cooling, humidity, and ventilation.

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The laboratory shall ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any measurement. Particular care shall be taken when sampling and environmental tests are undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of environmental tests shall be documented.

**5.3.2** The laboratory shall monitor, control and record environmental conditions as required by the relevant specifications, methods and procedures or where they influence the quality of the results. Due attention shall be paid, for example, to biological sterility, dust, electromagnetic disturbances, radiation, humidity, electrical supply, temperature, and sound and vibration levels, as appropriate to the technical activities concerned. Environmental tests shall be stopped when the environmental conditions jeopardize the results of the environmental tests.

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

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

**Samples – Cross-Contamination:** The laboratory shall have procedures in place to ensure that cross-contamination does not occur. Samples designated for volatile organics testing shall be segregated from other samples. Samples suspected of containing high levels of volatile organics shall be further isolated from other volatile organics sample. Storage blanks shall be used to verify that no cross-contamination has occurred.

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**5.3.4** Access to and use of areas affecting the quality of the environmental tests shall be controlled. The laboratory shall determine the extent of control based on its particular circumstances.

**5.3.5** Measures shall be taken to ensure good housekeeping in the laboratory. Special procedures shall be prepared where necessary.

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

a) access and entryways to the laboratory;

b) sample receipt area(s);

c) sample storage area(s);

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

e) data handling and storage area(s).

## 5.4 Environmental Test Methods and Method Validation

### 5.4.1 General

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

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

**Test Method/SOP Updating:** All documentation of methods (for example, instructions, standards, manuals, SOPs) shall be reviewed for accuracy and adequacy at least annually, or whenever procedural method changes occur, and updated as appropriate.

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#### 5.4.1.1 Standard Operating Procedures (SOPs)

Laboratories shall maintain SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

a) These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents with adequate detail to allow someone similarly qualified, other than the analyst, to reproduce the procedures used to generate the test result.

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

**SOPs – Requirements:** Where existing methods are specified as required for a project, requirements contained within that method shall be followed. Any modifications to existing method requirements require project-specific approval by DoD personnel.

Although published test methods may be included as part of an SOP, to fulfill the complete requirements of the SOP (as listed in Section 5.4.1.2.b, items 1-23), laboratories likely will be required to provide additional information beyond the published test method documentation.

**33**

c) Copies of all SOPs shall be accessible to all personnel.

d) The SOPs shall be organized.

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

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

**SOPs – Archiving of SOPs:** All SOPs shall be archived for historical reference in accordance with Section 4.12 (Control of Records).

**34**

#### 5.4.1.2 Laboratory Method Manual(s)

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

**SOPs – Modifications to Existing Methods:** Where existing methods are specified as required for a project, requirements contained within that method shall be followed. Any modifications to existing method requirements require project-specific approval by DoD personnel.

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

**SOPs – Analytical Method SOPs:** These requirements apply to all Analytical Method SOPs. Although published test methods may be included as part of an SOP, to fulfill the complete requirements of the SOP, laboratories may be required to provide additional information beyond the published test method documentation (if not addressed elsewhere), including, but not limited to:

- Troubleshooting;
- Personnel qualifications;
- Data management and records; and
- Computer hardware and software.

**36**

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

### 5.4.2 Selection of Methods

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

#### 5.4.2.1 Sources of Methods

a) Methods published in international, regional or national standards shall preferably be used. The laboratory shall ensure that it uses the latest valid edition of a standard unless it is not appropriate

or possible to do so. When necessary, the standard shall be supplemented with additional details to ensure consistent application.

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

c) When the client does not specify the method to be used or where methods are employed that are not required, the methods shall be fully documented and validated (see 5.4.2.2, 5.4.5, and Appendix C), and be available to the client and other recipients of the relevant reports. The laboratory shall select appropriate methods that have been published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment. Laboratory-developed methods or methods adopted by the laboratory may also be used if they are appropriate for the intended use and if they are validated. The client shall be informed as to the method chosen.

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

#### 5.4.2.2 Demonstration of Capability

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

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

**Capability – New Methods Capability:** In the case of a laboratory introducing a new method, demonstration of performance shall be determined using an external source of information, when available (for example, the published method). If there is no external source of information, the laboratory shall use comparisons provided by DoD personnel. The laboratory shall not demonstrate capability by "benchmarking against itself" using internal comparisons to initial runs.

**37**

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

**Capability – Initial and Continuing:** The initial and continuing demonstration of capability shall include verification of method sensitivity checks (for example, through the use of quarterly method detection verification) and demonstrated measurements of accuracy and precision (such as the production and review of quality control charts). These requirements apply to each quality system matrix of concern.

In addition, continued proficiency (as discussed in item c below) shall, at a minimum, include annual successful completion of one of the options listed in Section 5.2.6.c.3 by each analyst.

**38**

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

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

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

**Capability – Change:** "Change" refers to any change in personnel, instrumentation, test method, or sample matrix that potentially affects the precision, accuracy, sensitivity, and selectivity of the output (for example, a change in the detector, column, matrix, or other components of the sample analytical system, or a method revision). Requirements for demonstration of capability are further addressed in Appendix C.

**39**

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

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

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

**Work Cell – Definition of Work Cell:** Additional guidance on this issue is provided in DoD clarification box C-2 in Section C.1. A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation, extraction, and analysis. To ensure that the entire preparation, extraction, and analysis process is completed by a group of capable individuals, the laboratory shall ensure that **each member** of the work cell (including a new member entering an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a "team," the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance.

A work cell may NOT be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

**40**

### 5.4.3 Laboratory-Developed Methods

The introduction of environmental test methods developed by the laboratory for its own use shall be a planned activity and shall be assigned to qualified personnel equipped with adequate resources.

Plans shall be updated as development proceeds and effective communication amongst all personnel involved shall be ensured.

### 5.4.4 Non-Standard Methods

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

### 5.4.5 Validation of Methods

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

**5.4.5.2** The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their published scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use. The minimum requirements shall be the initial test method evaluation requirements given in Appendix C.3 of this chapter.

**5.4.5.3** The range and accuracy of the values obtainable from validated methods (e.g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the clients' needs.

**Calibration Standards – Laboratory Involvement:** DoD recognizes that achievability of these limits/levels by the required method is a key variable. To avoid conflicts related to this issue, DoD expects laboratory involvement (Government or private) during the planning phase of the project (QAPP preparation) to ensure proper selection of methods and instrumentation. If the proposed laboratory for the project is unavailable for this consultation (for example, one has not yet been selected), a Government laboratory may be consulted to establish these parameters. This early involvement of a laboratory is integral in ensuring efficient planning and implementation of the project.

**41**

### 5.4.6 Estimation of Uncertainty of Measurement

**5.4.6.1** Environmental testing laboratories shall have and shall apply procedures for estimating uncertainty of measurement. In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid calculation of uncertainty of measurement. In these cases the laboratory shall at least attempt to identify all the components of uncertainty and make a reasonable estimation, and shall ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

In those cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions (see 5.10).

**5.4.6.2** When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

### 5.4.7 Control of Data

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

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

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

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

**Data Verification Procedures:** Data verification (review) shall consist of at least the following procedures:

1. Determinations of whether the results of testing, examining, or analyzing the sample meet the laboratory's requirements for interpretation, precision, and accuracy.
2. Checks to determine accuracy of calculations, conversions, and data transfers.
3. Checks for transcription errors, omissions, and mistakes.
4. Checks to determine consistency with project-specific measurement quality objectives (MQOs).
5. Checks to ensure that the appropriate preparatory and analytical SOPs and standardized methods were followed, and that chain-of-custody (COC) and holding time requirements were met.
6. Checks to ensure that requirements for calibration and calibration verification standards were met, and that QC samples (for example, method blanks, laboratory control samples (LCSs)) met criteria for precision, accuracy, and sensitivity.
7. Accurate explanation in the case narrative of any anomalous results and any corrective actions taken, and all data flags checked for appropriate and accurate use.
8. A tiered or sequential system of verification, consisting of at least three levels, with each successive check performed by a different person. This three-tiered approach should include (at a minimum) 100% review by the analyst, 100% verification review by a technically qualified supervisor or data review specialist, and a final administrative review. The final administrative review will verify that previous reviews were documented properly and that the data package is complete.

Additionally, as part of its internal quality assurance program, the quality manager, or designee, shall review at a minimum 10% of all data packages for technical completeness and accuracy. This review is part of the oversight program and does not have to be completed in "real time."

**42**

**5.4.7.2** When computers, automated equipment, or microprocessors are used for the acquisition, processing, recording, reporting, storage or retrieval of environmental test data, the laboratory shall ensure that:

**Electronic Data:** The following applies to audit trails as well as to test data.

In addition to meeting all requirements of this standard, DoD requires that laboratories employing electronic data processing equipment put in place a quality system for such activities that is consistent with the language in Sections 8.1 through 8.11 of the EPA document "2185 – Good Automated Laboratory Practices" (1995). The quality system shall address the following elements: laboratory management, personnel, quality assurance unit, LIMS raw data, software, security, hardware, comprehensive testing, records retention, facilities, and standard operating procedures. This quality system shall be documented in the laboratory's quality manual and appropriate SOPs.

**43**

a) computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use;

b) procedures are established and implemented for protecting the data; such procedures shall include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;

**Data – Automated Processes:** At a minimum, for those processes that are automated, a sample data test set shall be used to test and verify the correct operation of these data reduction procedures (including data capture, manipulation, transfer, and reporting). This shall be done anytime new software is purchased or the programming code is modified or otherwise manipulated, and applies even in cases where commercial software is used as part of the process.

**44**

c) computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data; and

d) it establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

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

## 5.5 Equipment

**Equipment Standards:** Equipment shall be capable of achieving the accuracy, precision, sensitivity, and selectivity required for the intended use of the generated data. The laboratory shall implement documented procedures to ensure that setup, maintenance, and adjustments to instrument operating parameters are documented, and that adjustments to instruments do not exceed the limits specified in the approved SOPs.

**45**

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

**5.5.2** Equipment and its software used for testing and sampling shall be capable of achieving the accuracy required and shall comply with specifications relevant to the environmental tests concerned. Before being placed into service, equipment (including that used for sampling) shall be calibrated or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications.

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

#### 5.5.2.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume.

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

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

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

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

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

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST traceable references where commercially available. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

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

**Volumetric Pipettes – Acceptance Criteria:** As listed in the table in DoD clarification box 48, mechanical pipettes shall be within 3% of known or true value (1 or 2% is recommended).

**46**

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

**Autoclaves:** The use of autoclaves during chemical tests is not typical, but it is an analytical option for limited methods (for example, mercury soil digestion). The typical use would be for sterilization purposes as described in item g below.

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g) For biological tests that employ autoclave sterilization see section D.3.8.

**Calibration – Calibration and Measurement Guidance:** The following table provides specific guidance with respect to the calibration and performance measurements associated with specific types of analytical support equipment. The criteria presented that go beyond those established by the American Society for Testing and Methods (ASTM) standards are currently in use by DoD components. They are presented here in consolidated form and will be formally adopted across DoD as a standardized requirement. The ASTM standards presented here are based on the latest edition available at the time of this manual's publication. As new editions are released, the latest revision of each ASTM standard shall be followed, unless State or project requirements differ.

**48**

| Analytical Support Equipment Assessment | Frequency of Check | Acceptance Criteria | Calibration Check Procedures and Performance Criteria References (latest edition) |
|---|---|---|---|
| Balance calibration check | Daily or before use with two weights that bracket target weight(s)<br><br>AND<br><br>Annual servicing by certified technician | Analytical balances: ±0.1% or ±0.5mg, whichever is larger, unless method-specific guidance exists.<br><br>Top-loading balances: see ASTM D 4753. | ASTM E 898, Standard Method of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances; ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid; ASTM D4753, Standard Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses |
| Standard weights | Every 5 years | Third-party certificate of acceptance | ASTM E 617, Standard Specification for Laboratory Weights and Precision Mass Standards and ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid. |
| Refrigerator/freezer temperature monitoring | Daily | Refrigerators: 4 ± 2 °C<br>Freezers: -10 to -20 °C<br><br>(This ASTM standard does not address freezers, but SW-846 has noted this freezer range in some methods.) | ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid |
| Thermometer calibration check | Liquid in glass – when received and annually<br>Electronic – quarterly at two temperatures that bracket target temperature(s) against a NIST-traceable thermometer; if only a single temperature is used, at the temperature of use | Appropriate correction factors applied or thermometer replaced | ASTM E 77, Standard Test Method for Inspection and Verification of Thermometers, and ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid<br><br>**(continued on next page)** |

| Analytical Support Equipment Assessment | Frequency of Check | Acceptance Criteria | Calibration Check Procedures and Performance Criteria References (latest edition) |
|---|---|---|---|
| Class B volumetric glassware | By lot at the time of receipt and when there is evidence of deterioration | 3% of known or true value (1 or 2% is recommended) | ASTM E 542, Standard Practice for Calibration of Volumetric Apparatus and ASTM E 969, Standard Specification for Glass Volumetric (Transfer) Pipets |
| Mechanical volumetric pipettes | Quarterly | Same as Class B volumetric glassware criteria | |
| Nonvolumetric glassware/labware verification<br><br>(Requirement is applicable only when used for measuring initial sample and final extract/digestate volumes.) | By lot at the time of receipt | 3% of known or true value. (Standard tolerance does not exist. Class B volumetric flasks are criteria between 0.8 and 0.05% for 5 mL to 2,000 mL, respectively – set at 3% to maintain consistency with pipette tolerance designation.) | ASTM E 542, Standard Practice for Calibration of Volumetric Apparatus |
| Drying ovens | Before and after use; for moisture content analysis, before use only | Compliance with method-specific requirements or within ± 5% of set temperature | ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid, and ASTM E 145, Standard Specification for Gravity-Convection and Forced-Ventilation Ovens<br>**48** |

#### 5.5.2.2 Instrument Calibration

This standard specifies the essential elements that shall define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data must be of known quality and be appropriate for a given regulation or decision. This standard does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are to be followed.

**Calibration – Instrument:** The DoD implementation clarification boxes included in Section 5.5. specify whether they apply only when method-specific guidance does not exist or whether they apply to all methods.

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##### 5.5.2.2.1 Initial Instrument Calibration

The following items are essential elements of initial instrument calibration:

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

b) Sufficient raw data records must be retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, analyst's initials or signature; concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration.

**Calibration (Initial) – Raw Data Records:** When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of the manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and signature/initials of person performing manual operation (electronic signature is acceptable).

**Applicable to all methods.**

**50**

c) Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification, unless otherwise required by regulation, method or program.

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

**Calibration – Second Source Standards:** The use of a standard from a second lot is acceptable when only one manufacturer of the calibration standard exists. Note: "manufacturer" refers to the producer of the standard, not the vendor.

The requirement for a second source standard for the initial calibration verification is waived if a second source standard is used for the continuing calibration verification. Deviations from this requirement require project-specific approval from appropriate DoD personnel (for example, project manager, quality manager).

The date of preparation of each standard shall be considered when evaluating its suitability for use. This consideration shall include an assessment of the stability of the standard solution, as well as its degradation rate.

The concentration of the second source standard shall be at or near the middle of the calibration range. Criteria for the acceptance of results from second source verification standards shall be established. Values chosen should be at least as stringent as those established for the continuing instrument calibration verification. The initial calibration verification shall be successfully completed prior to running any samples.

**Applicable only when method-specific guidance does not exist.**

**51**

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

**Calibration – Initial Calibration Points:** Criteria for the acceptance of an initial instrument calibration must be established (for example, correlation coefficient, relative standard deviation).

Exclusion of initial calibration points without technical justification is not allowed.

For example, in establishing an initial calibration curve, the calibration points used shall be a contiguous subset of the original set. In addition, the minimum linearity of the curve shall be determined either by a linear regression correlation coefficient greater than or equal to 0.995 or by a maximum percent relative standard deviation (%RSD) of 20% for each analyte.

Deviations from the above, including for problem compounds, are permitted with the approval of DoD personnel (for example, project manager, quality manager). See DoD clarification box 54 for guidance on the number of points required for a calibration curve.

**Applicable only when method-specific guidance does not exist.**

**52**

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

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

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

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

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

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

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

**Calibration – Quantitative Values in a Calibration Curve:**

The range of the accepted initial calibration curve reflects the quantitation range of the samples (i.e., only those sample results with concentrations contained within the range of the calibration curve are considered to be quantitative). Any data reported outside the calibration range shall be qualified as an estimated value (i.e., by a data qualifier "flag") and explained in the case narrative.

When sample concentrations **exceed** the upper limit of the calibration curve, samples shall be diluted and reanalyzed (if possible) to bring them within the calibration curve. When sample concentrations **exceed** the upper limit of the calibration curve or **fall below** the lower limit of the calibration curve, the resulting data shall be qualified as having estimated values and shall be flagged.

The **reporting limit** shall lie within the calibration range, at or above the LOQ. If the **client requires** a reporting limit that lies **below** the lowest standard of the calibration curve and below the LOQ, then method modification is required. For methods that require only one standard (i.e., lower limit of curve is the origin), the reporting limit shall be no lower than a low-level check standard, designed to verify the integrity of the curve at the lower limits.

See also DoD clarification box D-18, which addresses limits of detection, as well as definitions for limit of quantitation and reporting limit.

**Applicable only when method-specific guidance does not exist.**

**53**

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

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

**Calibration – Initial Calibration:** In completing work for DoD, the initial calibration range shall consist of a minimum of 5 contiguous calibration points for organics and a minimum of 3 contiguous calibration points for inorganics. All reported target analytes and surrogates shall be included in the initial calibration. For multicomponent analytes, such as PCBs, toxaphene, and dioxins/furans, a separate initial calibration may be required. See DoD clarification box 52 in Section 5.5.2.2.1.e and Appendix DoD-B for additional implementation requirements pertaining to this subject.

**Applicable when method-specific guidance does not exist.**

**54**

**5.5.3** Equipment shall be operated by authorized personnel. Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) shall be readily available for use by the appropriate laboratory personnel.

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

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

**5.5.5** The laboratory shall maintain records of each major item of equipment and its software significant to the environmental tests performed. The records shall include at least the following:

a) the identity of the item of equipment and its software;

b) the manufacturer's name, type identification, and serial number or other unique identification;

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

d) the current location;

e) the manufacturer's instructions, if available, or reference to their location;

f) dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;

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

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

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

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

**5.5.6** The laboratory shall have procedures for safe handling, transport, storage, use and planned maintenance of measuring equipment to ensure proper functioning and in order to prevent contamination or deterioration.

**5.5.7** Equipment that has been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits, shall be taken out of service. It shall be isolated to prevent its use or clearly labeled or marked as being out of service, until it has been repaired and shown by calibration or test to perform correctly. The laboratory shall examine the effect of the defect or departure from specified limits on previous environmental tests and shall institute the "Control of nonconforming work" procedure (see 4.9).

**5.5.8** Whenever practicable, all equipment under the control of the laboratory and requiring calibration shall be labeled, coded or otherwise identified to indicate the status of calibration, including the date when last calibrated and the date or expiration criteria when recalibration is due.

**5.5.9** When, for whatever reason, equipment goes outside the direct control of the laboratory, the laboratory shall ensure that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

**5.5.10** When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

**Calibration – Continuing Instrument Calibration Verification:** The validity of the initial calibration shall be verified prior to sample analysis by an **acceptable** continuing instrument calibration verification with each analytical batch. As long as the continuing calibration verification (CCV) is acceptable, a new initial instrument calibration is not necessary.

**Applicable when method-specific guidance does not exist.**

**55**

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

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

c) Instrument calibration verification must be performed:

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

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

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

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

**Calibration – Continuing Calibration Verification Frequency:** When the methods specify that CCVs shall be run at specific sample intervals (for example, every 10 samples), the count of these samples shall be of field samples only. However, QC samples must be run with their associated batches. The grouping of QC samples from a variety of batches is not an acceptable practice. If the method does not specify an interval for periodic CCVs, at a minimum, every analytical batch should be bracketed (i.e., at least every 20 field samples). More frequent CCVs are recommended for more difficult matrices.

**Applicable to all methods.**

**56**

d) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations. Continuing calibration verification records must explicitly connect the continuing verification data to the initial instrument calibration.

**Calibration (Continuing) – Raw Data Records:** Raw data records shall also include the analyst's name.

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of the manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and signature/initials of person performing manual operation.

**Applicable to all methods.**

**57**

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

**Calibration – CCV Criteria:**

- The CCV standards shall be at or below the middle of the calibration range.
- The source of the standard(s) for analysis can be the standard(s) used for the initial calibration.
- The baseline for comparison for the CCV is the initial calibration (and the original standards). Specific criteria for evaluation of success or failure of the CCV may include percent difference/drift from the mean response factor established for the initial calibration, minimum response factor checks, and confirmation that the retention time is within an acceptable window.

**Applicable when method-specific guidance does not exist.**

**58**

If the continuing calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial instrument calibration must be performed.

**Calibration – Reporting Data from Noncompliant CCV:** If initial corrective action attempts fail and the CCV results are still outside established acceptance criteria, and the laboratory chooses to demonstrate the success of routine corrective action through the use of two consecutive CCVs, then the concentrations of the two CCVs must be at two different levels within the original calibration curve. As stated in DoD clarification box 58, at least one of the CCV standards shall fall below the middle of the initial calibration range.

**Applicable to all methods.**

**59**

If the laboratory has not verified calibration, sample analyses may not occur until the analytical system is calibrated or calibration verified. If samples are analyzed using a system on which the calibration has not yet been verified the results shall be flagged. Data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

1) when the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

2) when the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

**Calibration – Q Flag Reporting for Noncompliant CCV:** Project-specific permission from appropriate DoD personnel is required to report data generated from the initial run with the noncompliant CCV. If this permission is granted, and these data are reported, they shall be qualified through the use of a "Q" flag and explained in the case narrative.

**Applicable to all methods.**

**60**

**5.5.11** Where calibrations give rise to a set of correction factors, the laboratory shall have procedures to ensure that copies (e.g., in computer software) are correctly updated.

**5.5.12** Test equipment, including both hardware and software, shall be safeguarded from adjustments which would invalidate the test results.

## 5.6 Measurement Traceability

### 5.6.1 General

All equipment used for environmental tests, including equipment for subsidiary measurements (e.g., for environmental conditions) having a significant effect on the accuracy or validity of the result of the environmental test or sampling shall be calibrated before being put into service and on a continuing basis. The laboratory shall have an established program and procedure for the calibration of its equipment. This includes balances, thermometers, and control standards. Such a program shall include a system for selecting, using, calibrating, checking, controlling and maintaining measurement standards, reference materials used as measurement standards, and measuring and test equipment used to perform environmental tests.

### 5.6.2 Testing Laboratories

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

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

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

### 5.6.3 Reference Standards and Reference Materials

#### 5.6.3.1 Reference Standards

The laboratory shall have a program and procedure for the calibration of its reference standards. Reference standards shall be calibrated by a body that can provide traceability as described in 5.6.2.1. Such reference standards of measurement held by the laboratory (such as class S or equivalent weights or traceable thermometers) shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated. Reference standards shall be calibrated before and after any adjustment. Where commercially available, this traceability shall be to a national standard of measurement.

#### 5.6.3.2 Reference Materials

Reference materials shall, where commercially available, be traceable to SI units of measurement, or to certified reference materials. Where possible, traceability shall be to national or international standards of measurement, or to national or international standard reference materials. Internal reference materials shall be checked as far as is technically and economically practicable.

#### 5.6.3.3 Intermediate Checks

Checks needed to maintain confidence in the status of reference, primary, transfer or working standards and reference materials shall be carried out according to defined procedures and schedules.

#### 5.6.3.4 Transport and Storage

The laboratory shall have procedures for safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity.

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

Documented procedures shall exist for the purchase, reception and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory shall retain records for all standards, reagents, reference materials and media including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material shall not be used unless its reliability is verified by the laboratory.

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

c) Records shall be maintained on standard and reference material preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

**Documentation – Lot Number:** The records shall include appropriate lot numbers for the standard. **61**

d) All containers of prepared standards and reference materials must bear a unique identifier and expiration date and be linked to the documentation requirements in 5.6.4.c above.

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

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

## 5.7 Sampling

**5.7.1** The laboratory shall have a sampling plan and procedures for sampling when it carries out sampling of substances, materials or products for subsequent environmental testing. The sampling plan as well as the sampling procedure shall be available at the location where sampling is undertaken. Sampling plans shall, whenever reasonable, be based on appropriate statistical methods. The sampling process shall address the factors to be controlled to ensure the validity of the environmental test results.

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

**Sampling Procedures:** Sampling procedures shall also address laboratory practices for the handling, subsampling, and documenting of extraneous materials (for example, rocks, twigs, vegetation) present in samples. The handling of multiphase samples shall be addressed in specific sampling procedures, as appropriate. The laboratory's subsampling procedures shall be compliant with ASTM D 6323, Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities, and EPA's Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples (EPA/600/R-03/027). Additionally, the laboratory shall use other recognized consensus standards (for example, ASTM standards) where available for these procedures.

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**5.7.2** Where the client requires deviations, additions or exclusions from the documented sampling procedure, these shall be recorded in detail with the appropriate sampling data and shall be included in all documents containing environmental test results, and shall be communicated to the appropriate personnel.

**5.7.3** The laboratory shall have procedures for recording relevant data and operations relating to sampling that forms part of the environmental testing that is undertaken. These records shall include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and diagrams or other equivalent means to identify the sampling location as necessary and, if appropriate, the statistics the sampling procedures are based upon.

### 5.8 Handling of Samples

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

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

**5.8.2** The laboratory shall have a system for identifying samples. The identification shall be retained throughout the life of the sample in the laboratory. The system shall be designed and operated so as to ensure that samples cannot be confused physically or when referred to in records or other documents. The system shall, if appropriate, accommodate a sub-division of groups of samples and the transfer of samples within and from the laboratory.

a) The laboratory shall have a documented system for uniquely identifying the samples to be tested, to ensure that there can be no confusion regarding the identity of such samples at any time. This system shall include identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code to each sample container received in the laboratory. The use of container shape, size or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.

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

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

d) The laboratory ID code shall be entered into the laboratory records (see 5.8.3.1.d) and shall be the link that associates the sample with related laboratory activities such as sample preparation.

e) In cases where the sample collector and analyst are the same individual, or the laboratory preassigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

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

**5.8.3.1 Sample Receipt Protocols**

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

1) All samples which require thermal preservation shall be considered acceptable if the arrival temperature is either within 2°C of the required temperature or the method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable. Samples that are hand delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

**Sampling – Temperature Measurements:** The temperature measurement, when applicable, shall be verified through the use of a temperature blank (for each transport container, such as a cooler) or other measurement when a temperature blank is not available (for example, an IR gun).
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2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or chlorine, prior to or during sample preparation or analysis.

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

i. sufficient sodium thiosulfate is added to each container to neutralize at minimum 5 mg/l of chlorine for drinking water and 15mg/l of chlorine for wastewater samples;

ii. one container from each batch of laboratory prepared containers or lot of purchased ready-to-use containers is checked to ensure efficacy of the sodium thiosulfate to 5 mg/l chlorine or 15mg/l chlorine as appropriate and the check is documented;

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

**Preservation of Samples – Chemical:** Procedures for checking chemical preservation using readily available techniques shall also be performed when the continued preservation of the sample is in question (due to sample interaction with the preservative), when samples cannot be checked upon receipt (for example, VOCs), and/or for samples whose preservative may have deteriorated for any other reason.
**64**

b) The results of all checks shall be recorded.

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

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

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

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

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

**Sampling – Documentation When Acceptance Criteria Are Not Met:** Additional guidance on this issue is provided in Section 5.10.2 (Test Reports).

Where there is any doubt as to the sample's suitability for testing, where the sample does not conform to the description provided, or where the test required is not fully specified, the laboratory shall consult the client for further instruction before proceeding. This consultation shall be immediate and timely (i.e., by the next business day or as specified in project plans).

**65**

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

**Electronic Databases:** Use of electronic database systems shall meet the requirements specified in Section 5.4.7.2 .

**66**

1) This sample receipt log shall record the following:

   i. client/project name,

   ii. date and time of laboratory receipt,

   iii. unique laboratory ID code (see 5.8.2), and

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

2) During the login process, the following information must be unequivocally linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request and readily available to individuals who will process the sample. Note: the placement of the laboratory ID number on the sample container is not considered a permanent record.

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

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

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

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

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

f) A complete chain of custody record form (Sections 4.12.2.5 and Appendix E), if utilized, shall be maintained.

#### 5.8.3.2 Sample Acceptance Policy

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

**Sample Acceptance:** The laboratory shall have procedures documented in the quality manual or related documentation (as discussed in Sections 4.2.3.i and 4.2.3.k) that address methods by which the laboratory confirms that it has the capability to accept new samples before such acceptance occurs. The laboratory shall also follow any additional method-specific requirements concerning sample acceptance.

**67**

a) proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

b) proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) use of appropriate sample containers;

d) adherence to specified holding times;

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

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

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

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

1) Samples which require thermal preservation shall be stored under refrigeration which is +/-2 of the specified preservation temperature unless method specific criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable.

**Preservation of Samples – Thermal:** When refrigeration or freezing is required, the laboratory shall ensure that monitoring is performed 7 days per week to ensure that the samples remain within an acceptable range. A variety of low-cost devices (for example, digital minimum/maximum thermometers with memory, circle chart thermometers) can be used to validate that the proper temperature is continuously maintained.

**68**

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

**Samples – Cross-Contamination:** The laboratory shall have procedures in place to ensure that cross-contamination does not occur. Samples designated for volatile organics testing shall be segregated from other samples. Samples suspected of containing high levels of volatile organics shall be further isolated from other volatile organics samples, or storage blanks shall be used to verify that no cross-contamination has occurred.

**69**

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

**Samples – Disposal of Records:** The laboratory shall maintain appropriate documentation and records demonstrating that samples have been properly disposed of, in accordance with Federal, State, and local regulations.

**70**

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

## 5.9 Assuring the Quality of Environmental Test and Calibration Results

### 5.9.1 General

The laboratory shall have quality control procedures for monitoring the validity of environmental tests undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

a) regular use of certified reference materials and/or internal quality control using secondary reference materials;

b) participation in interlaboratory comparison or proficiency-testing program (see Chapter 2 of NELAC)

**Proficiency Testing Program:** Laboratories that perform work for DoD are required to participate in a PT program, as defined in NELAC Chapter 2. Refer to the complete chapter and appendices for additional explanation and the NELAC website for current lists of fields of proficiency testing, PT Providers, and analyte acceptance limits.

**2.0 PROFICIENCY TESTING PROGRAM: INTERIM STANDARDS**

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**2.1 INTRODUCTION, SCOPE, AND APPLICABILITY**

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**2.1.3 Fields of Proficiency Testing**

The PT program is organized by fields of proficiency testing. The following elements collectively define fields of proficiency testing:

a) matrix type,
b) technology/method, and
c) analyte/analyte group

Current NELAC fields of proficiency testing are located on the NELAC Website.

Note: Laboratories are permitted to analyze one PT sample by multiple methods for a given analyte within a technology. If a laboratory reports more than one method per technology per study, an unacceptable result for any method would be considered a failed study for that technology for that analyte.

**2.4 LABORATORY ENROLLMENT IN PROFICIENCY TESTING PROGRAM(S)**

**2.4.1 Required Level of Participation**

To be accredited initially and to maintain accreditation, a laboratory shall participate in two single-blind, single-concentration PT studies, where available, per year for each field of proficiency testing for which it seeks or wants to maintain accreditation. Laboratories performing analysis in the United States must obtain PT samples from a PTOB/PTPA-approved PT Provider. Overseas laboratories must use a PT Provider that can demonstrate compliance with ISO Guide 34:2000, ISO Guide 43:1997, and ISO/IEC 17025. Each laboratory shall participate in at least two PT studies for each field of proficiency testing per year unless a different frequency for a given program is defined in the appendices. Section 2.5 describes the time period in which a laboratory shall analyze the PT samples and report the results. Data and laboratory evaluation criteria are discussed in Sections 2.6 and 2.7 of this chapter [Chapter 2 of NELAC].

**2.7 PT CRITERIA FOR LABORATORY ACCREDITATION**

**2.7.2 Initial or Continuing PT Studies**

A laboratory seeking to obtain or maintain accreditation shall successfully complete two initial or continuing PT studies for each requested field of proficiency testing within the most recent three rounds attempted. For a laboratory seeking to obtain accreditation, the most recent three rounds attempted shall have occurred within 18 months of the laboratory's accreditation date. Successful performance is described in Appendix C [of NELAC Chapter 2]. When a laboratory has been granted accreditation status, it shall continue to complete PT studies for each field of proficiency testing and maintain a history of at least two acceptable PT studies for each field of proficiency testing out of the most recent three. For initial accreditation, the laboratory must successfully analyze two sets of PT studies, the analyses to be performed at least 15 calendar days apart from the closing date of one study to the shipment date of another study for the same field of proficiency testing. For continuing accreditation, completion dates of successive proficiency rounds for a given field of proficiency testing shall be approximately six months apart. Failure to meet the semiannual schedule is regarded as a failed study.

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**2.7.4 Failed Studies and Corrective Action**

Whenever a laboratory fails a study, it shall determine the cause for the failure and take any necessary corrective action. It shall then document in its own records and provide to the Primary Accrediting Authority both the investigation and the action taken. If a laboratory fails two out of the three most recent studies for a given field of proficiency testing, its performance is considered unacceptable under the NELAC PT standard for that field. The laboratory shall then meet the requirements of initial accreditation as described in Section 2.7.2 - Initial or Continuing Accreditation.

**Appendix C - PT ACCEPTANCE CRITERIA AND PT PASS/FAIL CRITERIA**

**C.5.0 NELAC PT Study Pass/Fail Criteria**
**•••**
**C.5.3 Pass/fail Criteria For Analyte Group PT Samples**

Proficiency testing pass/fail evaluations for Analyte Group PT studies shall be determined as follows. To receive a score of "Pass", a laboratory must produce "Acceptable" results as defined in Section C.1 for 80% of the analytes in an Analyte Group PT Study. Greater than 20% "Not Acceptable" results shall result in the laboratory receiving a score of "Fail" for that group of analyte. ••• A "Not Acceptable" result for the same analyte in two out of three consecutive PT studies shall also result in the laboratory receiving a score of "Fail" for that analyte. The PCB analyte group is exempt from the 80% pass/fail criteria.

**Note:** ••• indicates text from NELAC Chapter 2 not repeated in this clarification box.

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c) replicate tests using the same or different methods;

d) retesting of retained samples;

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

**Audits – Laboratory Checks of Performance Audits:** This section requires the laboratory to continuously evaluate the quality of generated data by systematically and routinely implementing control checks that go beyond those required by the test methods. The results of these checks (examples of which are listed above) shall be routinely reviewed after they are performed in order to monitor and evaluate the quality and usability of data generated by the laboratory. Although a supplemental review of these checks shall be included as part of the annual internal audits, the laboratory shall also ensure that the results of these checks are reviewed (and corrective action taken) on a regular and timely basis following the actual completion of the check to remedy the problem, avoid its recurrence, and improve the quality system overall.

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### 5.9.2 Essential Quality Control Procedures

These general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., chemical, whole effluent toxicity, microbiological, radiological, air) and are further described in Appendix D. The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have detailed written protocols in place to monitor the following quality controls:

**Quality Control Actions:** Quality control actions should be both batch-specific and time-based (i.e., those required to be conducted at specific time periods, such as for tunes and method detection limits [MDLs]). Batch-specific quality control actions include sample-specific quality control actions such as surrogate spikes.

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

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

3) measures to assure the accuracy of the test method including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

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

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

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

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

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

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance criteria shall be used to determine the usability of the data. (See Appendix D)

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

d) The quality control protocols specified by the laboratory's method manual (5.4.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D or mandated methods or regulations (whichever are more stringent) are incorporated into their method manuals. When it is not apparent which is more stringent the QC in the mandated method or regulations is to be followed.

The essential quality control measures for testing are found in Appendix D of this Chapter.

## 5.10 Reporting the Results

### 5.10.1 General

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

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

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

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

**Reporting Requirements:** The reporting requirements for work produced for DoD are outlined in Appendix DoD-A. This appendix follows all the NELAC appendices.
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Laboratories that are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes (in-house or captive laboratories) shall have all applicable information specified in a) through m) below readily available for review by the accrediting authority. However, formal reports detailing the information are not required if:

a) the in-house laboratory is itself responsible for preparing the regulatory reports; or

b) the laboratory provides information to another individual within the organization for preparation of regulatory reports. The facility management must ensure that the appropriate report items are in the report to the regulatory authority if such information is required.

### 5.10.2 Test Reports

Each test report shall include at least the following information, unless the laboratory has valid reasons for not doing so, as indicated by 5.10.1.a and b:

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

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

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

   1) This requirement may be presented in several ways:

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

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

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

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

e) identification of the method used;

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

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

**Test Report Contents – Time of Analysis:** For DoD work, both date and **time** of analysis are considered to be essential information, regardless of the length of the holding time, and shall be included as part of the laboratory report. If the time of the sample collection is not provided, the laboratory must assume the most conservative (i.e. earliest) time of day.
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h) reference to the sampling plan and procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results;

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

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

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

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

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

### 5.10.3 Supplemental Information for Test Reports

**5.10.3.1** In addition to the requirements listed in 5.10.2, test reports shall, where necessary for the interpretation of the test results, include the following:

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

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

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

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

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

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

**5.10.3.2** In addition to the requirements listed in 5.10.2 and 5.10.3.1, test reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

a) the date of sampling;

b) unambiguous identification of the substance, material or product sampled (including the name of the manufacturer, the model or type of designation and serial numbers as appropriate);

c) the location of sampling, including any diagrams, sketches or photographs;

d) a reference to the sampling plan and procedures used;

e) details of any environmental conditions during sampling that may affect the interpretation of the test results;

f) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

### 5.10.4 Opinions and Interpretations

When opinions and interpretations are included, the laboratory shall document the basis upon which the opinions and interpretations have been made. Opinions and interpretations shall be clearly marked as such in a test report.

### 5.10.5 Environmental Testing Obtained from Subcontractors

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

### 5.10.6 Electronic Transmission of Results

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

### 5.10.7 Format of Reports

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

### 5.10.8 Amendments to Test Reports

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

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

Such amendments shall meet all the requirements of this Standard.

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

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# NELAC APPENDICES

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## APPENDIX A – REFERENCES

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## APPENDIX B – GLOSSARY

The following definitions are used in the text of Quality Systems. In writing this document, the following hierarchy of definition references were used: ISO 8402, ANSI/ASQC E-4, EPA's Quality Assurance Division Glossary of Terms, and finally definitions developed by NELAC. The source of each definition, unless otherwise identified, is the Quality Systems Committee.

**Quality Systems Definitions:** The Quality Systems Committee is the NELAC-appointed group that created and continues to modify NELAC Chapter 5 (Quality Systems). Terms not included in the NELAC Glossary, but defined by DoD, are included in gray text boxes throughout this Appendix.

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

**Accreditation:** The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

**Accrediting Authority:** The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation. (NELAC) [1.4.2.3]

**Accuracy:** The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

**Aliquot:** A discrete, measured, representative portion of a sample taken for analysis. (DoD, EPA QAD Glossary)

**Analyst:** The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

**Analyte:** The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

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

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

**Batch:** Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

**Blank:** A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

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

**Calibration:** Set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (VIM: 6.11)

1) In calibration of support equipment the values realized by standards are established through the use of Reference Standards that are traceable to the International System of Units (SI).
2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

**Calibration Curve:** The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

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

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

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

**Chain of Custody Form:** A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC)

**Chemical:** Any element, compound, or mixture of elements and/or compounds. Frequently, chemical substances are classified by the CAS rules of nomenclature for the purposes of identification for a hazard evaluation. (OSHA Glossary)

**Client:** The party that has agreed to pay the bill for services rendered by the laboratory, and with whom the laboratory has a contractual relationship for that project. For a laboratory, this is typically the prime contractor who originally hires the laboratory for the project, and who signs the contract as the receiver of services and resulting data. In cases where the laboratory has a direct contractual relationship with DoD, the client shall be the Government's authorized contracting officer. The contracting officer, as the client, shall consult with the Government's authorized technical representative when dealing with laboratory technical issues. It is understood that typically other "Clients" are present at other levels of the project, but they may be removed from the day-to-day decision-making (for example, installation representatives, service center representatives, various other Government officials). Specific circumstances may require the direct notification of these other clients, in addition to the prime contractor or DoD representative; these circumstances shall be included as part of specific project requirements. (DoD)

**Compound:** A unique combination of chemical elements, existing in combination to form a single chemical entity. (DoD)

**Confirmation:** Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors; or
- Additional cleanup procedures. (NELAC)

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

**Consensus Standards:** A protocol established by a recognized authority (for example, American Society for Testing and Materials [ASTM], American National Standards Institute [ANSI], or the Institute for Electrical and Electronic Engineers [IEEE]).

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

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

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

**Definitive Data:** Data that are generated using rigorous analytical methods, such as approved EPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data in the form of paper printouts or electronic files. Data shall satisfy QA/QC requirements. For data to be definitive, either analytical or total measurement error shall be determined and documented. (Data Quality Objectives Process for Superfund)

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

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

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

**Environmental Program:** An organized effort that assesses environmental concerns and leads to the collection of data, either in the field or through laboratory analysis. (Variation on EPA QAD Glossary for Terms: Environmentally related measurement, environmental sample)

**Finding:** An assessment conclusion, referenced to a NELAC Standard and supported by objective evidence that identifies a deviation from a NELAC requirement.

**Holding Times (Maximum Allowable Holding Times):** The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR Part 136)

**Holding Times (DoD Clarification):** The time elapsed from the time of sampling to the time of extraction or analysis, as appropriate.

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

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

**International System of Units (SI):** The coherent system of units adopted and recommended by the General Conference on Weights and Measures. (CCGPM) (VIM 1.12)

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

**Key Staff:** At a minimum, the following managerial and supervisory staff (however named) – executive staff (for example, Chief Executive Officer, Chief Operating Officer, laboratory director, technical director); technical directors/supervisors (for example, section supervisors for organics and inorganics); quality assurance systems directors/supervisors (for example, quality manager, quality auditors); and support systems directors/supervisors (for example, information systems supervisor, purchasing director, project manager).

**Laboratory:** A body that calibrates and/or tests. (ISO 25)

**Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):** A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC).

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

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

**Limits of Quantitation (LOQ):** The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

**Limit of Quantitation (DoD Clarification):** For DoD, the lowest standard of the calibration establishes the LOQ, but it must be greater than or equal to 3 times the LOD.

**Manager (however named):** The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

**Matrix:** The substrate of a test sample

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

- Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- Non-Potable Water: Any aqueous sample excluded from the definition of Drinking Water matrix. Includes surface water, groundwater, effluents, water treatment chemicals, and TCLP or other extracts.
- Solid and Chemical Materials: Includes soils, sediments, sludges, products and by-products of an industrial process that results in a matrix not previously defined.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device. (NELAC)

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

- Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
- Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous Liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges and other matrices with > 15% settleable solids.
- Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
- Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter or other device. (NELAC)

**Matrix Spike (spiked sample or fortified sample):** A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)

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

**May:** Denotes permitted action, but not required action. (NELAC)

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

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

**Method:** 1. See Test Method. 2. Logical sequence of operations, described generically, used in the performance of measurements. (VIM 2.4)

**Method Blank:** A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

**Method Detection Limit:** One way to establish a Limit of Detection, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

**Must:** Denotes a requirement that must be met. (Random House College Dictionary)

**National Accreditation Database:** The publicly accessible database listing the accreditation status of all laboratories participating in NELAP. (NELAC)

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

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

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

**Nonconformance:** An indication or judgment that a product or service has not met the requirements of the relevant specifications, contract or regulation; also the state of failing to meet the requirements.

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

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

**Precision:** The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

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

**Proficiency Testing:** A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

**Proficiency Testing Oversight Body/Proficiency Testing Provider Accreditor (PTOB/PTPA):** An organization with technical expertise, administrative capacity and financial resources sufficient to implement and operate a national program of PT provider evaluation and oversight that meets the responsibilities and requirements established by NELAC standards. (NELAC)

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

**Proficiency Testing Study Provider:** Any person, private party, or government entity that meets stringent criteria to produce and distribute NELAC PT samples, evaluate study results against published performance criteria and report the results to the laboratories, primary accrediting authorities, PTOB/PTPA, and NELAP. (NELAC)

**Proficiency Test Sample (PT):** A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

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

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

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

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

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

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

**Quality System:** A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-4 1994)

**Quantitation Range:** DoD is concerned with both the upper and lower limits of quantitation. The quantitation range is defined by the low and high calibration standards.

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

**Reagent Blank (method reagent blank):** A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all

subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

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

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

**Reference Toxicant:** The toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results (see Appendix D, Section 2.1.f). (NELAC)

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

**Reporting Limit:** A data value specified by the client based on sensitivity requirements from project-specific action levels. If initially set by the client below the laboratory's LOQ, method modification is required or the client will be required to accept the laboratory's LOQ as the lowest technically valid value that can be provided by the laboratory. For methods that require only one standard and a blank, a low-level check standard shall be required to establish the LOQ. The reporting limit shall be no lower than this value. Note: There may be numbers reported to the client that are below the reporting limit. These numbers must be flagged appropriately. When the analysis demonstrates a non-detect at the LOD, the data shall be flagged with a "U." The value reported to the client is the LOD, adjusted by any dilution factor used in the analysis. When an analyte is detected between the LOQ and the LOD, the data shall be flagged with a "J." The value reported is an estimation.

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

**Sample:** Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.

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

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

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

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

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

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

**Standard Method:** A test method issued by an organization generally recognized as competent to do so.

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

**Standardized Reference Material (SRM):** A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

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

**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. (QAMS)

**Target Analytes:** 1) Analytes specifically named by a client (also called project-specific analytes) or 2) if no project-specific analytes are provided, the target analytes will be the list found in Appendix DoD-C.

**Technical Director:** Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

**Test:** A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

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

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

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

**Tolerance Chart:** A chart in which the plotted quality control data is assessed via a tolerance level (e.g., +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g., +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)

**Traceability:** The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

**Tune:** An injected standard required by the method as a check on instrument performance for mass spectrometry.

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

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

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

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

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

**Sources:**

American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996

American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991

ANSI/ASQC E4, 1994

ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for Radiobioassay Laboratories

International Standards Organization (ISO) Guides 2, 30, 8402

International Vocabulary of Basic and General Terms in Metrology (VIM): 1984. Issued by BIPM, IEC, ISO and OIML

National Institute of Standards and Technology (NIST)

National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards

Random House College Dictionary

U.S. EPA Quality Assurance Management Section (QAMS), Glossary of Terms of Quality Assurance Terms, 8/31/92 and 12/6/95

U.S. EPA Quality Assurance Division (QAD)

40 CFR Part 136

Webster's New World Dictionary of the American Language

# APPENDIX C – DEMONSTRATION OF CAPABILITY

## C.1 PROCEDURE FOR DEMONSTRATION OF CAPABILITY

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

**Capability – Change:** "Change" refers to any change in personnel, instrumentation, test method, or sample matrix that potentially impacts the precision, accuracy, sensitivity, and selectivity of the output (for example, a change in the detector, column, or other components of the sample analytical system, or a method revision). All new analysts, regardless of experience on that instrument in another laboratory, shall complete a demonstration of capability.

**C-1**

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

**Work Cell – Individual Demonstration of Capability:** To ensure that the entire preparation, extraction, and analysis process is completed by a group of capable individuals, the laboratory shall ensure that **each member** of the work cell (including a new member entering an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. The DOC will be as described for continued proficiency in Section 5.2.6.c.3.

It is not the intent of DoD to require each combination/permutation of work cell members to demonstrate group capability since DOC is for the individual only. Even though the work cell operates as a "team," the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance. For example, if multiple individuals contribute to a singe analytical result (e.g., perform preparation, extraction, and analysis) and that result meets appropriate acceptance criteria, then all individuals have demonstrated capability.

A work cell may NOT be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270), represented by one analyst who has demonstrated capability for that step. DoD assumes the work cell has demonstrated capability when each individual in the work cell has demonstrated capability for his/her area of responsibility.

**C-2**

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

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

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

The following steps shall be performed if required by mandatory test method or regulation. It is the responsibility of the laboratory to document that other approaches to DOC are adequate, this shall be

documented in the laboratory's Quality Manual, e.g., for Whole Effluent Toxicity Testing see section D.2.1.a.1.

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

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

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

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

**Capability – New Methods Evaluation:** In the case where the laboratory is introducing a new method, these criteria shall be determined using an external source of information when available (for example, the published method). If there is no external source of information, the laboratory shall use comparisons provided by DoD personnel. The laboratory shall not "benchmark against itself" by using internal comparisons to initial runs to establish these criteria.

**C-3**

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

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

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

   2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

## C.2 CERTIFICATION STATEMENT

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

**Capability – Certification Statement:** All repeated incidences of testing to meet a demonstration of capability shall be documented and packaged with the final certification statement.

**C-4**

**Demonstration of Capability**
**Certification Statement**

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

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

We, the undersigned, CERTIFY that:

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

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

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

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

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

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

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

(1) True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

## C.3 INITIAL TEST METHOD EVALUATION

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

**QC Requirements for Laboratory Developed or Non-Standard Methods:** As part of method development, and to ensure continuous quality of data, the laboratory must propose standard QC requirements consistent with similar methods or technology (see Appendix DoD-B). At a minimum these QC requirements should address:

- Calibration(s),
- Contamination,
- Precision and bias,
- Interference (selectivity), and
- Analyte identification.

Acceptance of a laboratory developed or non-standard method requires approval by DoD personnel.

**C-5**

### C.3.1 Limit of Detection (LOD)

a) The laboratory shall determine the LOD for the method for each target analyte of concern in the quality system matrices. All sample-processing steps of the analytical method shall be included in the determination of the LOD.

b) The validity of the LOD shall be confirmed by qualitative identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests and 1-4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data.

c) An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature, or, when test results are not to be reported to the LOD (versus the limit of quantitation or working range of instrument calibration), according to Appendices D.1.2, D.4.5, D.5.4, and D.6.6. Where an LOD study is not performed, the laboratory may not report a value below the Limit of Quantitation.

**Verification of LOD:** In verifying the LOD, all requisite requirements for analyte detection must be met, for example, ion abundance, second column confirmation, or pattern recognition for multicomponent analytes.

**C-6**

### C.3.2 Limit of Quantitation (LOQ)

a) The laboratory shall determine the LOQ for each analyte of concern according to a defined, documented procedure.

b) The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH).

c) The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. A successful

analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objectives for accuracy. This single analysis is not required if the bias and precision of the measurement system is evaluated at the LOQ.

**Validation of the LOQ**: The LOQ must not be set any lower than the low-level calibration standard for multipoint calibration or no lower than a low-level calibration check sample for single point calibration.
**C-7**

### C.3.3 Evaluation of Precision and Bias

a) Standard methods – The laboratory shall evaluate the precision and bias of a standard method for each analyte of concern for each quality system matrix according to the single-concentration four-replicate recovery study procedures in Appendix C.1 above (or alternate procedure documented in the quality manual when the analyte cannot be spiked into the sample matrix and QC samples are not commercially available).

b) Non-standard methods – For laboratory-developed test methods or non-standard test methods as defined at 5.4.3 and 5.4.4 that were not in use by the laboratory before July 2003, the laboratory must have a documented procedure to evaluate precision and bias. The laboratory must also compare results of the precision and bias measurements with criteria established by the client, by criteria given in the reference method or criteria established by the laboratory.

Precision and bias measurements must evaluate the method across the analytical calibration range of the method. The laboratory must evaluate precision and bias in the relevant quality system matrices and must process the samples through the entire measurement system for each analyte of interest.

**Precision and Bias:** The mean percent recovery and standard deviation for the LCS for non-standard methods must be calculated and compared to the published DoD LCS mean percent recovery and standard deviation (Appendix DoD-D). If the laboratory generates LCS data for analytes not found in the DoD appendix, the in-house laboratory–generated limits should be used. In either case, the calculated mean and standard deviation must be at least as good as the DoD published limits, where they exist, or as good or better than the published limits for similar methods or technologies. In no case should the lower LCS control limit be less than 10%.
**C-8**

Examples of a systematic approach to evaluate precision and bias could be the following:

Analyze QC samples in triplicate containing the analytes of concern at or near the limit of quantitation, at the upper-range of the calibration (upper 20%) and at a mid-range concentration. Process these samples on different days as three sets of samples through the entire measurement system for each analyte of interest. Each day one QC sample at each concentration is analyzed. A separate method blank shall be subjected to the analytical method along with the QC samples on each of the three days. (Note that the three samples at the LOQ concentration can demonstrate sensitivity as well.) For each analyte, calculate the mean recovery for each day, for each level over days, and for all nine samples. Calculate the relative standard deviation for each of the separate means obtained. Compare the standard deviations for the different days and the standard deviations for the different concentrations. If the different standard deviations are all statistically insignificant (e.g., F-test), then compare the overall mean and standard deviation with the established criteria from above.

**New Matrix:** Prior to initial analysis of a new or unknown sample matrix, a minimum of 3 MS/MSD samples in said matrix must be analyzed. The spike concentration should be within a range of 1-4 times the estimated concentration of the environmental samples, if known, otherwise, at the regulatory limit or mid-point of the calibration range, whichever is lower. The percent mean recoveries and standard deviations for each analyte recovered in the new matrix must be compared to the DoD LCS means and control limits generated for clean matrices and should be at least as good as those published in Appendix DoD-D.

**C-9**

A validation protocol such as the Tier I, Tier II, and Tier III requirements in US EPA Office of Water's Alternate Test Procedure (ATP) approval process.

### C.3.4 Evaluation of Selectivity

The laboratory shall evaluate selectivity by following the checks established within the method, which may include mass spectral tuning, second column confirmation, ICP inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, and electrode response factors.

**Selectivity for Non-Standard Methods:** When a historic selectivity check has not been identified, use the most common selectivity check for a similar method or what is most typically used for the specific instrument or technology.

**C-10**

# APPENDIX D – ESSENTIAL QUALITY CONTROL REQUIREMENTS

**DoD Quality Control Requirements:** Appendix DoD-B contains tables that consolidate DoD data quality requirements that apply to EPA's *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (SW-846). In addition, introductory material identifies definitions of QC checks and clarifies DoD's interpretation of method requirements. This appendix follows all the NELAC appendices.

**D-1**

The quality control protocols specified by the laboratory's method manual (5.4.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals and/or the Laboratory Quality Manual.

All quality control measures shall be assessed and evaluated on an on-going basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter 5, e.g., 5.9.2, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing, W.E.T. testing, microbiology testing, radiochemical testing and air testing.

**Quality Control – Corrective Action:** When quality control measures fail the acceptance criteria specified in these requirements, corrective action shall be taken. Different corrective responses may be appropriate in different situations, based on project-specific requirements and the magnitude of the problem. Examples of corrective actions include:

- Determining the source of the problem,
- Notifying the client,
- Reprocessing samples,
- Using data qualifiers to "flag" data, and
- Adding commentary in laboratory reports.

**D-2**

## D.1 CHEMICAL TESTING

### D.1.1 Positive and Negative Controls

**Target Analyte Lists:** The laboratory shall analyze those target analytes identified by the client on a project-specific basis. If project-specific information is not available or is incomplete, then the target analyte lists in Appendix DoD-C shall be used. This appendix follows all the NELAC appendices.

**D-3**

#### D.1.1.1 Negative Control - Method Performance

a) Purpose: The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The method blank shall be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure. Procedures shall be in place to determine if a method blank is contaminated. Any affected samples associated with a contaminated method blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

b) Frequency: The method blank shall be analyzed at a minimum of 1 per preparation batch. In those instances for which no separate preparation method is used (example: volatiles in water)

the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

c) Composition: The method blank shall consist of a quality system matrix that is similar to the associated samples and is known to be free of the analytes of interest.

d) Evaluation Criteria and Corrective Action: While the goal is to have no detectable contaminants, each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. The source of contamination shall be investigated and measures taken to minimize or eliminate the problem and affected samples reprocessed or data shall be appropriately qualified if:

**Reporting Limit:** For DoD, the reporting limit is defined by the client. See definition in Appendix B.
**D-4**

1) The concentration of a targeted analyte in the blank is at or above the reporting limit as established by the test method or by regulation, AND is greater than 1/10 of the amount measured in any sample.

2) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

3) When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank shall be evaluated as to the best corrective action for the samples (e.g., reprocessing or data qualifying codes). In all cases the corrective action must be documented.

**Method Blanks:** If the method blank contamination exceeds **one-half** the reporting limit, the laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

The concentrations of common laboratory contaminants shall not exceed the reporting limit.

Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect.

If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying codes.

**Applicable when method-specific guidance does not exist.**
**D-5**

### D.1.1.2 Positive Control - Method Performance

#### D.1.1.2.1 Laboratory Control Sample (LCS)

a) Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicates that the analytical system is “out of control”. Any affected samples associated with an out of control LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes.

b) Frequency: The LCS shall be analyzed at a minimum of 1 per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available such as total suspended

solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

c) Composition: The LCS is a quality system matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. NOTE: the matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may consist of a media containing known and verified concentrations of analytes or as Certified Reference Material (CRM). All analyte concentrations shall be within the calibration range of the methods. The following shall be used in choosing components for the spike mixtures:

The components to be spiked shall be as specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period.

1) For methods that include 1-10 targets, spike all components;

2) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;

3) For methods with more than 20 targets, spike at least 16 components.

**Spiking Compounds:**

- For DoD, all target analytes must be spiked in the LCS. Target analytes are defined by the project or in Appendix DoD-C. For evaluation and acceptance criteria see Appendices DoD-B and DoD-D.
- For multicomponent analytes (e.g. PCBs), the LCS should be spiked with the same constituents as the calibration standard. Multiple samples may be necessary to avoid interference.
- The concentration of the spiked compounds shall be at or below the midpoint of the calibration range or at the appropriate level of concern.

**D-6**

d) Evaluation Criteria and Corrective Action: The results of the individual batch LCS are calculated in percent recovery or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation.

The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria.

**Laboratory Control Sample (LCS):** DoD has established LCS control limits based on a multi-laboratory study. The acceptability of LCS results within a preparatory batch shall be determined using project-specified limits or these DoD limits, if project limits are not available. (See Appendix DoD-D for further explanation. This appendix follows all the NELAC appendices.) If DoD limits are not available for certain analytes, the laboratory shall base LCS acceptability on its in-house limits. The in-house limits must be consistent with any project-specific limits or the limits in Appendix DoD-D, if project-specific limits are not provided.

At a minimum the laboratory in-house limits shall:

- Be statistically derived using scientifically valid and documented procedures,
- Meet the limits specified in the method, if available,
- Be updated on an annual basis and re-established after major changes in the analytical process (e.g., new instrumentation, new chemist),
- Be based on at least 30 data points generated under the same analytical process, and
- Not exclude failed LCS recovery data and statistical outliers from the calculation, unless there is a documented and scientifically valid reason (e.g., bad LCS standard, leaking purging cell).

DoD recommends that control limits be set at mean ± 3 times the standard deviation of recovery data. In addition, DoD strongly recommends that control charts be maintained and used to detect trends and prevent out of control conditions. Control limits shall be continually monitored for shifts in mean recovery, changes in standard deviation, and development of trends.

**D-7**

A LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.

e) If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

**Marginal Exceedance Limits**: DoD defines ME limits as 4 standard deviations around the mean. See Appendix DoD-D.

**D-8**

The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.

**LCS Failure:** DoD does not allow any project-specific analytes of concern to exceed its LCS control limits, even marginally (see Section D.3 of Appendix DoD-D for clarification of project-specific analytes of concern and determination of LCS failure). In addition, DoD does not feel it is appropriate to control batch acceptance on poor performing analytes (see Section D.5 of Appendix DoD-D for further explanation).

**D-9**

The number of allowable marginal exceedances is as follows:

1) >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit;

2) 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit;

3) 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit;

4) 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit;

5) 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit;

6) <11 analytes in LCS, no analytes allowed in ME of the LCS control limit;

Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken. Laboratories must have a written procedure to monitor the application of marginal exceedance allowance to the LCS to ensure random behavior.

**Random Marginal Exceedance:** DoD considers the same analyte exceeding the LCS control limit 2 out of 3 consecutive LCS to be indicative of non-random behavior.

**D-10**

#### D.1.1.3 Sample Specific Controls

The laboratory must document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of matrix specific Quality Control (QC) samples and are designed as data quality indicators for a specific sample using the designated test method. These controls alone are not used to judge laboratory performance.

Examples of matrix specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD); sample duplicates; and surrogate spikes. The laboratory shall have procedures in place for tracking, managing, and handling matrix specific QC criteria including spiking appropriate components at appropriate concentrations, calculating recoveries and relative percent difference, evaluating and reporting results based on performance of the QC samples.

##### D.1.1.3.1 Matrix Spike; Matrix Spike Duplicates

a) Purpose: Matrix specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.

b) Frequency: The frequency of the analysis of matrix specific samples shall be determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the test method.

**Matrix Spike/Matrix Spike Duplicate Frequency:** Each preparatory batch of DoD samples must contain an associated MS and MSD using the same environmental matrix collected for the specific DoD project. If adequate sample material is not available, then the lack of MS/MSDs shall be noted in the case narrative. Additional MS/MSDs may be required by project-specific needs for quality control.

For exceptions to the requirements for matrix spike duplicates, see DoD clarification box D-15.

**D-11**

c) Composition: The components to be spiked shall be as specified by the mandated test method. Any permit specified analytes, as specified by regulation or client requested analytes shall also be included. If there are no specified components, the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period.

1) For methods that include 1-10 targets, spike all components;

2) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;

3) For methods with more than 20 targets, spike at least 16 components.

**Spiking Compounds:**

- For DoD, all target analytes must be spiked in the project-specific MS and MSD. Target analytes are defined by the project or in Appendix DoD-C. For evaluation and acceptance criteria see Appendices DoD-B and DoD-D.
- For multicomponent analytes (e.g., PCBs), the project-specific MS and MSD should be spiked with the same constituents as the calibration standard. Multiple samples may be necessary to avoid interference.
- The concentration of the spiked compounds shall be at or below the midpoint of the calibration range or at the appropriate level of concern. If the native concentration is known, the MS/MSD should be spiked 1-4 times that concentration.

**D-12**

d) Evaluation Criteria and Corrective Action: The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), relative percent difference (RPD), or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation for %R, RPD or other statistical treatment used.

**RPD Calculation:** For DoD, RPD must be calculated as a comparison of measured concentrations.

**D-13**

The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix spike results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

**Matrix Spike/Matrix Spike Duplicate Criteria:** The results of all MS/MSDs must be evaluated against the acceptance criteria for LCS specified by DoD (see Appendix DoD-B for flagging and corrective action and Appendix DoD-D for acceptance criteria).

**D-14**

#### D.1.1.3.2 Matrix Duplicates

a) Purpose: Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.

b) Frequency: The frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e.g., Data Quality Objectives) or as specified by the mandated test method.

**Matrix Duplicates Frequency:** If the known concentration of concern is greater than 5 times LOQ, a matrix duplicate may be analyzed in place of the MSD. A matrix spike is still required (see DoD clarification box D-11). Duplicate analysis should be performed at a minimum frequency of once per preparatory batch per matrix type.

**D-15**

c) Composition: Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.

d) Evaluation Criteria and Corrective Action: The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e.g., absolute differences). The laboratory shall document the calculation for relative percent difference or other statistical treatments.

**RPD Calculation:** For DoD, RPD must be calculated as a comparison of measured concentrations.

**D-16**

Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix duplicates results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

#### D.1.1.3.3 Surrogate Spikes

a) Purpose: Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for every sample matrix.

b) Frequency: Except where the matrix precludes its use or when not commercially available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate test methods.

c) Composition: Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method or MQO. They are often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of select compounds.

d) Evaluation Criteria and Corrective Action: The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory should determine internal criteria and document the method used to establish the limits. Surrogates outside the acceptance criteria must be evaluated for the effect indicated for the

individual sample results. The appropriate corrective action may be guided by the data quality objectives or other site specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria should include appropriate data qualifiers.

**Surrogate Spike Criteria:** Surrogate spikes should be compared to project-specific limits specified by the client or the control limits published in Appendix DoD-D, if project-specific limits are not available. If DoD limits are not available for certain surrogates, the laboratory should compare results to its in-house limits.

**D-17**

### D.1.2 Limit of Detection and Limit of Quantitation

All procedures used must be documented. Documentation must include the quality system matrix type. All supporting data must be retained.

#### D.1.2.1 Limit of Detection (LOD)

The laboratory shall utilize a test method that provides an LOD that is appropriate and relevant for the intended use of the data. An LOD is not required for a test method when test results are not reported outside of the calibration range. LODs shall be determined by the protocol in the mandated test method or applicable regulation. If the protocol for determining LODs is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

**Limits of Detection:** DoD requires results between the LOD and LOQ to be reported, therefore it is anticipated that an LOD must be established for all test methods performed for DoD. A method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from analysis of a sample in a given matrix containing the analyte. A laboratory may establish an LOD by determining the MDL or through an alternative approach that demonstrates the analytes can be reliably detected at 99% confidence. If an alternative approach is used, DoD requires verification checks samples be performed as described below. The general requirements below apply to MDL studies and alternative LOD determination.

Requirements established in 40 CFR 136B are the baseline source of information for determining MDLs. Other published statistical methods may be appropriate as supplemental resources in determining MDLs (for example, Hubaux and Vos studies may be appropriate for methods that do not require preparation, such as GC/MS volatiles in water). The following list clarifies and expands on the fundamental requirements and principles outlined in 40 CFR 136B, and shall be followed when performing work for DoD:

- As stated in 40 CFR 136B, MDLs shall be determined using a minimum of seven replicates. If more than seven replicates are processed, data cannot be excluded, unless exclusion is supported with sound, documented, technically based justification.
- The appropriateness of the analyte concentration in the seven replicates shall be evaluated based on the ratio between the mean recovered concentration and the calculated MDL. The ratio should be between 1 to 5 for reagent water matrix and 1 to 10 for other matrices. If the ratio for any target analyte is outside the acceptable range, the spiked concentration should be adjusted and the MDL studies repeated.
- An LOD or MDL study shall be performed for each analyte and matrix.
- MDLs shall be generated for all applicable matrices, using, at a minimum, a purified matrix free of the analytes of interest (for example, Ottawa sand, reagent-grade water). For metals, Teflon chips or glass beads can be used to simulate the soil matrix.
- MDLs shall be generated for all preparatory and cleanup methods routinely used on samples.

**D-18 (continued on next page)**

- If multiple instruments with identical configurations are used in the laboratory, then the laboratory shall conduct an MDL study on at least one of the instruments and confirm the attainability of that MDL on all instruments by using an MDL verification check sample. The MDL verification check shall be performed quarterly on every instrument.
- If multiple MDL results are generated from multiple instruments with identical configurations, then the highest MDL among those may be used in reporting data from all of those instruments. If a lower MDL is reported for specific samples, then the samples must have been run on that specific instrument on which the lower MDL was generated.
- An MDL verification check shall always be performed immediately following an MDL study.
- If an annual MDL study is not performed, MDL verification checks shall be performed **quarterly**. If the quarterly MDL verification check fails, additional MDL verification checks shall be performed at a higher level to set a higher MDL, or the MDL study shall be reconducted.
- DoD requires that the MDL verification check sample be spiked at **approximately 2 times** the current reported MDL. The MDL verification check sample shall be acceptable if the laboratory can reliably detect and identify by method-specified criteria all analytes in the check sample. If the method has no confirmation, the check sample must produce a signal that is at least 3 times the instrument's noise level.
- Deviations from the above are permitted with the approval of DoD personnel.

**D-18**

a) The LOD shall be initially determined for the compounds of interest in each test method in a quality system matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the LOD must be determined in the quality system matrix of interest (see definition of matrix).

b) LOD must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

c) The laboratory must have established procedures to relate LOD with LOQ.

d) The LOD must be verified annually for each quality system matrix, method and analyte according to the procedure specified in C.3.

#### D.1.2.2 Limit of Quantitation (LOQ)

a) Any established LOQ must be above the LOD.

b) The LOQ must be verified annually for each quality system matrix, method and analyte according to the procedure specified in C.3. Alternatively, the annual LOQ verification is not required if the LOD is reevaluated or verified according to D.1.2.d above.

**Quantitation Range Establishment:** DoD is concerned with both the lower and upper limits of quantitation. For DoD, the quantitation range is defined by the low and high calibration standards and the low standard of the initial calibration curve (or the low-level calibration check standard for methods that require only one standard) establishes the LOQ. The LOQ must be at least three times the LOD. Results outside the quantitation range must be reported as estimated.

In addition, in the cases of compounds that are identified by a recognizable pattern (for example, PCBs, toxaphene, technical-chlordane), the limit of quantitation is not based solely on the limit of detection of the various components, but on the concentration of the mixture at which the pattern becomes recognizable to the analyst.

**D-19**

### D.1.3 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

**Data Reduction Procedures – Automated Processes:** At a minimum, for those processes that are automated, a sample data test set shall be used to test and verify the correct operation of these data reduction procedures (including data capture, manipulation, transfer, and reporting). This shall be done anytime the programming code is modified or otherwise manipulated and applies even in cases where commercial software is used as part of the process.

**D-20**

### D.1.4 Quality of Standards and Reagents

a) The source of standards shall comply with 5.6.2.2.2.

b) Reagent Quality, Water Quality and Checks:

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

2) Water - The quality of water sources shall be monitored and documented and shall meet requirements.

**SOPs – Water Quality in Method SOPs:** When water quality is not specified in the method, the default water quality shall be specified in the method-specific SOPs (for example, ASTM Type I or II) and be of known, documented, and appropriate quality.

**D-21**

3) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.

### D.1.5 Selectivity

a) The laboratory shall evaluate selectivity by following the checks established within the method, which may include mass spectral tuning, second column confirmation, ICP inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, and electrode response factors.

b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

**Data Confirmation:** This requirement may be waived by the client in the case of periodic monitoring of well-characterized media that are tested by the same laboratory. For data that are required to be confirmed, all results shall be reported as confirmed or unconfirmed. If unconfirmed data are reported, they shall be identified separately in the report, with a narrative explaining why the data were not confirmed. Evaluation criteria for the confirmation of results shall be as specified by the method, unless otherwise specified by DoD personnel (see Appendix DoD-B). If method-specific requirements do not exist, the laboratory shall develop and document acceptance criteria for the confirmation of results.

**D-22**

c) The laboratory shall document acceptance criteria for mass spectral tuning.

**Mass Spectral Tuning – Acceptance Criteria:** These acceptance criteria are specified by the method, unless otherwise specified by DoD personnel.

**D-23**

### D.1.6 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

## D.2 TOXICITY TESTING

These standards apply to laboratories measuring the toxicity and/or bioaccumulation of contaminants in effluents (whole effluent toxicity or WET), receiving waters, sediments, elutriates, leachates and soils. In addition to the essential quality control standards described below, some methods may have additional or other requirements based on factors such as the type of quality system matrix evaluated.

### D.2.1 Positive and Negative Controls

a) Positive Control - Reference toxicant tests demonstrate a laboratory's ability to obtain consistent results with the test method and evaluate the overall health and sensitivity of test organisms over time.

1) The laboratory must demonstrate its ability to obtain consistent results with standard reference toxicants (SRT) and complete an initial Demonstration of Capability (DOC) in order to attain accreditation in toxicity testing methods.

i) An initial DOC shall consist of five or more acceptable SRT tests for each test method, species and endpoint with different batches of organisms. Appropriate negative controls (water, sediment, or soil) shall be tested at the frequency and duration specified in the test method. Initial DOCs shall be prepared in accordance with the requirements of Appendix C.

ii) Initial DOC is established by maintenance of SRT test results on control charts. A laboratory shall record the control performance and statistical endpoints (such as NOEC or ECp) for each method species and endpoint on control charts. Initial DOC is established where 95% of the test results required in D.2.1a)1)i) fall within the control limits established in accordance with D.2.1a)1)iii) and meet test acceptability criteria (TAC). The laboratory shall evaluate precision (i.e., coefficient of variation, CV) or sensitivity (i.e., statistical minimum significant difference, SMSD) measures (see D.2.1a)1)iv)) for these tests against method specific or (lacking the former) laboratory-derived criteria to determine validity of the initial DOC.

iii) For endpoints that are point estimates (ICp, ECp) control charts are constructed by plotting the cumulative mean and the control limits which consist of the upper and lower 95% confidence limits (+/- 2 standard deviations). In case of highly variable point estimates which exceed method-specific criteria the control chart limits are adjusted accordingly. For endpoints from hypothesis tests (NOEC, NOAEC) the values are plotted directly and the control limits consist of one concentration interval above and below the concentration representing the central tendency (i.e., the mode).

iv) For endpoints that are point estimates the cumulative mean CV is calculated and for endpoints from hypothesis tests, the SMSD is calculated. These values are maintained on a control chart.

2) Ongoing laboratory performance shall be demonstrated by routine SRT testing for each test method and species and endpoint in accordance with the minimum frequency requirements specified in D.2.1.a.3.

i) Intralaboratory precision is determined on an ongoing basis through the use of control charts as established in D.2.1.a)1)ii. The control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC50 for acute tests, or as appropriate hypothesis test values, such as the NOEC or NOAEC, over time within a laboratory.

ii) After initial laboratory DOC is determined, the control limits and CV for an individual test method endpoints, and species shall be adjusted as additional test results are obtained. After 20 data points are collected for a test method and species, the control chart is maintained using only the last 20 data points, i.e., each successive mean value and control limit is calculated using only the last 20 values.

iii) Control chart limits are expected to be exceeded occasionally regardless of how well a laboratory performs. Acceptance limits for point estimates (ICp, ECp) which are based on 95% confidence limits should theoretically be exceeded for one in twenty tests. Depending on the dilution factor and test sensitivity, control charts based on hypothesis test values (NOEC, NOAEC) may be expected to be exceeded on a similar frequency. Test results which fall outside of control chart limits at a frequency of 5% or less, or which fall just outside control chart limits (especially in the case of highly proficient laboratories which may develop relatively narrow acceptance limits over time), are not rejected *de facto.* Such data are evaluated in comparison with control chart characteristics including the width of the acceptance limits and the degree of departure of the value from acceptance limits.

iv) Laboratories shall develop acceptance/rejection policies, consistent with the test methods, for SRT data which considers source of test organisms, the direction of deviation, test dilution factor, test sensitivity (for hypothesis test values), testing frequency, out-of-control test frequency, relative width of acceptance limits, inter-test CV, and degree of difference between test results and acceptance limits.

v) In the case of reference toxicant data which fails to meet control chart acceptance criteria, the test data are examined for defects, corrective action taken, and the test repeated if necessary, using a different batch of organisms or the data is qualified.

3) The frequency of ongoing laboratory reference toxicant testing shall be as follows unless the method specifically requires less frequent SRT tests (e.g., sediment tests):

i) For test methods conducted at a frequency of monthly or greater, SRT tests shall be conducted at an ongoing frequency of monthly.

ii) For test methods and species commonly used in the laboratory, but which are tested at a frequency of less than monthly, SRT tests shall be conducted concurrently with the environmental test.

iii) If the test organisms are obtained from an outside source the sensitivity of each batch of organisms received from a supplier shall be determined via a concurrent SRT test unless the supplier can provide control chart data for the last five SRT tests using the same SRT and test conditions. Supplied SRT data may not be older than six months.

iv) The DOC for an analyst shall be consistent with 5.2.6.c)3) but the frequency need not exceed the method specified requirements and D.2.1.a)3).

4) These standards do not currently specify a particular reference toxicant and dilution series however, if the state or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements. All reference toxicant tests conducted for a given test method and species must use the same reference toxicant, test concentrations, dilution water and data analysis methods. A dilution factor of 0.5x or greater shall be used for both acute and chronic tests.

5) The reference toxicant tests shall be conducted following the same procedures as the environmental toxicity tests for which the precision is being evaluated, unless otherwise specified in the test method (for example, 10-day sediment tests employ 96-h water-only reference toxicant tests). The test duration, laboratory dilution water, feeding, organism age, range and density, test volumes, renewal frequency, water quality measurements, and the number of test concentrations, replicates and organisms per replicate shall be the same as specified for the environmental toxicity test.

b) Negative Control - Control, Brine Control, Control Sediment, Control Soil or Dilution Water

1) The standards for the use, type and frequency of testing of negative controls are specified by the test methods and by permit or regulation and shall be followed. A negative control is included with each test to evaluate test performance and the health and sensitivity of the specific batch of organisms.

2) Appropriate additional negative controls shall be included when sample adjustments (for example addition of thiosulfate for dechlorination) or solvent carriers are used in the test.

3) Test Acceptability Criteria (TAC) - The test acceptability criteria specified in the test method must be achieved for both the reference toxicant and the effluent or environmental sample toxicity test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity tests.

### D.2.2 Variability and/or Reproducibility

Intralaboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item D.2.1.a above.

### D.2.3 Accuracy

This principle is not applicable to Toxicity Testing.

### D.2.4 Test Sensitivity

a) The SMSD shall be calculated according to the formula specified by the test method and reported with the test results.

b) Point estimates: (LCp, ICp, or ECp) - Confidence intervals shall be reported as a measure of the precision around the point estimate value, when the calculation is possible.

c) The SMSD shall be calculated and reported for only hypothesis test values, such as the NOEC or NOAEC.

### D.2.5 Selection of Appropriate Statistical Analysis Methods

a) If required, methods of data analysis and endpoints are specified by language in the regulation, permit or the test method.

b) Dose Response Curves – The data shall be plotted in the form of a curve relating the dose of the chemical or concentration of sample to cumulative percentage of test organisms demonstrating a response such as death. Evaluation criteria shall be established for interpretation of concentration or dose response curves.

### D.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in toxicity tests is specified in the test method except the reference standard. All reference standards shall be prepared from chemicals which are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in 5.5.2.1.d.

**Typographical Correction:** The above reference should read Section 5.6.3 instead of 5.5.2.1.d **D-24**

c) Only reagent-grade water collected from distillation or deionization units is used to prepare reagents.

### D.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit or regulation.

### D.2.8 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid cross-contamination.

b) Laboratory space must be adequate for the types and numbers of tests performed. The building must provide adequate cooling, heating and illumination for conducting testing and culturing; hot and cold running water must be available for cleaning equipment.

c) Air used for aeration of test solutions, dilution waters and cultures must be free of oil and fumes.

d) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s)) and the names(s) of the taxonomic expert(s) must be kept on file at the laboratory. When organisms are obtained from an outside source the supplier must provide this same information.

e) Instruments used for routine support measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, ammonia and weight shall be calibrated, and/or standardized per manufacturer's instructions. As these are support measurements, only the calibration and verification requirements specified at 5.5.2.1 apply. All measurements and calibrations shall be documented.

**Calibration – Chemical and Physical Parameters:** Instruments used for routine measurements of chemical and physical parameters, such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, weight, and temperature shall be calibrated and/or standardized per manufacturer's instructions and Section 5.5.2.1. All measurements and calibrations shall be documented.

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f) Test temperature shall be maintained as specified for the test method. Temperature control equipment must be adequate to maintain the required test temperature(s). The average daily temperature of the test solutions must be maintained within the method specified range. The minimum frequency of measurement shall be once per 24 hour period. The test temperature for continuous-flow toxicity tests shall be recorded and monitored continuously. Where electronic data loggers are used, temperature shall be monitored at a frequency sufficient to capture temporal variations of the environmental control system.

g) Reagent grade water, prepared by any combination of distillation, reverse osmosis, ion exchange, activated carbon and particle filtration, shall meet the method specified requirements.

h) The quality of the standard dilution water used for testing or culturing must be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. Water used for culturing and testing shall be analyzed for toxic metals and organics whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified. It is recognized that the analyte lists of some methods manuals may not include all potential toxicants, are based on estimates of chemical toxicity available at the time of publication and may specify detection limits which are not achievable in all matrices. However, for those analytes not listed, or for which the measured concentration or limit of detection is greater than the method-specified limit, the laboratory must demonstrate that the analyte at the measured concentration or reported limit of detection does not exceed one tenth the expected chronic value for the most sensitive species tested and/or cultured. The expected chronic value is based on professional judgment and the best available scientific data. The "USEPA Ambient Water Quality Criteria Documents" and the EPA AQUIRE database provide guidance and data on acceptability and toxicity of individual metals and organic compounds.

**Toxicity Test Conditions – Water Quality:** Water used for culturing and testing shall, at a minimum, be analyzed **annually** for toxic metals and organics.

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i) The quality of the food used for testing or culturing must be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. The laboratory shall have written procedures for the evaluation of food acceptance.

j) A subset of organisms used in bioaccumulation tests must be analyzed at the start of the test (baseline) for the target compounds to be measured in the bioaccumulation tests.

**Toxicity Test Conditions – Organisms:** The above requirement also applies to bioconcentration and bioavailability tests.

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k) Test chamber size and test solution volume shall be as specified in the test method. All test chambers used in a test must be identical.

l) Test organisms shall be fed the quantity and type food or nutrients specified in the test method. They shall also be fed at the intervals specified in the test methods.

m) All organisms in a test must be from the same source. Where available certified seeds are used for soil tests.

n) All organisms used in tests, or used as broodstock to produce neonate test organisms (for example cladocerans and larval fish), must appear healthy, show no signs of stress or disease and exhibit acceptable survival (90% or greater) during the 24 hour period immediately preceding use in tests.

o) All materials used for test chambers, culture tanks, tubing, etc. and coming in contact with test samples, solutions, control water, sediment or soil or food must be non-toxic and cleaned as described in the test methods. Materials must not reduce or add to sample toxicity. Appropriate materials for use in toxicity testing and culturing are described in the referenced manuals.

p) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the test methods and shall be documented at least quarterly. For algal and plant tests, the light intensity shall be measured and recorded at the start of each test.

q) The health and culturing conditions of all organisms used for testing shall be documented by the testing laboratory. Such documentation shall include culture conditions (e.g., salinity, hardness, temperature, pH) and observations of any stress, disease or mortality. When organisms are obtained from an outside source, the laboratory shall obtain written documentation of these water quality parameters and biological observations for each lot of organism received. These observations shall adequately address the 24 hour time period referenced in item D.2.8.n. above. The laboratory shall also record each of these observations and water quality parameters upon the arrival of the organisms at the testing laboratory.

r) Age and the age range of the test organisms must be as specified in the test method. Supporting information, such as hatch dates and times, times of brood releases and metrics (for example, chironomid head capsule width) shall be documented.

s) The maximum holding time of effluents (elapsed time from sample collection to first use in a test) shall not exceed 36 hours; samples may be used for renewal up to 72 hours after first use except as prescribed by and approved by the regulatory agency having authority for program oversight.

t) All samples shall be chilled to 0 to 6°C during or immediately after collection (see requirements in section 5.8.3.1) except as prescribed by the method and approved by the regulatory agency having authority for program oversight.

u) Organisms used in a given test must be from the same batch.

v) All tests shall have the minimum number of replicates per treatment as prescribed by the method.

w) The control population of Ceriodaphnia in chronic effluent or receiving water tests shall contain no more than 20% males.

x) The culturing of C. dubia shall be adequate such that blocking by parentage can be established.

y) Dissolved oxygen and pH in aquatic tests shall be within acceptable range at test initiation and aeration (minimal) is provided to tests if, and only if, acceptable dissolved oxygen concentrations cannot be otherwise maintained or if specified by the test method.

z) Test soils or sediments must be within the geochemical tolerance range of the test organism.

aa) An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each test method). The acceptability of the test shall depend on the experience and professional judgment of the technical director and the permitting authority.

## D.3 MICROBIOLOGY TESTING

These standards apply to laboratories undertaking microbiological analysis of environmental samples. Microbiological testing refers to and includes the detection, isolation, enumeration, or identification of microorganisms and/or their metabolites, or determination of the presence or absence of growth in materials and media.

### D.3.1 Sterility Checks and Blanks, Positive and Negative Controls

a) Sterility Checks and Blanks

The laboratory shall demonstrate that the filtration equipment and filters, sample containers, media and reagents have not been contaminated through improper handling or preparation, inadequate sterilization, or environmental exposure.

1) A sterility blank shall be analyzed for each lot of pre-prepared, ready-to-use medium (including chromofluorogenic reagent) and for each batch of medium prepared in the laboratory. This shall be done prior to first use of the medium.

2) For filtration technique, the laboratory shall conduct one beginning and one ending sterility check for each laboratory sterilized filtration unit used in a filtration series. The filtration series may include single or multiple filtration units, which have been sterilized prior to beginning the series. For pre-sterilized single use funnels a sterility check shall be performed on one funnel per lot. The filtration series is considered ended when more than 30 minutes elapses between successive filtrations. During a filtration series, filter funnels must be rinsed with three 20-30 ml portions of sterile rinse water after each sample filtration. In addition, laboratories must insert a sterility blank after every 10 samples or sanitize filtration units by UV light after each sample filtration.

3) For pour plate technique, sterility blanks of the medium shall be made by pouring, at a minimum, one uninoculated plate for each lot of pre-prepared, ready-to-use media and for each batch of medium prepared in the laboratory.

4) Sterility checks on sample containers shall be performed on at least one container for each lot of purchased, pre-sterilized containers. For containers prepared and sterilized in the laboratory, a sterility check shall be performed on one container per sterilized batch with non-selective growth media.

5) A sterility blank shall be performed on each batch of dilution water prepared in the laboratory and on each batch of pre-prepared, ready-to-use dilution water with non-selective growth media.

6) At least one filter from each new lot of membrane filters shall be checked for sterility with non-selective growth media.

b) Positive Controls

Positive culture controls demonstrate that the medium can support the growth of the target organism(s), and that the medium produces the specified or expected reaction to the target organism(s).

1) Each pre-prepared, ready-to-use lot of medium (including chromofluorogenic reagent) and each batch of medium prepared in the laboratory shall be tested with at least one pure culture of a known positive reaction. This shall be done prior to first use of the medium.

c) Negative Controls

Negative culture controls demonstrate that the medium does not support the growth of non-target organisms or does not demonstrate the typical positive reaction of the target organism(s).

Each pre-prepared, ready-to-use lot of selective medium (including chromofluorogenic reagent) and each batch of selective medium prepared in the laboratory shall be analyzed with one or more known negative culture controls, i.e., non-target organisms, as appropriate to the method. This shall be done prior to first use of the medium.

### D.3.2 Test Variability/Reproducibility

For test methods that specify colony counts such as membrane filter or plated media, duplicate counts shall be performed monthly on one positive sample, for each month that the test is performed. If the lab has two or more analysts, each analyst shall count typical colonies on the same plate. Counts must be within 10% difference to be acceptable. In a laboratory with only one microbiology analyst, the same plate shall be counted twice by the analyst, with no more than 5% difference between the counts.

### D.3.3 Method Evaluation

a) Laboratories are required to demonstrate proficiency with the test method prior to first use. This shall be achieved by comparison to a method already approved for use in the laboratory, or by analyzing a minimum of ten spiked samples whose quality system matrix is representative of those normally submitted to the laboratory, or by analyzing and passing one proficiency test series provided by an approved proficiency sample provider. The laboratory shall maintain this documentation as long as the method is in use and for at least 5 years past the date of last use.

b) Laboratories shall participate in the Proficiency Test programs identified by NELAP (4.1.5.k or 5.9.1). The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data.

### D.3.4 Test Performance

a) All growth and recovery media must be checked to assure that the target organism(s) respond in an acceptable and predictable manner (see D.3.1.b).

b) To ensure that analysis results are accurate, target organism identity shall be verified as specified in the method, e.g., by use of the completed test, or by use of secondary verification tests such as a catalase test.

### D.3.5 Data Reduction

The calculations, data reduction and statistical interpretations specified by each test method shall be followed.

### D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared from commercial dehydrated powders or may be purchased ready to use. Media may be prepared by the laboratory from basic ingredients when commercial media are not available or when it can be demonstrated that commercial media do not provide adequate results. Media prepared by the laboratory from basic ingredients must be tested for performance (e.g., for selectivity, sensitivity, sterility, growth promotion, growth inhibition) prior to first use. Detailed testing criteria information must be defined in either the laboratory's test methods, SOPs, Quality Manual, or similar documentation.

b) Reagents, commercial dehydrated powders and media shall be used within the shelf-life of the product and shall be documented according to 5.6.4.

c) Distilled water, deionized water or reverse-osmosis produced water free from bactericidal and inhibitory substances shall be used in the preparation of media, solutions and buffers. The quality of the water shall be monitored for chlorine residual, specific conductance, and heterotrophic bacteria plate count monthly (when in use), when maintenance is performed on the water treatment system, or at startup after a period of disuse longer than one month.

   Analysis for metals and the Bacteriological Water Quality Test (to determine presence of toxic agents or growth promoting substances) shall be performed annually. Results of these analyses shall meet the specifications of the required method and records of analyses shall be maintained for five years. (An exception to performing the Bacteriological Water Quality Test shall be given to laboratories that can supply documentation to show that their water source meets the criteria, as specified by the method, for Type I or Type II reagent water.)

d) Media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer's instructions or the test method. Documentation for media prepared in the laboratory shall include date of preparation, preparer's initials, type and amount of media prepared, manufacturer and lot number, final pH of the media, and expiration date. Documentation for media purchased pre-prepared, ready-to-use shall include manufacturer, lot number, type and amount of media received, date of receipt, expiration date of the media, and pH of the media.

### D.3.7 Selectivity

a) In order to ensure identity and traceability, reference cultures used for positive and negative controls shall be obtained from a recognized national collection, organization, or manufacturer recognized by the NELAP Accrediting Authority. Microorganisms may be single use preparations or cultures maintained by documented procedures that demonstrate the continued purity and viability of the organism.

   1) Reference cultures may be revived (if freeze-dried) or transferred from slants and subcultured once to provide reference stocks. The reference stocks shall be preserved by a technique which maintains the characteristics of the strains. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they must not be re-frozen and re-used.

2) Working stocks shall not be sequentially cultured more than five times and shall not be subcultured to replace reference stocks.

### D.3.8 Constant and Consistent Test Conditions

a) Laboratory Facilities

Floors and work surfaces shall be non-absorbent and easy to clean and disinfect. Work surfaces shall be adequately sealed. Laboratories shall provide sufficient storage space, and shall be clean and free from dust accumulation. Plants, food, and drink shall be prohibited from the laboratory work area.

b) Laboratory Equipment

1) Temperature Measuring Devices

Temperature measuring devices such as liquid-in-glass thermometers, thermocouples, and platinum resistance thermometers used in incubators, autoclaves and other equipment shall be the appropriate quality to meet specification(s) in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement and they shall be calibrated to national or international standards for temperature (see 5.6.2.2.2). Calibration shall be done at least annually.

2) Autoclaves

i) The performance of each autoclave shall be initially evaluated by establishing its functional properties and performance, for example heat distribution characteristics with respect to typical uses. Autoclaves shall meet specified temperature tolerances. Pressure cookers shall not be used for sterilization of growth media.

ii) Demonstration of sterilization temperature shall be provided by use of continuous temperature recording device or by use of a maximum registering thermometer with every cycle. Appropriate biological indicators shall be used once per month to determine effective sterilization. Temperature sensitive tape shall be used with the contents of each autoclave run to indicate that the autoclave contents have been processed.

iii) Records of autoclave operations shall be maintained for every cycle. Records shall include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out) and analyst's initials.

iv) Autoclave maintenance, either internally or by service contract, shall be performed annually and shall include a pressure check and calibration of temperature device. Records of the maintenance shall be maintained in equipment logs.

v) The autoclave mechanical timing device shall be checked quarterly against a stopwatch and the actual time elapsed documented.

3) Volumetric Equipment

Volumetric equipment shall be calibrated as follows:

i) equipment with movable parts such as automatic dispensers, dispensers/diluters, and mechanical hand pipettes shall be verified for accuracy quarterly.

ii) equipment such as filter funnels, bottles, non-class A glassware, and other marked containers shall be calibrated once per lot prior to first use.

iii) the volume of the disposable volumetric equipment such as sample bottles, disposable pipettes, and micropipette tips shall be checked once per lot.

4) UV Instruments

UV instruments, used for sanitization, shall be tested quarterly for effectiveness with an appropriate UV light meter or by plate count agar spread plates. Replace bulbs if output is less than 70% of original for light tests or if count reduction is less than 99% for a plate containing 200 to 300 organisms.

5) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements (see Section 5.5.2.1.d).

6) Incubators, Water Baths, Ovens

i) The stability and uniformity of temperature distribution and time required after test sample addition to re-establish equilibrium conditions in incubators and water baths shall be established. Temperature of incubators and water baths shall be documented twice daily, at least four hours apart, on each day of use.

ii) Ovens used for sterilization shall be checked for sterilization effectiveness monthly with appropriate biological indicators. Records shall be maintained for each cycle that include date, cycle time, temperature, contents and analyst's initials.

7) Labware (Glassware and Plasticware)

i) The laboratory shall have a documented procedure for washing labware, if applicable. Detergents designed for laboratory use must be used.

ii) Glassware shall be made of borosilicate or other non-corrosive material, free of chips and cracks, and shall have readable measurement marks.

iii) Labware that is washed and reused shall be tested for possible presence of residues which may inhibit or promote growth of microorganisms by performing the Inhibitory Residue Test annually, and each time the lab changes the lot of detergent or washing procedures.

iv) Washed labware shall be tested at least once daily, each day of washing, for possible acid or alkaline residue by testing at least one piece of labware with a suitable pH indicator such as bromothymol blue. Records of tests shall be maintained.

## D.4 RADIOCHEMICAL TESTING

These standards apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. These procedures for radiochemical analysis may involve some form of chemical separation followed by detection of the radioactive decay of analyte (or indicative daughters) and tracer isotopes where used. For the purpose of these standards procedures for the determination of radioactive isotopes by mass spectrometry (e.g., ICP-MS or TIMS) or optical (e.g., KPA) techniques are not addressed herein.

### D.4.1 Negative and Positive Controls

a) Negative Controls

1) Method Blank - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The method blank result shall be assessed against the specific acceptance criteria [see 5.4.1.2.b)18] specified in the laboratory method manual [see 5.4.1.2]. When the specified method blank acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b) 19 and 20] shall be followed and results reported with appropriate data qualifying codes. The occurrence of a failed method blank acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

2) In the case of gamma spectrometry, generally a non-destructive analysis, a method blank shall be prepared using a calibrated counting geometry similar to that used for the samples. The container of the appropriate geometry can be empty or filled to similar volume to partially simulate gamma attenuation due to a sample matrix.

3) There shall be no subtraction of the required method blank [see D.4.1.a)1] result from the sample results in the associated preparation or analytical batch unless permitted by method or program. This does not preclude the application of any correction factor (e.g., instrument background, analyte presence in tracer, reagent impurities, peak overlap, etc.) to all analyzed samples, both program/project submitted and internal quality control samples. However, these correction factors shall not depend on the required method blank result in the associated analytical batch.

4) The method blank sample shall be prepared with similar aliquot size to that of the routine samples for analysis and the method blank result and acceptance criteria [5.4.1.2.b)18] shall be calculated in a manner that compensates for sample results based upon differing aliquot size.

b) Positive Controls

1) Laboratory Control Samples - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The laboratory control sample result shall be assessed against the specific acceptance criteria [see 5.4.1.2.b)18] specified in the laboratory method manual [see 5.4.1.2]. When the specified laboratory control sample acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b)19 and 20] shall be followed. The occurrence of a failed laboratory control sample acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

2) Matrix Spike - Shall be performed at a frequency of one per preparation batch for those methods which include a chemical separation process without the use of an internal standard or carrier, and where there is sufficient sample to do so. Although gross alpha, gross beta and tritium measurements do not involve a chemical separation process, matrix spikes shall be performed for these analyses on aqueous samples. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The matrix spike result shall be assessed against the specific acceptance criteria [see 5.4.1.2.b)18] specified in the laboratory method manual [see 5.4.1.2]. When the specified matrix spike acceptance criteria is not met, the specified corrective action and contingencies [see 5.4.1.2.b)19 and 20] shall be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a]. The lack of sufficient sample aliquot size to perform a matrix spike shall be noted in the laboratory report.

3) The activity of the laboratory control sample shall: (1) be at least 5 times the limit of detection and (2) at a level comparable to that of routine samples when such information is available if the sample activities are expected to exceed 5 times the limit of detection.

4) The activity of the matrix spike analytes(s) shall be greater than five times the limit of detection.

5) The laboratory standards used to prepare the laboratory control sample and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration and must meet the requirements for reference standards provided in D.4.7.a).

6) The matrix spike shall be prepared by adding a known activity of target analyte after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.). Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g., plutonium, Pu 238 and Pu 239, using alpha spectrometry), only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified limit of detection each shall be assessed against the specified acceptance criteria.

7) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope the laboratory control sample and matrix spike shall contain isotopes that represent the low (e.g., americium-241), medium (e.g., cesium-137) and high (e.g., cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

8) The laboratory control sample shall be prepared with similar aliquot size to that of the routine samples for analyses.

c) Other Controls

1) Tracer - For those methods that utilize a tracer (i.e., internal standard) each sample result shall have an associated tracer recovery calculated and reported. The tracer shall be added to the sample after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The tracer recovery for each sample result shall be one of the quality control measures to be used to assess the associated sample result acceptance. The tracer recovery shall be assessed against the specific acceptance criteria [see 5.4.1.2.b) 18] specified in the laboratory method manual [see 5.4.1.2]. When the specified tracer recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b) 19 and 20] shall be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

2) Carrier - For those methods that utilize a carrier for recovery determination, each sample shall have an associated carrier recovery calculated and reported. The carrier shall be added to the sample after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The carrier recovery for each sample shall be one of the quality control measures to be used to assess the associated sample result acceptance. The carrier recovery shall be assessed against the specific acceptance criteria [see 5.4.1.2.b) 18] specified in the laboratory method manual [see 5.4.1.2]. When the specified carrier recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b) 19 and 20] shall be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

### D.4.2 Analytical Variability/Reproducibility

a) Replicate - Shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria [see 5.4.1.2.b) 18] specified in the laboratory method manual [see 5.4.1.2]. When the specified replicate acceptance criteria is not met the specified corrective action and contingencies [see 5.4.1.2.b) 19 and 20] shall be followed. The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.10.3.1.a].

b) For low level samples (less than approximately three times the limit of detection) the laboratory may analyze duplicate laboratory control samples or a replicate matrix spike (matrix spike and a matrix spike duplicate) to determine reproducibility within a preparation batch.

### D.4.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Initial Demonstration of Capability - (section 5.4.2.2 and Appendix C) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type (e.g., different detection technique), personnel or method.

b) Proficiency Test Samples - The results of such analysis (4.1.5.k and 5.9.1) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

### D.4.4 Radiation Measurement Instrumentation

Because of the stability and response nature of modern radiation measurement instrumentation, it is not typically necessary to verify calibrate of these systems each day of use. However, verification of calibration is required as outlined in (b) below. This section addresses those practices that are necessary for proper calibration and those requirements of section 5.5.2.2 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

a) Instrument Calibration

1) Given that activity detection efficiency is independent of sample activity at all but extreme activity levels, the requirements of subsections f, h and i of 5.5.2.2.1 are not applicable to radiochemical method calibrations except mass attenuation in gas-proportional counting and sample quench in liquid scintillation counting. Radiation measurement instruments are subject to calibration prior to initial use, when the instrument is placed back in service after malfunctioning and the instrument's response has changed as determined by a performance check or when the instrument's response exceeds predetermined acceptance criteria for the instrument quality control.

2) Instrument calibration shall be performed with reference standards as defined in section D.4.7a. The standards shall have the same general characteristics (i.e., geometry, homogeneity, density, etc.) as the associated samples.

3) The frequency of calibration shall be addressed in the laboratory method manual [see 5.4.1.2.b)13] if not specified in the method. A specific frequency (e.g., monthly) or observations from the associated control or tolerance chart, as the basis for calibration shall be specified.

b) Continuing Instrument Calibration Verification (Performance Checks)

Performance checks shall be performed using appropriate check sources and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the detector response has not significantly changed and therefore the instrument calibration has not changed. The same check source used in the preparation of the tolerance chart or control chart at the time of calibration shall be used in the calibration verification of the instrument. The check sources must provide adequate counting statistics for a relatively short count time and the source should be sealed or encapsulated to prevent loss of activity and contamination of the instrument and laboratory personnel.

1) For gamma spectroscopy systems, the performance checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.

2) For alpha spectroscopy systems, the performance check for energy calibration shall be performed on a weekly basis and the performance check for counting efficiency shall be performed on at least a monthly basis.

3) For gas-proportional and liquid scintillation counters, the performance check for counting efficiency shall be performed on a day of use basis. For batches of samples that uninterruptedly count for more than a day a performance check can be performed at the beginning and end of the batch as long as this time interval is no greater than one week. Verification of instrument calibration does not directly verify secondary calibrations, e.g., the mass efficiency curve or the quench curve.

4) For scintillation counters the calibration verification for counting efficiency shall be performed on a day of use basis.

c) Background Measurement

Background measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality objectives. These values may be subtracted from the total measured activity in the determination of the sample activity.

1) For gamma spectroscopy systems, background measurements shall be performed on at least a monthly basis.

2) For alpha spectroscopy systems, background measurements shall be performed on at least a monthly basis.

3) For gas-proportional counters background measurements shall be performed at least on a weekly basis.

4) For scintillation counters, background measurements shall be performed each day of use.

d) Instrument Contamination Monitoring

The laboratory shall have a written procedure for monitoring radiation measurement instrumentation for radioactive contamination. The procedure shall indicate the frequency of the monitoring and shall indicate criteria, which initiates corrective action.

### D.4.5 Minimum Detectable Activity (MDA)/Minimum Detectable Concentration (MDC)/Lower Level of Detection (LLD)

a) Must be determined prior to sample analysis and must be redetermined each time there is a significant change in the test method or instrument type.

b) The procedures employed must be documented and consistent with mandated method or regulation.

### D.4.6 Data Reduction

a) Refer to Section 5.4.7.2, "Computers and Electronic Data Related Requirements," of this document.

b) Measurement Uncertainties - Each result shall be reported with the associated measurement uncertainty. The procedures for determining the measurement uncertainty must be documented and be consistent with mandated method and regulation.

### D.4.7 Quality of Standards and Reagents

a) The quality control program shall establish and maintain provisions for radionuclide standards.

1) Reference standards that are used in a radiochemical laboratory shall be obtained from the National Institute of Standards and Technology (NIST), or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.

3) Laboratories should consult with the supplier if the lab's verification of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory shall not use a value other than the decay corrected certified value. The laboratory shall have a written procedure for handling, storing and establishment of expiration dates for reference standards.

b) All reagents used shall be analytical reagent grade or better.

### D.4.8 Constant and Consistent Test Conditions

The laboratory shall maintain a radiological control program that addresses analytical radiological control. The program shall address the procedures for segregating samples with potentially widely varying levels of radioactivity. The radiological control program shall explicitly define how low level and high level samples will be identified, segregated and processed in order to prevent sample cross-contamination. The radiological control program shall include the measures taken to monitor and evaluate background activity or contamination on an ongoing basis.

## D.5 AIR TESTING

These standards shall apply to samples that are submitted to a laboratory for the purpose of analysis. They do not apply to field activities such as source air emission measurements or the use of continuous analysis devices.

### D.5.1 Negative and Positive Controls

a) Negative Controls

1) Method Blanks – Shall be performed at a frequency of at least one (1) per batch of twenty (20) environmental samples or less per sample preparation method. The results of the

method blank analysis shall be used to evaluate the contribution of the laboratory provided sampling media and analytical sample preparation procedures to the amount of analyte found in each sample. If the method blank result is greater than the limit of quantitation and contributes greater than 10% of the total amount of analyte found in the sample, the source of the contamination must be investigated and measures taken to eliminate the source of contamination. If contamination is found, the data shall be qualified in the report.

2) Collection Efficiency – Sampling trains consisting of multiple sections (e.g., filters, sorbent tubes, impingers) that are received intact by the laboratory, shall be separated into "front" and "back" sections if required by the client. Each section shall be processed and analyzed separately and the analytical results reported separately.

b) Positive Controls

1) Laboratory Control Sample (LCS) – Shall be analyzed at a rate of at least one (1) per batch of twenty (20) or fewer samples per sample preparation method for each analyte. If a spiking solution is not available, a calibration solution, whose concentration approximates that of the samples, shall be included in each batch and with each lot of media. If a calibration solution must be used for the LCS, the client will be notified prior to the start of analysis. The concentration of the LCS shall be relevant to the intended use of the data and either at a regulatory limit or below it.

c) Surrogates – Shall be used as required by the test method or if requested by the client.

d) Matrix spike – Shall be used as required by the test method, or if requested by the client.

### D.5.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates – Shall be analyzed at a minimum of 1 in 20 samples per sample batch. The laboratory shall document their procedure to select the use of appropriate types of spikes and duplicates. The selected samples(s) shall be rotated among client samples so that various sample matrix problems may be noted and/or addressed. Poor performance in the spikes and duplicates may indicate a problem with the sample composition and shall be reported to the client.

### D.5.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Capability – (Sections 5.2.6 and 5.4.2.2) shall be performed prior to the analysis of any samples and with a significant change in instrument type, personnel, quality system matrix, or test method.

b) Calibration – Calibration protocols specified in Section 5.5.2 shall be followed.

c) Proficiency Test Samples – The results of such analyses (4.1.5.k or 5.9.1) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

### D.5.4 Limit of Detection

The requirements of D.1.2.1 shall apply.

### D.5.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

### D.5.6 Quality of Standards and Reagents

a) The source of standards shall comply with 5.6.2.2.2.

b) The purity of each analyte standard and each reagent shall be documented by the laboratory through certificates of analyses from the manufacturer/vendor, manufacturer/vendor specifications, and/or independent analysis.

**Expiration Date of Standards and Reagents:** In addition to the purity, the date of expiration of each analyte standard and each reagent shall be documented by the laboratory.

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c) In methods where the purity of reagents is not specified, analytical reagent grade or higher quality, if available, shall be used.

### D.5.7 Selectivity

The laboratory shall develop and document acceptance criteria for test method selectivity such as absolute and relative retention times, wavelength assignments, mass spectral library quality of match, and mass spectral tuning.

### D.5.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) The laboratory shall document that all sampling equipment, containers and media used or supplied by the laboratory meet required test method criteria.

c) If supplied or used by the laboratory, procedures for field equipment decontamination shall be developed and their use documented.

d) The laboratory shall have a documented program for the calibration and verification of sampling equipment such as pumps, meter boxes, critical orifices, flow measurement devices and continuous analyzers, if these equipment are used or supplied by the laboratory.

## D.6 ASBESTOS TESTING

These standards apply to laboratories undertaking the examination of asbestos samples. These standards are organized by analytical technique including transmission electron microscopy (TEM) for the analysis of water, wastewater, air, and bulk samples; phase contrast microscopy (PCM) for analysis of workplace air; and polarized light microscopy (PLM) for analysis of bulk samples. These procedures for asbestos analysis involve sample preparation followed by detection of asbestos. If NIST SRMs specified below are unavailable, the laboratory may substitute an equivalent reference material with a certificate of analysis.

### D.6.1 Negative Controls

#### D.6.1.1 Transmission Electron Microscopy

##### D.6.1.1.1 Water and Wastewater

a) Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each 24 shall be tested for background

level. When using glass bottles, four bottles from each 24 shall be tested. An acceptable bottle blank level is defined as ≤ 0.01 MFL > 10 μm. (EPA /600/R-94/134, Method 100.2, Section 8.2)

b) A process blank sample consisting of fiber-free water shall be run before the first field sample. The quantity of water shall be ≥ 10 mL for a 25-mm diameter filter and ≥ 50 mL for a 47-mm diameter filter. (EPA /600/R-94/134, Method 100.2, Section 11.8)

**D.6.1.1.2 Air**

a) A blank filter shall be prepared with each set of samples. A blank filter shall be left uncovered during preparation of the sample set and a wedge from that blank filter shall be prepared alongside wedges from the sample filters. At minimum, the blank filter shall be analyzed for each 20 samples analyzed. (40 CFR Part 763, Appendix A to Subpart E (AHERA), Table 1)

b) Maximum contamination on a single blank filter shall be no more than 53 structures/mm$^2$. Maximum average contamination for all blank filters shall be no more than 18 structures/mm$^2$. (AHERA, III.F.2)

**D.6.1.1.3 Bulk Samples**

a) Contamination checks using asbestos-free material, such as the glass fiber blank in SRM 1866 (Page C-3, NIST Handbook 150-3, August 1994) shall be performed at a frequency of 1 for every 20 samples analyzed. The detection of asbestos at a concentration exceeding 0.1% will require an investigation to detect and remove the source of the asbestos contamination.

b) The laboratory must maintain a list of non-asbestos fibers that can be confused with asbestos (Section 7.5, Page C-8, NIST Handbook 150-3, August 1994). The list must include crystallographic and/or chemical properties that disqualify each fiber being identified as asbestos (Section 2.5.5.2.1 Identification, Page 54, EPA/600/R-93/116).

c) The laboratory should have a set of reference asbestos materials from which a set of reference diffraction and X-ray spectra have been developed.

**D.6.1.2 Phase Contrast Microscopy**

At least two (2) field blanks (or 10% of the total samples, whichever is greater) shall be submitted for analysis with each set of samples. Field blanks shall be handled in a manner representative of actual handling of associated samples in the set with a single exception that air shall not be drawn through the blank sample. A blank cassette shall be opened for approximately thirty (30) seconds at the same time other cassettes are opened just prior to analysis. Results from field blank samples shall be used in the calculation to determine final airborne fiber concentration. The identity of blank filters should be unknown to the counter until all counts have been completed. If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

**D.6.1.3 Polarized Light Microscopy**

a) Friable Materials - At least one blank slide must be prepared daily or with every 50 samples analyzed, whichever is less. This is prepared by mounting a subsample of an isotropic verified non-ACM (e.g., fiberglass in SRM 1866) in a drop of immersion oil ($n_D$ should reflect usage of various $n_D$'s) on a clean slide, rubbing preparation tools (forceps, dissecting needles, etc.) in the mount and placing a clean coverslip on the drop. The entire area under the coverslip must be scanned to detect any asbestos contamination. A similar check must be made after every 20 uses of each piece of homogenization equipment. An isotropic verified non-ACM must be homogenized in the clean equipment, a slide prepared with the material and the slide scanned for asbestos contamination. (This can be substituted for the blank slide mentioned in this section.)

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

### D.6.2 Test Variability/Reproducibility

#### D.6.2.1 Transmission Electron Microscopy

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

##### D.6.2.1.1 Water and Wastewater

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

a) Replicate – A second, independent analysis shall be performed on the same grids but on different grid openings than used in the original analysis of a sample. Results shall be within 1.5X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)

b) Duplicate – A second aliquot of sample shall be filtered through a second filter, prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)

c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings used in the original analysis of a sample. The two sets of results shall be compared according to Turner and Steel (NISTIR 5351). This shall be performed at a frequency of 1 per 20 samples. Qualified analysts must maintain an average of $\geq$ 80% true positives, $\leq$ 20% false negatives, and $\leq$ 10% false positives.

##### D.6.2.1.2 Air

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

The laboratory and TEM analysts must obtain mean analytical results on NIST SRM 1876b so that trimmed mean values fall within 80% of the lower limit and 110% of the upper limit of the 95% confidence limits as published on the certificate. These limits are derived from the allowable false positives and false negatives given in Section D.6.2.1.2c, Verified Analysis, below. SRM 1876b shall be analyzed a minimum of once per year by each TEM analyst.

The laboratory must have documentation demonstrating that TEM analysts correctly classify at least 90% of both bundles and single fibrils of asbestos structures greater than or equal to 1 $\mu$m in length in known standard materials traceable to NIST, such as NIST bulk asbestos SRM 1866.

Interlaboratory analyses shall be performed to detect laboratory bias. The frequency of interlaboratory verified analysis must correspond to a minimum of 1 per 200 grid square analyses for clients.

If more than 1 TEM is used for asbestos analysis, intermicroscope analyses must be performed to detect instrument bias.

a) Replicate – A second, independent analysis shall be performed in accordance with Section D.6.2.1.1.a. (AHERA, Table III)

b) Duplicate – A second wedge from a sample filter shall be prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (AHERA, Table III)

c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section D.6.2.1.1.c. (AHERA, Table III)

#### D.6.2.1.3 Bulk Samples

Determination of precision and accuracy should follow guidelines in NISTIR 5951, Guide for Quality Control on the Qualitative and Quantitative Analysis of Bulk Asbestos Samples: Version 1. Because bulk samples with low (<10%) asbestos content are the most problematic, a laboratory's quality control program should focus on such samples. At least 30% of a laboratory's QC analyses shall be performed on samples containing from 1% to 10% asbestos.

a) Intra-Analyst Precision - At least 1 out of 50 samples must be reanalyzed by the same analyst. For single analyst laboratories, at least 1 out of every 10 samples must be reanalyzed by the same analyst.

b) Inter-Analyst Precision - At least 1 out of 15 samples must be reanalyzed by another analyst. Inter-analyst results will require additional reanalysis, possibly including another analyst, to resolve discrepancies when classification (ACM vs. non-ACM) errors occur, when asbestos identification errors occur, or when inter-analyst precision is found to be unacceptable.

c) Inter-Laboratory Precision - The laboratory must participate in round robin testing with at least one other laboratory. Samples must be sent to this other lab at least four times per year. These samples must be samples previously analyzed as QC samples. Results of these analyses must be assessed in accordance with QC requirements. As a minimum, the QC requirements must address misclassifications (false positives, false negatives) and misidentification of asbestos types.

### D.6.2.2 Phase Contrast Microscopy

a) Inter-Laboratory Precision – Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory quality assurance program that as a minimum includes participation of at least two (2) other independent laboratories. Each laboratory shall participate in round robin testing at least once every six (6) months with at least all the other laboratories in its inter-laboratory quality assurance group. Each laboratory shall submit slides typical of its own workload for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology. Results of this QA program shall be posted in each laboratory to keep the microscopists informed.

b) Intra- and Inter-Analyst Precision – Each analyst shall select and count a prepared slide from a "reference slide library" on each day on which air counts are performed. Reference slides shall be prepared using well-behaved samples taken from the laboratory workload. Fiber densities shall

cover the entire range routinely analyzed by the laboratory. These slides shall be counted by all analysts to establish an original standard deviation and corresponding limits of acceptability. Results from the daily reference sample analysis shall be compared to the statistically derived acceptance limits using a control chart or a database. It is recommended that the labels on the reference slides be periodically changed so that the analysts do not become familiar with the samples. Intra- and inter-analyst precision may be estimated from blind recounts on reference samples. Inter-analyst precision shall be posted in each laboratory to keep the microscopists informed.

**D.6.2.3 Polarized Light Microscopy**

Refer to Section D.6.2.1.3.

**D.6.3 Other Quality Control Measures**

**D.6.3.1 Transmission Electron Microscopy**

**D.6.3.1.1 Water and Wastewater**

a) Filter preparations shall be made from all six asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between 1 and 20 structures (> 10 μm) per 0.01 $mm^2$. One of these preparations shall be analyzed independently at a frequency of 1 per 100 samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with Turner and Steel (NISTIR 5351).

b) NIST SRM 1876b must be analyzed annually by each analyst. Results shall be evaluated in accordance with limits published for that SRM. Comment: This SRM is not strictly appropriate for waterborne asbestos but analysts can demonstrate general TEM asbestos competence by producing results within the published limits of this (the only recognized TEM counting standard) SRM.

**D.6.3.1.2 Air**

a) Filter preparations shall be made from all six asbestos types in accordance with Section D.6.3.1.1.a.

b) NIST SRM 1876b must be analyzed annually in accordance with Section D.6.3.1.1.b.

**D.6.3.1.3 Bulk Samples**

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite). Standards for the six asbestos types listed are available from NIST (SRMs 1866 and 1867). These materials can also be used as identification standards for AEM (Section 3.2.1 Qualitative Analysis, Page 57, EPA/600/R-93/116).

**D.6.3.2 Phase Contrast Microscopy**

a) Test for Non-Random Fiber Distribution - Blind recounts by the same analyst shall be performed on 10% of the filters counted. A person other than the counter should re-label slides before the second count. A test for type II error (NIOSH 7400, Issue 2, 15 August 1994, Section 13) shall be performed to determine whether a pair of counts by the same analyst on the same slide should be rejected due to non-random fiber distribution. If a pair of counts is rejected by this test, the remaining samples in the set shall be recounted and the new counts shall be tested against first counts. All rejected paired counts shall be discarded. It shall not be necessary to use this statistic on blank recounts.

b) All individuals performing airborne fiber analysis must have taken the NIOSH Fiber Counting Course for sampling and evaluating airborne asbestos dust or an equivalent course.

c) All laboratories shall participate in a national sample testing scheme such as the Proficiency Analytical Testing (PAT) program or the Asbestos Analysts Registry (AAR) program, both sponsored by the American Industrial Hygiene Association (AIHA), or equivalent.

#### D.6.3.3 Polarized Light Microscopy

a) Friable Materials - Because accuracy cannot be determined by reanalysis of routine field samples, at least 1 out of 100 samples must be a standard or reference sample that has been routinely resubmitted to determine analyst's precision and accuracy. A set of these samples should be accumulated from proficiency testing samples with predetermined weight compositions or from standards generated with weighed quantities of asbestos and other bulk materials (Perkins and Harvey, 1993; Parekh et al., 1992; Webber et al., 1982). At least half of the reference samples submitted for this QC must contain between 1 and 10% asbestos.

b) Non-Friable Materials - At least 1 out of 100 samples must be a verified quantitative standard that has routinely been resubmitted to determine analyst precision and accuracy.

### D.6.4 Method Evaluation

In order to ensure the accuracy of reported results, the following procedures shall be in place:

a) Demonstration of Capability – (Refer to Section 10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, or method.

b) Performance Audits – (Refer to Section 4.2j or 5.3.4) The results of such analyses shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

**Typographical Correction:** The reference in item a) should read Sections 5.2.6 and 5.4.2.2 instead of Section 10.2.1. The reference in item b) should read Sections 4.1.5.k and 5.9.1 instead of Sections 4.2.j and 5.3.4.

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### D.6.5 Asbestos Calibration

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

#### D.6.5.1 Transmission Electron Microscopy

AEM (Analytical Electron Microscopy) equipment requirements will not be discussed in this document.

##### D.6.5.1.1 Water and Wastewater

All calibrations listed below (unless otherwise noted) must be performed under the same analytical conditions used for routine asbestos analysis and must be recorded in a notebook and include date and analyst's signature. Frequencies stated below may be reduced to "before next use" if no samples are analyzed after the last calibration period has expired. Likewise, frequencies may have to be increased following non-routine maintenance or unacceptable calibration performance.

a) Magnification Calibration – Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000 and 20,000x. A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.1)

b) Camera Constant – The camera length of the TEM in the Selected Area Electron Diffraction (SAED) mode must be calibrated before SAED patterns of unknown samples are observed. The diffraction specimen must be at the eucentric position for this calibration. This calibration shall allow accurate (< 10% variation) measurement of layer-line spacings on the medium used for routine measurement, i.e., the phosphor screen or camera film. This must also allow accurate (< 5% variation) measurement of zone axis SAED patterns on permanent media, e.g., film. Calibrations shall be performed monthly to establish the stability of the camera constant (EPA /600/R-94/134, Method 100.2, Section 10.2). Where non-asbestiform minerals may be expected (e.g., winchite, richterite, industrial talc, vermiculite, etc.), an internal camera constant standard such as gold, shall be deposited and measured on each sample to facilitate accurate indexing of zone axis SAED patterns. In such cases, layer line analysis alone shall not be used. Calibration data must be displayed on control charts that show trends over time.

c) Spot Size – The diameter of the smallest beam spot at crossover must be less than 250 nm as calibrated quarterly. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.3)

d) Beam Dose – The beam dose shall be calibrated so that beam damage to chrysotile is minimized, specifically so that an electron diffraction pattern from a single fibril $\geq 1$ $\mu$m in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 15 seconds.

e) EDXA System

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

2) The ability of the system to resolve the Na K$\alpha$ line from the Cu L line shall be confirmed quarterly by obtaining a spectrum from the NIST SRM 1866 crocidolite sample on a copper grid.

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

4) The detector resolution shall be checked quarterly to ensure a full-width half-maximum resolution of < 175 eV at Mn K$\alpha$ (5.90 keV). Calibration data must be displayed on control charts that show trends over time.

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

6) The sensitivity of the detector for collecting x-rays from small volumes shall be documented quarterly by collecting resolvable Mg and Si peaks from a unit fibril of NIST SRM 1866 chrysotile.

f) Low Temperature Asher – The low temperature asher shall be calibrated quarterly by determining a calibration curve for the weight vs. ashing time of collapsed mixed-cellulose-ester (MCE) filters. Calibration data must be displayed on control charts that show trends over time.

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

**D.6.5.1.2 Air**

All calibrations must be performed in accordance with Section D.6.5.1.1, with the exception of magnification. Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 15,000 to 20,000x (AHERA, III.G.1.c). A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

**D.6.5.1.3 Bulk Samples**

All calibrations must be performed in accordance with Section D.6.5.1.2.

**D.6.5.2 Phase Contrast Microscopy**

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

b) The phase-shift limit of detection of the microscope shall be checked monthly or after modification or relocation using an HSE/NPL phase-contrast test slide for each analyst/microscope combination (refer to NIOSH 7400, Issue 2, 15 August 1994, Section 10b). This procedure assures that the minimum detectable fiber diameter (< ca. 0.25μm) for this microscope is achieved.

c) Prior to ordering the Walton-Beckett graticule, calibration, in accordance with NIOSH 7400, Issue 2, 15 August 1994, Appendix A, shall be performed to obtain a counting area 100 μm in diameter at the image plane. The diameter, $d_c$ (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule. The field diameter (D) shall be verified (or checked), to a tolerance of 100 μm ± 2 μm, with a stage micrometer upon receipt of the graticule from the manufacturer. When changes (zoom adjustment, disassembly, replacement, etc.) occur in the eyepiece-objective-reticle combination, field diameter must be re-measured (or re-calibrated) to determine field area ($mm^2$). Re-calibration of field diameter shall also be required when there is a change in interpupillary distance (i.e., change in analyst). Acceptable range for field area shall be 0.00754 $mm^2$ to 0.00817 $mm^2$. The actual field area shall be documented and used.

**D.6.5.3 Polarized Light Microscopy**

a) Microscope Alignment – To accurately measure the required optical properties, a properly aligned polarized light microscope (PLM) shall be utilized. The PLM shall be aligned before each use. (Section 2.2.5.2.3, EPA/600/R-93/116, July 1993)

b) Refractive Index Liquids – Series of $n_D$ = 1.49 through 1.72 in intervals less than or equal to 0.005. Refractive index liquids for dispersion staining, high-dispersion series 1.550, 1.605, 1.680. The accurate measurement of the refractive index (RI) of a substance requires the use of calibrated refractive index liquids. These liquids shall be calibrated at first use and semiannually, or next use, whichever is less frequent, to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or RI glass beads.

### D.6.6 Analytical Sensitivity

#### D.6.6.1 Transmission Electron Microscopy

##### D.6.6.1.1 Water and Wastewater

An analytical sensitivity of 200,000 fibers per liter (0.2 MFL) is required for each sample analyzed (EPA /600/R-94/134, Method 100.2, Section 1.6). Analytical sensitivity is defined as the waterborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the fraction of the filter sampled and the dilution factor (if applicable).

##### D.6.6.1.2 Air

An analytical sensitivity of 0.005 structures/$cm^2$ is required for each sample analyzed. Analytical sensitivity is defined as the airborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the effective surface area of the filter, the filter area analyzed, and the volume of air sampled (AHERA, Table I).

##### D.6.6.1.3 Bulk Samples

a) The range is dependent on the type of bulk material being analyzed. The sensitivity may be as low as 0.0001% depending on the extent to which interfering materials can be removed during the preparation of AEM specimens. (Section 2.5.2 Range, Page 51, EPA/600/R-93/116)

b) There should be an error rate of less than 1% on the qualitative analysis for samples that contain chrysotile, amosite, and crocidolite. A slightly higher error rate may occur for samples that contain anthophyllite, actinolite, and tremolite, as it can be difficult to distinguish among the three types. (Section 3, Page 10, NIST Handbook 150-3, August 1994)

#### D.6.6.2 Phase Contrast Microscopy

The normal quantitative working range of the test method is 0.04 to 0.5 fiber/$cm^2$ for a 1000 L air sample. An ideal counting range on the filter shall be 100 to 1300 fibers/$mm^2$. The limit of detection (LOD) is estimated to be 5.5 fibers per 100 fields or 7 fibers/$mm^2$. The LOD in fiber/$cm^2$ will depend on sample volume and quantity of interfering dust but shall be <0.01 fiber/$cm^2$ for atmospheres free of interferences. (NIOSH 7400, Issue 2, 15 August 1994)

#### D.6.6.3 Polarized Light Microscopy

The laboratory shall utilize a test method that provides a limit of detection that is appropriate and relevant for the intended use of the data. Limit of detection shall be determined by the protocol in the test method or applicable regulation.

### D.6.7 Data Reduction

#### D.6.7.1 Transmission Electron Microscopy

**Typographical Correction:** The references in item a) of Sections D.6.7.1.1, D.6.7.1.2, D.6.7.1.3, D.6.7.2, and D.6.7.3 should read Section 5.4.7.2 instead of Section 10.6.

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##### D.6.7.1.1 Water and Wastewater

a) The concentration of asbestos in a given sample must be calculated in accordance with EPA /600/R-94/134, Method 100.2, Section 12.1. Refer to Section 10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample (EPA /600/R-94/134, Method 100.2, Section 12.2.2).

##### D.6.7.1.2 Air

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized, e.g., AHERA. Refer to Section 10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

##### D.6.7.1.3 Bulk Samples

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – Proficiency testing for floor tiles analyzed by TEM following careful gravimetric reduction (New York ELAP Certification Manual Item 198.4) has revealed an interlaboratory standard deviation of approximately 20% for residues containing 70% or more asbestos. Standard deviations range from 20% to 60% for residues with lower asbestos content.

#### D.6.7.2 Phase Contrast Microscopy

a) Airborne fiber concentration in a given sample must be calculated in accordance with NIOSH 7400, Issue 2, 15 August 1994, Sections 20 and 21. Refer to Section 10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the intra-laboratory and inter-laboratory relative standard deviation with each set of results. (NIOSH 7400, Issue 2, 15 August 1994)

c) Fiber counts above 1300 fibers/mm$^2$ and fiber counts from samples with >50% of the filter area covered with particulate should be reported as "uncountable" or "probably biased". Other fiber counts outside the 100-1300 fibers/mm$^2$ range should be reported as having "greater than optimal variability" and as being "probably biased".

#### D.6.7.3 Polarized Light Microscopy

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Method Uncertainties – Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonable expanded errors (refer to EPA/600/R-93/116, July 1993, Table 2-1) should be generated for different concentrations of asbestos.

### D.6.8 Quality of Standards and Reagents

#### D.6.8.1 Transmission Electron Microscopy

a) The quality control program shall establish and maintain provisions for asbestos standards.

   1) Reference standards that are used in an asbestos laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

   2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

b) All reagents used shall be analytical reagent grade or better.

c) The laboratory shall have mineral fibers or data from mineral fibers that will allow differentiating asbestos from at least the following "look-alikes": fibrous talc, sepiolite, wollastonite, attapulgite (palygorskite), halloysite, vermiculite scrolls, antigorite, lizardite, pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample.

#### D.6.8.2 Phase Contrast Microscopy

Standards of known concentration have not been developed for this testing method. Routine workload samples that have been statistically validated and national proficiency testing samples such as PAT and AAR samples available from the AIHA may be utilized as reference samples (refer to Section D.6.2.2b) to standardize the optical system and analyst. All other testing reagents and devices (HSE/NPL test slide and Walton-Beckett Graticule) shall conform to the specifications of the method (refer to NIOSH 7400, Issue 2, 15 August 1994).

#### D.6.8.3 Polarized Light Microscopy

Refer to Section D.6.8.1.

### D.6.9 Constant and Consistent Test Conditions

The laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

## APPENDIX E - ADDITIONAL SOURCES OF INFORMATION
**(Non-Mandatory Appendix)**

Additional sources of information are available to assist laboratories in the design and implementation of a quality system. These materials may be found on the NELAC web page at www.epa.gov/ttn/nelac under the topic "Related Information."

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# DOD APPENDICES

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## APPENDIX DOD-A – REPORTING REQUIREMENTS

In the absence of client specified reporting criteria, the reporting requirements outlined below shall be used for hard-copy data reports from the laboratory. They are divided into mandatory requirements for all printed data reports, and optional requirements. Optional reporting requirements are those that may be required by a specific project, depending upon the needs of the project. The following elements are required: cover sheet, table of contents, case narrative, analytical results, sample management records, and QA/QC information. Information for third-party review may be required depending on project-specific requirements or the method being used. The requirements below do not dictate what records the laboratory should maintain.

1. 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)
- name and location of any subcontractor laboratories, and appropriate test method performed
- contract number
- 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
- total number of pages

2. Table of Contents. Laboratory data packages should 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. 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, and
- 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.

The case narrative shall provide:

- a table(s) summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical test methods were performed. If multiple laboratories performed analyses, the name and location of each laboratory should be associated with each sample.
- a list of samples that were received but not analyzed
- 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 samples and analytes for which manual integration was necessary
- appropriate notation of any other factors that could affect the sample results (e.g., air bubbles in VOC sample vials, excess headspace in soil VOC containers, the presence of multiple phases, sample temperature and sample pH excursions, container type or volume, etc.)
- identification of numerical results outside of limits of quantitation

4. 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
- matrix (soil, water, oil, etc.)
- date sample extracted or prepared
- date and time sample analyzed
- method numbers for all preparation, cleanup, and analysis procedures employed
- analyte or parameter
- method reporting limits and method limits of quantitation (at or above the low-level standard concentration) adjusted for sample-specific factors (e.g., aliquot size, dilution/concentration factors, moisture content)
- all samples and analytes for which manual integration occurred, including the cause and justification
- limits of detection or method detection limits
- analytical results with correct number of significant figures
- any data qualifiers assigned
- concentration units
- dilution factors
- any dilutions or concentrations for all reported data, and if neat or less diluted results are available, recorded and reported data from both runs
- percent moisture or percent solids (all soils are to be reported on a dry weight basis)

The following information is optional but may be required site-specifically:

- laboratory name and location (city and state)
- sample description
- sample preservation or condition at receipt
- date and time sample collected
- date sample received
- sample aliquot analyzed
- final extract volume
- CAS numbers
- statements of the estimated uncertainty of test results

5. Sample Management Records. These types of records include the documentation accompanying the samples:

- 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)
- telephone conversation records associated with actions taken or quality issues
- if the laboratory collected the sample, sampling procedures

6. QA/QC Information. The minimum internal QC data package must include:

- matrix spikes percent recovery
- relative percent difference (RPD) of required duplicates
- LCS percent recoveries
- in-house LCS control limits, if they exceed DoD limits (see Appendix DoD-D Section D.7)
- surrogate percent recoveries (organics)

- tracer recoveries (radiochemical)
- method blank results
- preparation, analysis, and other batch numbers
- QC acceptance criteria for MS, LCS, surrogates, etc.
- spike concentrations for MS, LCS, surrogates, etc.

7. Information for Third-Party Review. The information listed below is required if third-party (from outside the laboratory) data validation or verification is to be performed. This information is therefore optional and is provided only when the project-specific requirements specify that a third-party review will occur:

- calibration data from the initial calibration curve
- initial calibration verification (ICV)
- continuing calibration verification(s) (CCV)
- performance standards analyzed in conjunction with the test method (e.g., tuning standards, degradation check standards, etc.)
- preparation, analysis, and other batch numbers[1]
- raw data (e.g., chromatograms, mass spectrum results)
- matrix spike (MS), if applicable (includes spike target concentration levels, measured spike concentration, and calculated recoveries)[1]
- RPD of required duplicates (e.g., MS, LCS, field duplicates)[1]
- method blank results[1]
- LCS recoveries[1]
- surrogate recoveries (organics)[1]
- serial dilutions (SD) percent difference (inorganics)
- post-digestion spikes recovery (inorganics)
- project action levels, DQOs, MQOs, and associated acceptance criteria
- supporting documentation (e.g., run logs, sample preparation logs, standard preparation logs).

In addition, the data package for third party review may include summary forms from method detection limit studies.

The data validation guidelines for performance-based methods established in other DoD guidance on data review and data validation, EPA national functional guidelines, EPA regional functional guidelines, and project-specific guidelines for validation may all have distinct reporting formats. The appropriate validation guidelines should be consulted to determine what type of data package is required.

[1] Required for other purposes identified in number 6, QA/QC Information.

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## APPENDIX DOD-B – QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the method shall be followed. In some cases the method may be ambiguous or provide insufficient detail. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for analytical methods (SW-846) commonly used when investigating DoD sites. The tables specify the method requirements, when available, as well as additional clarification and/or requirements from DoD. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the appropriate section is made. The methods should always be referenced, however, for clarification purposes. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating DoD preferences when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed.

**SW-846 Methods**

This appendix is based on the method versions current at the time of publication, regardless of status (promulgated, draft, proposed, etc.). As methods are revised, subsequent versions of this manual may incorporate the changes. If the requirements in this appendix do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

Table B-1 below presents a summary of the definition, purpose, and evaluation of the major QC checks required in the subsequent QC tables (B-2 through B-10) for the various methods. The *definition* column describes generally what the QC check is and/or how it is performed. The *purpose* column describes why the check is important for assessing and measuring the quality of the data being generated. The *evaluation* column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms and a glossary relevant to this appendix follows Table B-10.

### TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION

| QC Check | Definition | Purpose | Evaluation |
|---|---|---|---|
| **Breakdown check (Endrin - Method 8081 only, DDT - Methods 8081 and 8270)** | Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions. | To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port. | If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration. |
| **Calibration blank (metals only)** | Reagent water containing no analytes of interest, but acidified to the same pH as all samples. | To determine the zero point of the calibration curve for all initial and continuing calibrations. | This is a required QC procedure. Continuing calibration blank responses above two times the MDL require corrective action. |
| **Confirmation of positive results (organics only)** | Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified. | To verify the identification of an analyte. | This is a required QC procedure. All positive results must be confirmed. |

## TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (CONTINUED)

| QC Check | Definition | Purpose | Evaluation |
|---|---|---|---|
| **Continuing calibration verification (CCV)** | The verification of the initial calibration that is required during the course of analysis 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. (IDQTF) | To verify that instrument response is reliable, and has not changed significantly from the current initial calibration curve. | If the values for the analytes are outside the acceptance criteria, the initial calibration may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging. |
| **Demonstrate acceptable analyst capability** | Analyst runs QC samples in series to establish his/her ability to produce data of acceptable accuracy and precision. | To establish the analyst's ability to produce data of acceptable accuracy and precision. | The average recovery and standard deviation of the replicates must be within designated acceptance criteria.<br><br>Analysis of field samples cannot be conducted until this check is successful. |
| **Dilution test (metals only)** | Analysis of a positive sample, which has been diluted to a concentration one-fifth of the original, to confirm that there is no interference in the original sample analysis. (Modified COE) | To assess matrix interference. | Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences, and such samples may be analyzed without using the method of standard additions. Results outside acceptance limits indicate a possible matrix effect. For ICP, a post-digestion spike must be run; for GFAA, a recovery test must be run. |
| **Distilled standards (one high and one low) (cyanide only)** | Standards are run through the distillation procedure and then compared to the undistilled standards' reported values. (Method) | To check the efficiency of the distillation process. | Results must agree to within ± 15% of the undistilled value before analysis can proceed. |
| **Duplicate sample (replicate)** | Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (Modified QSM) | To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix. | A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate.<br><br>If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system. |
| **GC column performance check (Methods 8280 and 8290 only)** | Analysis of method-specified compounds to verify chromatographic separation of dioxin isomers. (Method) | To evaluate the performance of the analytical system and establish retention time window markers for dioxin isomers. | Sample analysis cannot begin until method-specified criteria are met. |
| **Initial calibration for all analytes (ICAL)** | Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method. | To establish a calibration curve for the quantification of the analytes of interest. | Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The initial calibration must be successful before any samples or other QC check samples can be analyzed. |
| **Instrument detection limit (IDL) study (Methods 6010 and 6020 only)** | The process to determine the minimum concentration of a substance (analyte) that an instrument can differentiate from noise. The procedure for calculating varies by method. | To provide an evaluation of instrument sensitivity. | IDLs must be established before samples can be analyzed. |

## TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (CONTINUED)

| QC Check | Definition | Purpose | Evaluation |
|---|---|---|---|
| **Interference check solutions (ICP Only)** | A pair of solutions containing interfering elements that are used to verify the correction factors of analytes of concern. | To verify the established correction factors by analyzing the interference check solution at the beginning of the analytical sequence. | No samples can be run if this check does not pass acceptance criteria. |
| **Internal standards** | A known amount of standard added to all standards and samples as a reference for evaluating and controlling the precision and bias of the applied analytical method. (QSM) | To verify that the analytical system is in control. | Any samples associated with out-of-control results must be reanalyzed. |
| **Laboratory control sample (LCS) containing all analytes required to be reported** | A QC standard of known composition prepared using reagent free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is analyzed using the same sample preparation, reagents, and analytical methods employed for regular samples. (Guide to Environmental Analytical Methods, 2nd Edition) | To evaluate method performance by assessing the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix.<br><br>Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance). | This is a required QC check. The inability to achieve acceptable recoveries in the LCS indicates problems with the accuracy/bias of the measurement system.<br><br>Failure to achieve acceptable recoveries in a “clean” matrix is an indicator of possible problems achieving acceptable recoveries in field samples. |
| **Linear dynamic range or high-level check standards (ICP only)** | High-level check standard periodically analyzed to verify the linearity of the calibration curve at the upper end. | To verify quantitative accuracy of data up to the high-level standard. | This QC check establishes the upper linear range of the calibration. |
| **Low-level calibration check standard (ICP only)** | A reference standard that contains a quantity of analyte (greater than or equal to 3 times the LOD). | To confirm the accuracy of measurements at or near the LOQ. It establishes the LOQ of the calibration curve for those ICP methods that rely on single point calibration. It also may be used to validate a client’s reporting limit. | This QC check must be within acceptance criteria before any samples are analyzed. |
| **Matrix spike** | A sample prepared by adding a known amount of targeted analyte(s) to an aliquot of a specific environmental sample for which a separate determination of the target analyte concentration is available. (Modified G-5) | To assess the performance of the method as applied to a particular matrix.<br><br>Matrix spikes are used, for example, to determine the effect of the matrix on a method’s recovery efficiency.<br><br>The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix. | The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The laboratory should not correct for recovery; only report the results of the analyses and the associated matrix spike results and indicate that the results from these analyses have increased uncertainty. |

## TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (CONTINUED)

| QC Check | Definition | Purpose | Evaluation |
|---|---|---|---|
| **Matrix spike duplicate (MSD)** | A second replicate matrix spike prepared in the laboratory, spiked with an identical, known amount of targeted analyte(s), and analyzed to obtain a measure of the precision of the recovery for each analyte. (Modified QSM) | To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix.<br><br>Used to determine the precision of the intralaboratory analytical process for a specific sample matrix. | When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate.<br><br>Also, if the sample matrix is more homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system. |
| **Matrix verification sample (hexavalent chromium only)** | A pH-adjusted filtrate that has been spiked with hexavalent chromium to ensure that the sample matrix does not have a reducing condition or other interferents that could affect color development. (Modified Method) | To ensure that the sample matrix does not have a reducing condition or other interferents that affect color development. | To verify the absence of an interference, the spike recovery must be between 85% and 115%. If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7197, Chelation/Extraction) should be used. |
| **Method detection limit (MDL) verification check** | A low-level spike taken through the preparatory and analytical steps at approximately two times the MDL used to verify that the laboratory can detect analytes at the calculated MDL. | To validate the MDL on an ongoing basis. | If the MDL verification check fails, the MDL verification check shall be reprepped and reanalyzed at a higher level to set a higher MDL or the MDL study must be repeated. |
| **Method blank** | A sample of a matrix similar to the batch of associated samples (when available) in which no target analytes or interferences are present at concentrations that impact the analytical results. It is processed simultaneously with samples of similar matrix and under the same conditions as the samples. (Modified QSM) | To assess background interference or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure. | This is one of the QC samples used to measure laboratory accuracy/bias. This sample could indicate whether contamination is occurring during sample preparation and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analyte(s) in all samples in the associated preparatory batch, as appropriate. For common laboratory contaminants, no analytes detected > RL. See Section D.1.1.1 and DoD clarification box D-5. |
| **MDL study** | The process to determine the minimum concentration of a substance (analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40CFR part 136 Appendix B) | To determine the lowest concentration of an analyte that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero. | MDLs must be established prior to sample analysis. The reporting limit or LOQ is at least three times the MDL.<br><br>Used in combination with the MDL verification check to validate the MDL on an ongoing basis. |
| **Method of standard additions (ICP/GFAA only)** | Adding known amounts of standard to one or more aliquots of the processed sample solution. (Method) | To compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. | This is the method used when matrix interferences are present and do not allow determination of accurate sample results. |

## TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (CONTINUED)

| QC Check | Definition | Purpose | Evaluation |
|---|---|---|---|
| **Post digestion spike addition (ICP and ICP/MS only)** | An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects. | To confirm the presence of a matrix interference. Assess matrix effects based on, (1) the occurrence of new and unusual matrices included within the batch, or (2) contingency analysis based on serial dilution or matrix spike failures. | To verify the absence of an interference, the spike recovery must be between 75% and 125%. Results outside the acceptance limits require a method of standard additions (MSA) for all samples within the batch. |
| **Recovery test (GFAA only)** | An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects. | To confirm the presence of a matrix interference. Assess matrix effects based on, (1) the occurrence of new and unusual matrices included within the batch, or (2) contingency analysis based on serial dilution or matrix spike failures. | To verify the absence of an interference, the spike recovery must be between 85% and 115%. Results outside the acceptance limits require a MSA for all samples within the batch. |
| **Retention time window position establishment for each analyte (and surrogate) (all chromatographic methods only)** | Determination of the placement of the retention time window (i.e., start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the initial calibration. | To identify analytes of interest. | Incorrect window position may result in false negatives, require additional manual integrations, and/or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. |
| **Retention time window verification for each analyte (and surrogate) (all chromatographic methods only)** | A standard is used to verify that the width and position of the retention time windows are valid so that accurate analyte identification can be made during sample analysis. | To minimize the occurrence of both false positive and false negative results at each calibration verification. | The peaks from the standard used are compared to the retention time window established during the initial calibration (ICAL) to verify that the analytes of interest still fall within the window. |
| **Retention time window width calculated for each analyte (and surrogate) (non-MS chromatographic methods only)** | Determination of the length of time between sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or group of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes. | To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results. | Used to evaluate continued system performance. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis. |
| **Second source calibration verification** | A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration. (QSM) | To verify the accuracy of the initial calibration. | The concentration of the second-source calibration verification, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin. |

**TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (CONTINUED)**

| QC Check | Definition | Purpose | Evaluation |
|---|---|---|---|
| **Surrogate spike (organic analysis only)**<br><br>**Similar to recovery standards (Method 8280A only)** | A pure substance with properties that mimic the analyte of interest. Surrogates are compounds unlikely to be found in environmental samples and are added to samples to evaluate analytical efficiency by measuring their percent recovery. (Modified G-5 and CLP) | To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix.<br><br>Because surrogates are generally added to each sample in a batch, they can be used to monitor recovery on a sample-specific, rather than batch-specific basis. | Whereas the matrix spike is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS, matrix spike), the bias in the analytical system can be determined. |
| **Tuning (mass spectrometer methods only)** | The analysis of a standard compound to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis. (COE) | To verify the proper working of the mass spectrometer. | Proper tuning of the mass spectrometer must be verified prior to sample analysis. |

As always, project-specific requirements identified by the client supersede any requirements listed in the following tables. The requirements are meant to be the default, to be used when project-specific direction based on DQOs is not included.

Tables B-2 through B-10 are organized in most cases by instrument type. The applicable methods are specified in the table title. When there are exceptions (i.e., the QC check does not apply to all methods or instrument types in the table), they are noted in the first column of the table ("QC Check"). Each table contains the following fields (or columns):

- QC Check: The name of the QC measure that is required. If the check is only applicable to certain methods from the table, they will be noted in parentheses in this field.
- Minimum Frequency: Describes how often the QC check must be performed and, if relevant, at what point in the process (for example, prior to sample analysis). Some QC checks are only performed when another QC check fails. This will be noted in the minimum frequency field.
- Acceptance Criteria: The standard that the QC check must satisfy in order to proceed without performing corrective action. In some cases there are multiple options, all equivalently acceptable by DoD, for acceptance of a single QC check. These options will be listed and the appropriate option should be applied. There may be references to acceptance criteria published by DoD. The LCS control limits for certain methods can be found in Appendix DoD-D.
- Corrective Action: If a QC check does not meet the acceptance criteria specified in the preceding field, the corrective action field identifies what steps must be taken to ensure that the results will be valid. Requirements usually include finding the cause of failure of the acceptance criteria and rerunning the QC check. The corrective action field will also specify which other QC checks must be rerun to ensure valid data.
- Flagging Criteria: Where flagging is appropriate, the qualifier flag is listed in this field along with the criteria for using the flag. **Flagging should only be used as a last resort.** Data should only be flagged once corrective action has been performed. In many cases the field states "Flagging criteria is not appropriate." This means that corrective action must continue until the problem is solved and the QC check satisfies its acceptance criteria. Samples will not be accepted without successful completion of this QC check. This field will also specify when additional information should be detailed in the case narrative.
- Comments: This field contains further clarification of any of the previous five fields.

The following tables detail DoD-specific QC requirements for SW-846 methods, organized by instrument type:

Table B-2: Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (methods 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310 and 8330
Table B-3: Organic Analysis by Gas Chromatography/Mass Spectrometry (methods 8260 and 8270)
Table B-4: Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)
Table B-5: Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)
Table B-6: Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (methods 6010 and 7000 series)
Table B-7: Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (Method 6020)
Table B-8: Inorganic Analysis by Colorimetric Hexavalent Chromium (Method 7196)
Table B-9: Cyanide Analysis (methods 9010/9012)
Table B-10: Common Anions Analysis (Method 9056)

## TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C) | QC acceptance criteria published by DoD, if available; otherwise method-specified criteria. | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | Not applicable (NA) | This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete. |
| **Method detection limit (MDL) study** | At initial set-up and subsequently once per 12 month period; otherwise quarterly MDL verification checks shall be performed (see box D-18) | See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18). | NA | Samples cannot be analyzed without a valid MDL. |
| **Retention time (RT) window width calculated for each analyte and surrogate** | At method set-up and after major maintenance (e.g., column change) | RT width is ± 3 times standard deviation for each analyte RT from 72-hour study. | NA | NA | |
| **Breakdown check (Endrin/ DDT Method 8081 only)** | Daily prior to analysis of samples | Degradation ≤ 15% for both Endrin and DDT. | Correct problem then repeat breakdown check. | Flagging criteria are not appropriate. | No samples shall be run until degradation ≤ 15%. |

**TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)**

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Minimum five-point initial calibration for all analytes (ICAL)** | Initial calibration prior to sample analysis | One of the options below (except for Method 8082, which may only use Option 1 or 2):<br><br>Option 1: RSD for each analyte ≤ 20%<br><br>Option 2: linear least squares regression: r ≥ 0.995<br><br>Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order) | Correct problem then repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until ICAL has passed.<br><br>For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1232). In addition, a mid-level or lower standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor. |
| **Second source calibration verification** | Once after each initial calibration | Value of second source for all analytes within ± 20% of expected value (initial source) | Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has been verified. |
| **Retention time window position establishment for each analyte and surrogate** | Once per ICAL and at the beginning of the analytical shift | Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift. | NA | NA | |
| **Retention time window verification for each analyte and surrogate** | Each calibration verification standard | Analyte within established window | Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window. | Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window. | No samples shall be run without a verified retention time window at the initial verification. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO) or diesel range organics (DRO). |

## TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Calibration verification (initial [ICV] and continuing [CCV])** | ICV: Daily, before sample analysis<br>CCV: After every 10 field samples and at the end of the analysis sequence | All analytes within ± 20% of expected value from the ICAL | ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. See section 5.5.10 and box 55.<br><br>CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. | ICV: Flagging criteria are not appropriate.<br>CCV: Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification, if reanalysis is not possible. | If %D for an individual analyte is > 20%, no samples may be analyzed until the problem has been corrected. |
| **Method blank** | One per preparatory batch | No analytes detected > ½ RL. For common laboratory contaminants, no analytes detected ≥ RL. | Correct problem, then see criteria in box D-5; if required, reprep then reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch | |
| **Laboratory control sample (LCS) containing all analytes required to be reported, including surrogates** | One LCS per preparatory batch | QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D. | 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 (see full explanation in Appendix DoD-D) | If corrective action fails apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch | |
| **Matrix spike (MS)** | One MS per preparatory batch per matrix (see box D-11) | For matrix evaluation, use QC acceptance criteria specified by DoD for LCS. | Examine the project-specific DQOs. 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. | For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. |
| **Matrix spike duplicate (MSD) or sample duplicate** | One per preparatory batch per matrix | RPD ≤ 30% (between MS and MSD or sample and sample duplicate) | Examine the project-specific DQOs. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met. | The data shall be evaluated to determine the source of difference. |

**TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)**

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Surrogate spike (analytes identified in Appendix DoD-D)** | All field and QC samples | QC acceptance criteria for LCS specified by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria | For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed 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. | For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met.<br><br>For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | Alternative surrogates are recommended when there is obvious chromatographic interference. |
| **Confirmation of positive results (second column or second detector)** | All positive results must be confirmed (in Method 8081A exclude toxaphene and technical chlordane, in Method 8015B exclude GRO, DRO, and residual range organics (RRO)). | Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD $\leq$ 40%. | NA | Apply J-flag if RPD > 40% or Q-flag if sample is not confirmed. Discuss in the case narrative. | Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative. |
| **Results reported between LOD and LOQ** | NA | NA | NA | Apply J-flag to all results between LOD and LOQ. | |

## TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C). | QC acceptance criteria published by DoD, if available; otherwise method-specific criteria. | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | NA | This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete. |
| **MDL study** | At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-18) | See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18) | NA | Samples cannot be analyzed without a valid MDL. |
| **Tuning** | Prior to calibration and every 12 hours during sample analysis | Refer to method for specific ion criteria. | Retune instrument and verify. Rerun affected samples. | Flagging criteria are not appropriate | Problem must be corrected. No samples may be accepted without a valid tune. |
| **Breakdown check (DDT Method 8270C only)** | Daily prior to analysis of samples | Degradation ≤ 20% for DDT | Correct problem then repeat breakdown check | Flagging criteria are not appropriate | No samples shall be run until degradation ≤ 20%. Benzidine and pentachlorophenol should be present at their normal responses and no peak tailing should be observed. |

## TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Minimum five-point initial calibration for all analytes (ICAL)** | Initial calibration prior to sample analysis | 1. Average response factor (RF) for SPCCs:<br>VOCs - ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.<br>SVOCs - ≥ 0.050.<br>2. RSD for RFs for CCCs:<br>VOCs and SVOCs - ≤ 30% and one option below;<br>Option 1: RSD for each analyte ≤ 15%<br>Option 2: linear least squares regression r ≥ 0.995<br>Option 3: non-linear regression - coefficient of determination (COD) $r^2$ ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order) | Correct problem then repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until ICAL has passed. |
| **Second source calibration verification** | Once after each initial calibration | Value of second source for all analytes within ± 25% of expected value (initial source) | Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has been verified. |
| **Retention time window position establishment for each analyte and surrogate** | Once per ICAL | Position shall be set using the midpoint standard of the initial calibration curve. | NA | NA | |
| **Evaluation of relative retention times (RRT)** | With each sample | RRT of each target analyte in each calibration standard within ± 0.06 RRT units. | Correct problem, then rerun ICAL. | Flagging criteria are not appropriate. | |

## TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Calibration verification (CV)** | Daily, before sample analysis, and every 12 hours of analysis time | 1. Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050.<br>2. %Difference/Drift for CCCs: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration.) | Correct problem, then rerun CV. If that fails, repeat initial calibration. See section 5.5.10 and DoD clarification box 55. | Apply Q-flag if no sample material remains and analyte exceeds criteria. | |
| **Internal standards verification** | In all field samples and standards | Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL<br>EICP area within - 50% to + 100% of ICAL midpoint standard | Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. | If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards. | Sample results are not acceptable without a valid IS verification. |
| **Method blank** | One per preparatory batch | No analytes detected > ½ RL. For common laboratory contaminants, no analytes detected > RL. | Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. | |
| **LCS containing all analytes required to be reported, including surrogates** | One LCS per preparatory batch | QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D. | 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. (See full explanation in Appendix DoD-D.) | If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | |
| **MS** | One MS per preparatory batch per matrix (see box D-15) | For matrix evaluation, use QC acceptance criteria specified by DoD for LCS. | Examine the project-specific DQOs. 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. | 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. |

## TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **MSD or sample duplicate** | One per preparatory batch per matrix | RPD ≤ 30% (between MS and MSD or sample and sample duplicate) | Examine the project-specific DQOs. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met. | The data shall be evaluated to determine the source of difference. |
| **Surrogate spike (analytes identified in Appendix DoD-D)** | All field and QC samples | QC acceptance criteria for LCS published by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria. | For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. | For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met.<br><br>For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | |
| **Results reported between LOD and LOQ** | NA | NA | NA | Apply J-flag to all results between LOD and LOQ. | |

## TABLE B-4. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETRY (METHOD 8280)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C). | QC acceptance criteria established in-house by laboratory. | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | NA | This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete. |
| **MDL study** | At initial set-up and quarterly MDL verification checks shall be performed (see box D-18). | See 40 CFR 136B. MDL verification check must produce a signal at least 3 times the instrument's noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18). | NA | Samples cannot be analyzed without a valid MDL.<br>Refer to Sample Estimated Detection Limit. |
| **Tuning (MS methods only)** | Prior to analyzing calibration standards | Verify MS calibration per 8280A (7.13.1). | Retune instrument; verify. | Flagging criteria are not appropriate. | |
| **Retention time window defining mix** | At method set-up and prior to analyzing calibration standards | Verify descriptor switching times per 8280A (7.13.2). | Correct problem then repeat retention time window defining mix. | Flagging criteria are not appropriate. | |
| **GC column performance check (SP-2331 confirmation columns)** | Prior to initial calibration or calibration verification standards and for each 12-hour period of sample analysis | Peak separation between 2,3,7,8-TCDD and other TCDD isomers: Resolved with a valley of ≤ 25%, per 8280A (7.12.2)<br><br>For calibration verification standard only: Peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD must be resolved with a valley of ≤ 50%, per 8280A (7.13.3.6.1) | Correct problem then repeat column performance check. | Flagging criteria are not appropriate. | Needed only if using a column other than DB-5. |

## TABLE B-4. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETRY (METHOD 8280) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Column performance check (for DB-5 columns)** | Included with the ICAL standard (CC3) or the calibration verification standard | Peak separation of standard CC3: Peak between the 2,3,7,8-TCDD and 1,2,3,4-TCDD must be resolved with a valley of ≤ 25%, per 8280A (7.12.1).<br><br>For calibration verification standard only:<br>Peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD must be resolved with a valley of ≤ 50%, per 8280A (7.13.3.6.1) | Correct problem then repeat column performance check. | Flagging criteria are not appropriate. | |
| **Initial calibration for all analytes identified in Table 1 of Method 8280A (ICAL)** | Initial calibration prior to sample analysis and as needed by the failure of calibration verification standard. | Ion abundance ratios in accordance with 8280A Table 9 (7.13.3.1.1)<br>and<br>RSD of the RFs ≤ 15% for labeled IS and unlabeled PCDD/PCDF per 8280A (7.13.3.4) | Correct problem then repeat initial calibration | Apply Q-flag to all analytes with RSD > 15%. | Problem must be corrected. No samples may be run until ICAL has passed. |
| **Calibration verification**<br><br>**(Table 4 of Method 8280A – final concentrations of Standard CC3 of Table 1)** | At the beginning of each 12-hour period of sample analysis, after successful GC and MS resolution checks | Ion abundance in Table 9 of 8280A must be met for all PCDD/PCDF peaks, including labeled internal and recovery standards,<br>and<br>Sensitivity criteria of an S/N ratio > 2.5 for unlabeled PCDD/PCDF ions and > 10 for labeled internal and recovery standards per 8280A (7.13.3.6.3)<br>and<br>RF for each analyte and IS within ± 30% (% difference) of RF established in initial calibration | Correct problem, then rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples analyzed since the last successful calibration verification. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration verification has passed. |

## TABLE B-4. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETRY (METHOD 8280) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Sensitivity check**<br><br>**(Standard CC1 of Table 1 of Method 8280A)** | At the end of 12-hour sample analysis period or at the end of analysis (whichever comes first)<br><br>(Injection must be done within the 12-hour period.) | See criteria for retention time check, ion abundances, and S/N ratios noted above for calibration and response verification standard per 8280A (7.13.3.7) | 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 criteria are not appropriate. | Nondetects and samples with positive results above the method quantitation limit do not need to be reanalyzed. |
| **Method blank** | One per preparatory batch | No analytes detected ≥ LOD for the analyte or ≥ 5% of the associated regulatory limit for the analyte or ≥ 5% of the sample result for the analyte, whichever is greater, per 8280A (8.4.3) | Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to the result for specific analyte(s) in all samples in the associated preparatory batch | |
| **LCS containing analytes identified in Table 5 of Method 8280A** | One LCS per preparatory batch | QC acceptance criteria specified by DoD; see box D-7 and Appendix DoD-D. | 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. (See full explanation in Appendix DoD-D.) | If corrective action fails or if insufficient sample is available for reanalysis, apply Q-flag to specific analytes in all samples in the associated preparatory batch. | LCS compounds are the same as the MS compounds identified in Table 5 of Method 8280A (8.4.2). |
| **MS containing analytes identified in Table 5 of Method 8280A** | One MS per preparatory batch per matrix (see box D-15) | For evaluation of MS, use QC acceptance criteria specified by DoD for LCS. | Examine the project-specific DQOs. 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. | Check other QC measures to verify matrix interference. For instance, verify that the LCS shows control of the batch analysis. Also verify sample recoveries for the internal standards, recovery, and cleanup standards for an indication of potential impact. |
| **MSD or sample duplicate** | One per preparatory batch per matrix | RPD ≤ 20% (between MS and MSD or sample and sample duplicate) | Examine the project-specific DQOs. 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. | MSD spike includes the MS compounds identified in Table 5 of Method 8280A. |
| **Internal standards (IS) identified in Table 3 of Method 8280A** | Every field sample, standard, and QC sample | % recovery for each IS in the original sample (prior to any dilutions) must be within 25-150%, per 8280A (7.15.5.2) | Correct problem, then reprep and reanalyze the sample(s) with failed IS. | Apply Q-flag to results of all affected samples. | |

## TABLE B-4. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETRY (METHOD 8280) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Sample PCDD/PCDF identification** | Identify all positive sample detections per 8280A (7.14.5) | Verify that absolute RT at maximum height is within -1 to +3 secs. 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 8280A (7.14.5.1)<br>and<br>Absolute RTs of the recovery standards must be within ±10 sec. of those in the calibration verification standard (7.14.5.1)<br>and<br>All ions listed in Table 8 of Method 8280A must be present in the SICP, must maximize simultaneously (+2 sec.), and must have not saturated the detector (7.14.5.2)<br>and<br>S/N ratio of IS's ≥ 10 times background noise. Remaining ions on Table 8 must have an S/N ratio ≥ 2.5 times the background noise (7.14.5.3)<br>and<br>Ion abundance in Table 9 of 8280A must be met for all analytes, internal, and recovery standards (7.14.5.4) | Correct problem, then reprep and reanalyze the sample(s) with failed criteria for any of the internal, recovery, or cleanup standards.<br><br>If PCDPE is detected or if sample peaks present do not meet all identification criteria, calculate the EMPC (estimated maximum possible concentration) according to 8280A (7.15.7). | Flagging criteria is not appropriate. | |

## TABLE B-4. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETRY (METHOD 8280) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Sample specific estimated detection limit (EDL)** | Calculated for each 2,3,7,8-substituted isomer that was not identified | Per 8280A (7.15.6) | NA | NA | |
| **Sample estimated maximum possible concentration (EMPC)** | Determined for each 2,3,7,8-substituted isomer that did not meet ion abundance ratio criteria (Table 9, Method 8280A) or PCDFs where peak representing a corresponding PCDPE was detected | Response for both quantitation ions must be ≥ 2.5 times S/N ratio of background (7.15.7); all other criteria from sample PCDD/PCDF identification above; PCDE peak at the same RT ( ± 2 sec.) must have S/N < 2.5 | NA | NA | |
| **Sample 2,3,7,8-TCDD toxicity equivalents (TE) concentration** | All positive detections | Per 8280A (7.15.8) | NA | NA | Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD. |
| **Results reported between LOD and LOQ** | Positive detections calculated per 8280A (7.15.1) | NA | NA | Apply J-flag to all results between LOD and LOQ. | |

## TABLE B-5. 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 |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C) | QC acceptance criteria established in-house by laboratory. | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | NA | This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete |
| **MDL study** | At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-18). | See 40 CFR 136B. MDL verification check must produce a signal at least 3 times the instrument's noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18). | NA | Samples cannot be analyzed without a valid MDL. Refer to Sample EDL. |
| **Tuning** | At the beginning and the end of each 12-hour period of analysis | Static resolving power ≥ 10,000 (10% valley) for identified masses per 8290 (7.6.2.2 and 8.2.2.1/8.2.2.3), <u>and</u><br>Lock-mass ion between lowest and highest masses for each descriptor and level of reference compound ≤10% full-scale deflection, per 8290 (8.2.2.2) | Retune instrument; verify. Rerun affected samples. | Flagging criteria are not appropriate. | Recommend that a check of static resolution also be documented before and after each analysis. |
| **GC column performance check** | Prior to initial calibration or calibration verification.<br><br>Use GC performance check solution (see Table 7 of Method 8290). | Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of ≤ 25%, per 8290 (8.2.1.2)<br><u>and</u><br>Identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram (8.2.1.2) | Correct problem then repeat column performance check. | Flagging criteria are not appropriate. | |

## TABLE B-5. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (METHOD 8290) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **GC column performance check (continued)** | | and<br>Absolute retention times for switching from one homologous series to the next ≥ 10 sec. For all components of the mixture (8.2.1.3) | | | |
| **Initial calibration for all analytes identified in Table 5 of Method 8290 (ICAL)** | Initial calibration prior to sample analysis, as needed by the failure of calibration verification standard, and when a new lot is used as standard source for HRCC-3, sample fortification (IS), or recovery solutions | Ion abundance ratios in accordance with criteria in Table 8 of Method 8290 (7.7.1.4.1/7.7.2.3)<br>and<br>S/N ratio ≥ 10 for all target analyte ions (7.7.2.2)<br>and<br>RSD ≤ 20% for the response factors (RF) for all 17 unlabeled standards and RSD ≤ 30% for the RFs for the 9 labeled IS (7.7.2.1) | Correct problem, then repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until ICAL has passed. |
| **Routine (continuing) calibration check**<br><br>**(Table 5 of 8290 - final concentrations of HRCC3)** | At the beginning of each 12-hour period, and at the end of each analytical sequence | Ion abundance ratios in accordance with criteria in Table 8 of Method 8290 (7.7.4.3)<br>and<br>For unlabeled standards, RF within ± 20% D of RF established in initial calibration (7.7.4.1)<br>and<br>For labeled standards, RF within ± 30% D of RF established in initial calibration (7.7.4.2) | Correct problem, repeat calibration verification standard one more time. If that fails, then repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.<br><br>Evaluation of corresponding labeled/unlabeled standards may impact the corrective action required (see 8290 section 7.7.4.4).<br><br>End-of-run CCV: If the RF for unlabeled standards ≤ 25% D and the RF for labeled standards ≤ 35% D (relative to the RF established in the initial calibration), the mean RF from the two daily CCVs | Flagging criteria are not appropriate for routine calibration check.<br>Q-flag (noncompliances) for end-of-run continuing calibration check. | Problem must be corrected. No samples may be run until routine (beginning of 12-hour period) calibration verification has passed. |

## TABLE B-5. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (METHOD 8290) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Routine (continuing) calibration check (continued)** | | | must be used for quantitation of impacted samples instead of the ICAL mean RF value. (8.3.2.4)<br><br>If ending CCV RF > 25% D or > 35% D for unlabeled and labeled standards, respectively, a new ICAL must be run immediately (within 2 hr.). Reanalyze samples with positive detections, if necessary. | | |
| **Method blank** | One per preparatory batch, run after calibration standards and before samples | No analytes detected > ½ RL | Correct problem then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to the result for specific analyte(s) in all samples in the associated preparatory batch. | |
| **Laboratory duplicate** | One per preparatory batch | RPD ≤ 25%, per 8290 (8.3.5.1.1) | Refer to MS. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met. | |
| **Matrix spike (MS)** | One MS per preparatory batch per matrix (see box D-11) | QC acceptance criteria for lab's in-house control limits | Check other QC measures to verify matrix interference. For instance, verify that the PT sample shows control of the batch analysis. Also verify sample recoveries for the internal, recovery and cleanup standards for an indication of potential impact. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met. | MS spike includes all compounds identified in Table 5 of Method 8290 at the concentration corresponding to HRCC-3 standard. |
| **Matrix spike duplicate (MSD) or sample duplicate** | One per preparatory batch per matrix | RPD ≤ 20%, per 8290 (8.3.6.4) | Refer to MS. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met. | MSD spike includes all compounds identified in Table 5 of Method 8290 at the concentration corresponding to HRCC-3 standard. |

## TABLE B-5. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (METHOD 8290) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Field blanks and/or rinsates** | Per project requirements (see 8290 section 8.3.4) | Per project requirements | Per project requirements | Per project requirements | |
| **Proficiency Testing (PT) sample** | Per project requirements (see 8290 section 8.3.1) | Per project requirements | Correct problem, then reprep and reanalyze the PT and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient sample material is available. | If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | |
| **Internal standards (IS) identified in Table 2 of Method 8290** | Every field sample, standard, and QC sample | %Recovery for each IS in the original sample (prior to dilutions) must be within 40-135% per 8290 (8.4) | Correct problem, then reprep and reanalyze the sample(s) with failed IS. | Apply Q-flag to results of all affected samples. | |
| **Sample PCDD/PCDF identification** | Identify all positive sample detections per 8290 (7.8.4) | <u>2,3,7,8-substituted isomers with labeled standards:</u> Absolute RT at maximum height within -1 to +3 seconds of that for corresponding labeled standard<br><u>2,3,7,8-substituted isomers with unlabeled standards:</u> RRT within 0.005 RRT units of that in calibration verification standard<br><u>Non-2,3,7,8-substituted isomers:</u> RT within RT window established by column performance check solution for corresponding homologue, per 8290 (7.8.4.1)<br><u>and</u><br>Ions for quantitation must maximize simultaneously (± 2 sec.)<br><u>and</u><br>Ion abundance ratios in accordance with criteria on Table 8 of 8290 (7.8.4.2),<br><u>and</u><br>S/N ratio of IS's ≥ 10 times background noise | Correct problem, then reprep and reanalyze the sample(s) with failed criteria for any of the internal, recovery, or cleanup standards.<br><br>If PCDPE is detected or if sample peaks present do not meet ion abundance ratio criteria, calculate the EMPC (estimated maximum possible concentration) according to 8290 (7.9.5.2). | Flagging criteria are 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) (see 8290 section 3.4) |

## TABLE B-5. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (METHOD 8290) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Sample PCDD/PCDF identification (continued)** | | and<br>S/N ratio of all remaining ions for unlabeled analytes ≥ 2.5 times background noise (7.8.4.3)<br>and<br>For PCDF: No signal present having a S/N ratio ≥ 2.5 for the corresponding ether (PCDPE) detected at the same retention time ( ± 2 sec) (7.8.4.4) | | | |
| **Sample specific estimated detection limit (EDL)** | For each 2,3,7,8-substituted isomer that is not identified | Per 8290 (7.9.5) | NA | NA | |
| **Sample estimated maximum possible concentration (EMPC)** | Every sample that indicates a detection ≥ 2.5 times S/N response | Identification criteria in 8290 (7.4.5) must be met, and response for both quantitation ions must be ≥ 2.5 times S/N ratio for background (7.9.5.2.1). | NA | NA | |
| **Sample 2,3,7,8-TCDD toxicity equivalents (TE) concentration** | All positive detections, as required | Per 8290 (7.9.7) | NA | NA | Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD |
| **Results reported between LOD and LOQ** | Positive detections calculated per 8290 (7.9.1) | NA | NA | Apply J-flag to all results between LOD and LOQ. | |

## TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C) | QC acceptance criteria published by DoD, if available; otherwise method-specified criteria | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | NA | This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete. |
| **MDL study** | At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-18). | See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18). | NA | Samples cannot be analyzed without a valid MDL. |
| **Instrument detection limit (IDL) study (ICP only)** | At initial set-up and after significant change | Detection limits established shall be ≤ MDL. | NA | NA | Samples cannot be analyzed without a valid IDL. |
| **Linear dynamic range or high-level check standard (ICP only)** | Every 6 months | Within ± 10% of expected value | NA | NA | |

## TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Initial calibration for all analytes (ICAL)**<br><br>**(ICP: minimum one high standard and a calibration blank; GFAA: minimum three standards and a calibration blank; CVAA: minimum 5 standards and a calibration blank)** | Daily initial calibration prior to sample analysis | ICP: No acceptance criteria unless more than one standard is used, in which case r ≥ 0.995.<br><br>GFAA: r ≥ 0.995<br><br>CVAA: r ≥ 0.995 | Correct problem and repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until ICAL has passed. |
| **Second source calibration verification (ICV)** | Once after each initial calibration, prior to sample analysis | Value of second source for all analyte(s) within ± 10% of expected value (initial source) | Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has been verified. |
| **Continuing calibration verification (CCV)** | After every 10 samples and at the end of the analysis sequence | ICP: within ± 10% of expected value<br>GFAA: within ± 20% of expected value<br>CVAA: within ± 20% of expected value | Correct problem, rerun calibration verification. If that fails, then repeat initial calibration.<br>Reanalyze all samples since the last successful calibration verification. | Flagging criteria are not appropriate. | Problem must be corrected. Results may not be reported without a valid CCV. |
| **Low-level calibration check standard (ICP only)** | Daily, after one-point initial calibration | Within ±20% of expected value | Correct problem, then reanalyze. | Flagging criteria are not appropriate. | No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit. |

## TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Method blank** | One per preparatory batch | No analytes detected > ½ RL<br>For common laboratory contaminants, no analytes detected > RL | Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. | |
| **Calibration blank** | Before beginning a sample run, after every 10 samples, and at end of the analysis sequence | No analytes detected > 2 x MDL | Correct problem, then reprep and reanalyze calibration blank and previous 10 samples | Apply B-flag to all results for specific analyte(s) in all samples associated with the blank. | |
| **Interference check solutions (ICS) (ICP only)** | At the beginning of an analytical run | ICS-A:<br>Absolute value of concentration for all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes)<br>ICS-AB:<br>Within ± 20% of expected value | Terminate analysis; locate and correct problem; reanalyze ICS. | Flagging criteria are not appropriate. | No samples may be analyzed without a valid ICS. |
| **LCS containing all analytes required to be reported** | One LCS per preparatory batch | QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D. | 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. (See full explanation in Appendix DoD-D.) | If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | |
| **Dilution test** | Each preparatory batch or when a new or unusual matrix is encountered | Five-fold dilution must agree within ± 10% of the original determination | ICP: Perform post-digestion spike (PDS) addition<br><br>GFAA: Perform recovery test<br><br>CVAA: Perform matrix spike | Flagging criteria are not appropriate. | Only applicable for samples with concentrations > 50 x MDL (ICP) or > 25 x MDL (GFAA and CVAA). |
| **Post-digestion spike (PDS) addition (ICP only)** | When dilution test fails or analyte concentration in all samples < 50 x MDL | Recovery within 75-125% of expected result. | Run samples by method of standard additions (MSA) or see flagging criteria. | Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition. | The spike addition should produce a level between 10 - 100 x MDL. |

## TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Recovery test (GFAA only)** | When dilution test fails or analyte concentration in all samples < 25 x MDL | Recovery within 85-115% of expected result. | Run samples by method of standard addition (MSA) or see flagging criteria. | Apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85-115% range. | |
| **Method of standard additions (MSA) or Internal standard calibration** | When matrix interference is suspected | NA | NA | NA | Document use of MSA in the case narrative. |
| **MS** | One MS per preparatory batch per matrix (see box D-15) | For matrix evaluation, use QC acceptance criteria specified by DoD for LCS. | Examine the project-specific DQOs. 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. | 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. |
| **MSD or sample duplicate** | One per preparatory batch per matrix | RPD $\leq$ 20% (between MS and MSD or sample and sample duplicate) | Examine the project-specific DQOs. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met. | The data shall be evaluated to determine the source of difference. |
| **Results reported between LOD and LOQ** | NA | NA | NA | Apply J-flag to all results between LOD and LOQ | |

## TABLE B-7. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY (METHOD 6020)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel or test method (see Appendix C) | QC acceptance criteria published by DoD, if available; otherwise method-specified criteria | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | NA | This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete. |
| **MDL study** | At initial set-up and once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-18) | See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18). | NA | Samples cannot be analyzed without a valid MDL. |
| **IDL study** | At initial set-up and after significant change | Detection limits established shall be ≤ MDL. | NA | NA | Samples cannot be analyzed without a valid IDL. |
| **Tuning** | Prior to initial calibration | Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height; For stability, RSD ≤ 5% for at least four replicate analytes | Retune instrument then reanalyze tuning solutions. | Flagging criteria are not appropriate. | No analysis shall be performed without a valid MS tune. |
| **Initial calibration (ICAL)**<br><br>**(minimum one high standard and a calibration blank)** | Initial calibration prior to sample analysis | If more than one calibration standard is used, r ≥ 0.995 | Correct problem, then repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until ICAL has passed. |
| **Second source calibration verification** | Once after each ICAL, prior to beginning a sample run | Value of second source for all analytes within ± 10% of expected value (initial source) | Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has been verified. |
| **Low-level calibration check standard** | Daily, after one-point initial calibration | Within ± 20% of expected value | Correct problem, then reanalyze. | Flagging criteria are not appropriate. | No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit. |

## TABLE B-7. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY (METHOD 6020) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Continuing calibration verification (CCV)** | After every 10 samples and at the end of the analysis sequence | All analytes within ± 10% of expected value | Correct problem, rerun calibration verification. If that fails, then repeat initial calibration.<br>Reanalyze all samples since the last successful calibration verification. | Flagging criteria are not appropriate. | Problem must be corrected. Results may not be reported without a valid CCV. |
| **Linear dynamic range or high-level check standard** | Every 6 months | Within ±10% of expected value | NA | NA | |
| **Method blank** | One per preparatory batch | No analytes detected > ½ RL. For common laboratory contaminants, no analytes detected > RL. | Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. | |
| **Calibration blank** | Before beginning a sample run, after every 10 samples, and at end of the analysis sequence | No analytes detected > 2 x MDL | Correct problem, then reprep and reanalyze calibration blank and previous 10 samples. | Apply B-flag to all results for specific analyte(s) in all samples associated with the blank. | |
| **Interference check solutions (ICS-A and ICS-AB)** | At the beginning ofan analytical run | ICS-A:<br>Absolute value of concentration for all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes)<br><br>ICS-AB:<br>Within ±20% of expected value | Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all affected samples. | If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS. | |
| **LCS containing all analytes required to be reported** | One LCS per preparatory batch | QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D | 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. (See full explanation in Appendix DoD-D.) | If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | |

## TABLE B-7. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY (METHOD 6020) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Dilution test** | Each preparatory batch | Five-fold dilution must agree within ± 10% of the original measurement | Perform post-digestion spike addition. | Flagging criteria are not appropriate. | Only applicable for samples with concentrations > 100 x MDL. |
| **Post digestion spike addition** | When dilution test fails or analyte concentration for all samples < 100 x MDL | Recovery within 75-125% of expected results | Run samples by method of standard additions (MSA) or see flagging criteria. | Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition. | |
| **Method of standard additions (MSA)** | When matrix interference is suspected | NA | NA | NA | Document use in the case narrative. |
| **MS** | One MS per preparatory batch per matrix (see box D-15) | For matrix evaluation, use QC acceptance criteria specified by DoD for LCS. | Examine the project-specific DQOs. 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. | 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. |
| **MSD or sample duplicate** | One per preparatory batch per matrix | RPD < 20% (between MS and MSD or sample and sample duplicate) | Examine the project-specific DQOs. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met. | The data shall be evaluated to determine the source of difference. |
| **Internal standards (IS)** | Every sample | IS intensity within 30-120% of intensity of the IS in the initial calibration | Perform corrective action as described in Method 6020 (8.3). | Flagging criteria are not appropriate. | |
| **Results reported between LOD and LOQ** | NA | NA | NA | Apply J-flag to all results between LOD and LOQ. | |

## TABLE B-8. INORGANIC ANALYSIS BY COLORIMETRIC HEXAVALENT CHROMIUM (METHOD 7196)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel or test method (see Appendix C). | QC acceptance criteria published in method; otherwise QC acceptance criteria established in-house by laboratory. | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | NA | This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete. |
| **MDL study** | At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-18) | See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18). | NA | Samples cannot be analyzed without a valid MDL. |
| **Reference blank (reagent water)** | Before beginning standards or sample analysis | NA | NA | NA | Used for blank subtraction of standards, field and QC samples.<br>For turbid field samples, a turbidity blank must be used instead of the reference blank (using a sample aliquot prepped in accordance with 7196A (7.1)) |
| **Initial calibration (ICAL)**<br><br>**(minimum three standards and a calibration blank)** | Daily initial calibration prior to sample analysis | $r \geq 0.995$ | Correct problem and repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until ICAL has passed. |
| **Second source calibration verification (ICV)**<br><br>**(also known as independently prepared check standard)** | Before beginning a sample run | Value of second source within ± 10% of expected value (initial source) | Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat calibration.<br>Reanalyze all samples since last successful calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has been verified. |

## TABLE B-8. INORGANIC ANALYSIS BY COLORIMETRIC HEXAVALENT CHROMIUM (METHOD 7196) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Continuing calibration verification (CCV)** | After every 15 samples and at the end of the analysis sequence | Value of CCV within ± 10% of expected value (ICV) | Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has been verified. |
| **Sample matrix verification**<br><br>**(also known as matrix spike)** | Once for every sample matrix analyzed | Spike recovery within 85-115% | If check indicates interference, dilute and reanalyze sample; persistent interference indicates the need to use alternative method or analytical conditions, or to use method of standard additions. | Flagging criteria are not appropriate. | Verification check ensures lack of reducing condition or interference from matrix. Additional corrective actions are identified in Method 7196A (7.4 and 7.5). |
| **Method blank** | One per preparatory batch | No analytes detected > ½ RL | Correct problem then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. | |
| **LCS** | One LCS per preparatory batch | QC acceptance criteria specified by DoD; see box D-7 and Appendix DoD-D | Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix DoD-D). | If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | |
| **MSD or sample duplicate** | Aqueous matrix: One per every 10 project samples per matrix<br>Solid matrix: One per preparatory batch per matrix | Aqueous matrix: RPD ≤ 20% (between MS and MSD or sample and sample duplicate)<br>Solid matrix: RPD ≤ 30% | Examine project-specific DQOs. 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 not met. | Refer to sample matrix verification sample for MS data evaluation. |
| **Pre-digestion matrix spikes (solid matrix samples only, Method 3060)** | One soluble and insoluble pre-digestion MS analyzed per preparatory batch prior to analysis | MS recoveries within 75-125% | Correct problem and rehomogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results. | If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | |
| **Results reported between LOD and LOQ** | NA | NA | NA | Apply J-flag to all results between LOD and LOQ. | |

## TABLE B-9. CYANIDE ANALYSIS (METHODS 9010/9012)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C) | QC acceptance criteria published by DoD, if available; otherwise use method-specified criteria. | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | NA | This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete. |
| **MDL study** | At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-18). | See 40 CFR 136B. MDL verification check must produce a signal at least 3 times the instrument's noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18). | NA | Samples cannot be analyzed without a valid MDL |
| **Multipoint initial calibration curve**<br><br>**(six standards and a calibration blank)** | Initial daily calibration prior to sample analysis | Correlation coefficient ≥ 0.995 for linear regression | Correct problem, then repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has passed. |
| **Distilled standards**<br><br>**(one high and one low)** | Once per multipoint calibration | Within ± 15% of true value | Correct problem, then repeat distilled standards. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until distilled standards have passed. |
| **Second source calibration verification check standard** | Once after each multipoint calibration | Value of second source within ± 15% of expected value (initial source) | Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has been verified. |
| **Method blank** | One per preparatory batch | No analytes detected > ½ RL | Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. | |

## TABLE B-9. CYANIDE ANALYSIS (METHODS 9010/9012) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **LCS** | One LCS per preparatory batch | QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D | Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix DoD-D). | If corrective action fails apply Q-flag to the specific analyte in all samples in the associated preparatory batch. | |
| **MS/MSD** | For 9010: one MS/MSD pair per preparatory batch per matrix<br>For 9012: one MS/MSD pair per every 10 samples per matrix | For matrix recovery evaluation, use QC acceptance criteria specified by DoD for LCS.<br>For precision evaluation, RPD ≤ 20% (between MS or MSD). | Examine the project-specific DQOs. Contact the client as to additional measures to be taken. | For the specific analyte in the parent sample, apply J-flag if acceptance criteria are not met. | 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. If RPD > 20%, evaluate data to determine source of the difference between the MS and MSD. |
| **Sample duplicate (replicate)** | Once per every 20 samples | %D of duplicate within ± 20% of sample | Correct problem and reanalyze sample and duplicate | Apply Q-flag if sample cannot be rerun or reanalysis does not correct problem. | |
| **Results reported between LOD and LOQ** | NA | NA | NA | Apply J-flag to all results between LOD and LOQ. | |

## TABLE B-10. COMMON ANIONS ANALYSIS (METHOD 9056)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Demonstrate acceptable analyst capability** | Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C) | QC acceptance criteria published by DoD, if available; otherwise use method-specified criteria. | Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f). | NA | This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is completed. |
| **MDL study** | At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-18). | See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level. | Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18). | NA | Samples cannot be analyzed without a valid MDL. |
| **Retention time window width calculated for each analyte** | After method set-up and after major maintenance (e.g., column change) | RT width is ± 3 times standard deviation for each analyte over 24-hour period. | NA | NA | |
| **Multipoint calibration for all analytes**<br><br>**(minimum three standards and one calibration blank)** | Initial calibration prior to sample analysis | Correlation coefficient ≥ 0.995 for linear regression. | Correct problem, then repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No sample may be run until calibration has passed. |
| **Second source calibration verification** | Once after each multipoint calibration | Value of second source for all analytes within ± 10% of expected value (initial source). | Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. | Flagging criteria are not appropriate. | Problem must be corrected. No samples may be run until calibration has been verified. |
| **Retention time window position establishment for each analyte** | Once per multipoint calibration | Position shall be at midpoint of initial calibration curve. | NA | NA | |
| **Retention time window verification for each analyte** | Each calibration verification | Analyte within established window. | Correct problem, then reanalyze all samples analyzed since the last retention time check. If they fail, redo ICAL and reset retention time window. | Flagging criteria are not appropriate. | No samples shall be run without a verified retention time window. |

## TABLE B-10. COMMON ANIONS ANALYSIS (METHOD 9056) (CONTINUED)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|---|---|---|
| **Initial calibration verification (ICV)** | Daily before sample analysis, when eluent is changed, and with every batch of samples | All analytes within ± 10% of expected value and retention times within appropriate windows | Correct problem, rerun ICV. If that fails, then repeat initial calibration (see section 5.5.10 and box 59). | Flagging criteria are not appropriate. | No samples may be run without verifying initial calibration. |
| **Midrange continuing calibration verification (CCV)** | After every 10 samples and at the end of the analysis sequence | Instrument response within ± 10% of expected value | Correct problem, then repeat CCV and reanalyze all samples since last successful calibration verification | Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. | |
| **Method blank** | One per preparatory batch | No analytes detected > ½ RL For common laboratory contaminants, no analysis detected > RL | Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. | |
| **LCS containing all analytes required to be reported** | One LCS per preparatory batch | QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D. | 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. (See full explanation in Appendix DoD-D). | If corrective action fails apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. | |
| **MS** | One MS per preparatory batch per matrix (see box D-15) | For matrix evaluation, use QC acceptance criteria specified by DoD for LCS. | Examine the project-specific DQOs. 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. | 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. |
| **MSD** | One per preparatory batch per matrix | RPD ≤ 20% (between MS and MSD) | Examine the project-specific DQOs. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met. | The data shall be evaluated to determine the source of difference. |
| **Sample Duplicate (replicate)** | One per every 10 samples | %D ≤ 10% (between sample and sample duplicate) | Correct problem and reanalyze sample and duplicate. | If corrective action fails, apply Q-flag to specific analyte(s) in the sample. | |
| **Results reported between LOD and LOQ** | NA | NA | NA | Apply J-flag to all results between LOD and LOQ. | |

## ACRONYMS FOR APPENDIX DOD-B

**CC3:** The third of five solutions for instrument calibration used in Method 8280
**CCC:** Calibration check compounds
**CCV:** Continuing calibration verification
**CFR:** Code of Federal Regulations
**COD:** Coefficient of determination
**COE:** Army Corps of Engineers
**CV:** Calibration verification
**CV-IS:** Calibration verification of internal standards
**D:** Difference or drift
**DDT:** 2,2-bis(p-chlorophenyl)-1,1,1-trichloroethane/dichlorodiphenyl-trichloroethane/p,p'-DDT
**DoD:** Department of Defense
**DQO:** Data quality objective
**DRO:** Diesel range organics
**EDL:** Estimated detection limit
**EICP:** Extracted ion current profile
**GC:** Gas chromatography
**GC/MS:** Gas chromatography/mass spectrometry
**GFAA:** Graphite furnace atomic absorption spectrophotometry
**GRO:** Gasoline range organics
**HPLC:** High performance liquid chromatography
**HxCDD:** Hexachlorodibenzo-p-dioxin (solution used for calibration verification)
**ICAL:** Initial calibration
**ICP:** Inductively coupled plasma atomic emission spectrometry
**ICP/MS:** Inductively coupled plasma/mass spectrometry
**ICS:** Interference check solution
**ICV:** Initial Calibration Verification
**IS:** Internal standard
**IDL:** Instrument detection limit
**LCS:** Laboratory control sample
**LOD:** Limit of detection
**LOQ:** Limit of quantitation
**MDL:** Method detection limit
**MS:** Mass spectrometry
**MS:** Matrix spike
**MSA:** Method of standard additions
**MSD:** Matrix spike duplicate
**PCB:** Polychlorinated biphenyl
**PCDD:** Polychlorinated dibenzodioxin
**PCDF:** Polychlorinated dibenzofuran
**PDS:** Post-digestion spike
**PE:** Performance evaluation
**PT:** Proficiency testing
**QC:** Quality control
**QSM:** DoD Quality Systems Manual for Environmental Laboratories
**RF:** Response factor
**RL:** Reporting limit
**RPD:** Relative percent difference
**RRO:** Residual range organics
**RRT:** Relative retention time
**RSD:** Relative standard deviation
**RT:** Retention time
**SICP:** Selected ion current profile
**S/N:** Signal to noise ratio
**SPCC:** System performance check compound

**SVOC:** Semivolatile organic compound
**TCDD:** Tetrachlorodibenzo-p-dioxin
**TCDF:** Tetrachlorodibenzofuran
**VOC:** Volatile Organic Compound

## GLOSSARY FOR APPENDIX DOD-B

**Aliquot:** A discrete, measured, representative portion of a sample taken for analysis. (DoD; EPA QAD glossary)

**Analyte:** The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA glossary)

**Atomization:** A process in which a sample is converted to free atoms. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

**Congener:** A member of a class of related chemical compounds (e.g., PCBs, PCDDs).

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

**Duplicate:** The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

**Eluent:** A solvent used to carry the components of a mixture though a stationary phase. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

**Elute:** To extract; specifically, to remove (adsorbed material) from an adsorbent by means of a solvent. (Merriam-Webster's Collegiate Dictionary, 2000)

**Elution:** A process in which solutes are washed though a stationary phase by the movement of a mobile phase. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

**False Negative:** An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.

**False Positive:** An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

**Homologue:** One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, $CH_3OH$ (methanol), $C_2H_5OH$ (ethanol), $C_3H_7OH$ (propanol), $C_4H_9OH$ (butanol), etc., form a homologous series. (The Condensed Chemical Dictionary G.G.Hawley, ed. 1981)

**Interference, spectral:** Occurs when particulate matter from the atomization scatters the incident radiation from the source or when the absorption or emission of an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

**Interference, chemical:** Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

**Internal Standard:** A pure substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte. The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

**Isomer:** Generally, any two chemicals with the same chemical formula but a different structure. For example, hexane ($C_6H_{14}$) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane. (http://www.kcpc.usyd.edu.au/discovery/glossary-all.html)

**Matrix:** The collection of all of the various constituents making up an analytical sample. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

**Method of Standard Additions:** A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

**Retention Time:** The time between sample injection and the appearance of a solute peak at the detector. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

**Signal to Noise Ratio:** The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

**Standard:** Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by the US National Institute of Standards and Technology (NIST) and characterized for absolute content, independent of analytical test method.

## APPENDIX DOD-C – TARGET ANALYTE LISTS

The lists of analytes provided in this appendix are to be used as a default whenever no analyte list has been provided by the client. These analyte lists are to be used when the client identifies the analyses needed by general descriptions such as method number (for example, SW-846 Method 8330) and/or analyte group (for example, explosives). If a short list of specific analytes is not identified, the following target analyte lists shall be the default for those analyses identified as appropriate to the site. Throughout this manual, references to "target analytes" apply to project-specific analytes. Only when those are not available does "target analytes" apply to the lists presented in this appendix.

This appendix is not needed when DoD personnel have used site-specific information to identify project-specific target analytes. If only limited site-specific information is available, the following target analyte lists may be used as a baseline from which the client may add or subtract specific analytes to form the target analyte lists to be used by the laboratory.

**SW-846 Methods**

Although the target analyte lists in this appendix identify the associated SW-846 methods, the laboratory is not restricted to those methods when conducting analyses. If a new method that analyzes for the same group of analytes becomes available, the target analyte lists in this appendix still apply.

The surrogates listed are not meant to be comprehensive. Other surrogates may be substituted as appropriate. The laboratory must ensure that the surrogates used represent the chemical and physical properties associated with the target analytes.

The following target analyte lists were compiled by all three DoD components to include common analytes of concern at DoD sites as well as the Superfund list of 110 most frequently occurring chemicals.

The analytes are organized by analyte group and technology. The associated SW-846 method is identified for convenience only and is not meant to imply that the laboratory must conduct the analysis with these specific methods. The project-specific QAPP will identify the analytical method to be used, and the target analytes may be carried over to those different methods. In some cases analytes may be detected by multiple methods. The comment field in each table identifies alternative SW-846 method(s) listed in this appendix that can be used for analysis. The following tables list the default DoD target analytes for analyte groups commonly used by DoD:

Table C-1: Volatile Organic Compounds by GC/MS
Table C-2: Semivolatile Organic Compounds by GC/MS
Table C-3: Dioxins/Furans by GC/MS
Table C-4: Organophosphorus Pesticides by GC/FPD or NPD
Table C-5: Chlorinated Herbicides by GC/ECD
Table C-6: Polynuclear Aromatic Hydrocarbons by HPLC
Table C-7: Explosives by HPLC
Table C-8: Organochlorine Pesticides by GC/ECD
Table C-9: Polychlorinated Biphenyls by GC/ECD
Table C-10: Metals by ICP, ICP/MS, GFAA, and CVAA
Table C-11: Other Inorganics

[Note: Analytes often have many synonyms; refer to the CAS number when there is uncertainty regarding an analyte name.]

During a multi-laboratory study of laboratory control sample (LCS) recoveries, several compounds on the target analyte lists were identified as poor performing analytes for certain methods. These analytes are included in the target analyte lists in the following tables since they should be included in the calibration standard; however, they should be treated separately in the LCS. For further explanation on how to treat poor performing analytes when they are detected in the calibration or are target analytes of concern see Appendix DoD-D. The analytes are identified as poor performing analytes on the following tables. No data

were gathered for the following methods; therefore, no conclusions on analyte performance can be made: 8015, 8280, 8290, 8141, 7000 series (GFAA), 9010, 9012 and 9056.

## TABLE C-1. VOLATILE ORGANIC COMPOUNDS BY GC/MS TARGET ANALYTE LIST[2] (BASED ON SW-846 METHOD 8260)

| Volatile Organic Compound | CAS # | Comments | Volatile Organic Compound | CAS # | Comments |
|---|---|---|---|---|---|
| Acetone | 67-64-1 | | 1,1-Dichloropropene | 563-58-6 | |
| Benzene | 71-43-2 | | cis-1,3-Dichloropropene | 10061-01-5 | |
| Bromobenzene | 108-86-1 | | trans-1,3-Dichloropropene | 10061-02-6 | |
| Bromochloromethane | 74-97-5 | | Ethylbenzene | 100-41-4 | |
| Bromodichloromethane | 75-27-4 | | 2-Hexanone | 591-78-6 | |
| Bromoform | 75-25-2 | | Hexachlorobutadiene | 87-68-3 | |
| Bromomethane (Methyl bromide) | 74-83-9 | | Isopropylbenzene | 98-82-8 | |
| 2-Butanone (MEK) | 78-93-3 | | p-Isopropyltoluene | 99-87-6 | |
| n-Butylbenzene | 104-51-8 | | Methylene chloride | 75-09-2 | |
| sec-Butylbenzene | 135-98-8 | | 4-Methyl-2-pentanone (MIBK) | 108-10-1 | |
| tert-Butylbenzene | 98-06-6 | | Methyl tert-butyl Ether (MTBE) | 1634-04-4 | |
| Carbon disulfide | 75-15-0 | | Naphthalene | 91-20-3 | See also 8270 and 8310 |
| Carbon tetrachloride | 56-23-5 | | n-Propylbenzene | 103-65-1 | |
| Chlorobenzene | 108-90-7 | | Styrene | 100-42-5 | |
| Chlorodibromomethane[3] | 124-48-1 | | 1,1,1,2-Tetrachloroethane | 630-20-6 | |
| Chloroethane | 75-00-3 | | 1,1,2,2-Tetrachloroethane | 79-34-5 | |
| Chloroform | 67-66-3 | | Tetrachloroethene | 127-18-4 | |
| Chloromethane | 74-87-3 | | Toluene | 108-88-3 | |
| 2-Chlorotoluene | 95-49-8 | | 1,2,3-Trichlorobenzene | 87-61-6 | |
| 4-Chlorotoluene | 106-43-4 | | 1,2,4-Trichlorobenzene | 120-82-1 | |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | | 1,1,1-Trichloroethane | 71-55-6 | |
| 1,2-Dibromoethane (Ethylene dibromide) | 106-93-4 | | 1,1,2-Trichloroethane | 79-00-5 | |
| Dibromomethane | 74-95-3 | | Trichloroethene | 79-01-6 | |
| 1,2-Dichlorobenzene | 95-50-1 | See also 8270 | Trichlorofluoromethane | 75-69-4 | |
| 1,3-Dichlorobenzene | 541-73-1 | See also 8270 | 1,2,3-Trichloropropane | 96-18-4 | |
| 1,4-Dichlorobenzene | 106-46-7 | See also 8270 | 1,2,4-Trimethylbenzene | 95-63-6 | |
| Dichlorodifluoromethane | 75-71-8 | | 1,3,5-Trimethylbenzene | 108-67-8 | |
| 1,1-Dichloroethane | 75-34-3 | | Vinyl chloride | 75-01-4 | |
| 1,2-Dichloroethane | 107-06-2 | | o-Xylene | 95-47-6 | |
| 1,1-Dichloroethene | 75-35-4 | | m,p-Xylene | 108-38-3/ 106-42-3 | |
| cis-1,2-Dichloroethene | 156-59-2 | | Xylene (total)[4] | 1330-20-7 | |

[2] Vinyl acetate has often been included on DoD target analyte lists for Method 8260 in the past. Data indicate that it may not consistently produce quantitative data with this method. Therefore, it has purposely been removed from the target analyte list. The compound may be added back to the list on a project-specific basis.
[3] Though not selected by DoD, this compound was retained due to its inclusion on the Superfund list of 110 most frequently occurring chemicals.
[4] Data may be reported on a project-specific basis as Total Xylene; however, for purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-

## TABLE C-1. VOLATILE ORGANIC COMPOUNDS BY GC/MS TARGET ANALYTE LIST (CONTINUED) (BASED ON SW-846 METHOD 8260)

| Volatile Compound | CAS # | Comments | Volatile Compound | CAS # | Comments |
|---|---|---|---|---|---|
| trans-1,2-Dichloroethene | 156-60-5 | | 4-Bromofluorobenzene | 460-00-4 | Surrogate |
| 1,2-Dichloropropane | 78-87-5 | | Dibromofluoromethane | 1868-53-7 | Surrogate |
| 1,3-Dichloropropane | 142-28-9 | | 1,2-Dichloroethane-$d_4$ | 17060-07-0 | Surrogate |
| 2,2-Dichloropropane | 594-20-7 | | Toluene-$d_8$ | 2037-26-5 | Surrogate |

## TABLE C-2. SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS TARGET ANALYTE LIST[5] (BASED ON SW-846 METHOD 8270)

| Semivolatile Compound | CAS # | Comments | Semivolatile Compound | CAS # | Comments |
|---|---|---|---|---|---|
| Acenaphthene | 83-32-9 | See also 8310 | 2,4-Dinitrotoluene | 121-14-2 | |
| Acenaphthylene | 208-96-8 | See also 8310 | 2,6-Dinitrotoluene | 606-20-2 | |
| Anthracene | 120-12-7 | See also 8310 | 1,2-Diphenylhydrazine | 122-66-7 | |
| Benzidine | 92-87-5 | | Di-n-octyl phthalate | 117-84-0 | |
| ***Benzoic acid***[6,7] | 65-85-0 | | Fluoranthene | 206-44-0 | See also 8310 |
| Benz[a]anthracene | 56-55-3 | See also 8310 | Fluorene | 86-73-7 | See also 8310 |
| Benzo[b]fluoranthene | 205-99-2 | See also 8310 | Hexachlorobenzene | 118-74-1 | |
| Benzo[k]fluoranthene | 207-08-9 | See also 8310 | Hexachlorobutadiene | 87-68-3 | |
| Benzo[g,h,i]perylene | 191-24-2 | See also 8310 | Hexachloroethane | 67-72-1 | |
| Benzo[a]pyrene | 50-32-8 | See also 8310 | Indeno[1,2,3-cd]pyrene | 193-39-5 | See also 8310 |
| Benzyl alcohol | 100-51-6 | | Isophorone | 78-59-1 | |
| Bis(2-chlorethoxy)methane | 111-91-1 | | 2-Methylnaphthalene | 91-57-6 | |
| Bis(2-chloroethyl) ether | 111-44-4 | | 2-Methylphenol | 95-48-7 | |
| Bis(2-chloroisopropyl) ether | 108-60-1 | | 3-Methylphenol/4-Methylphenol | 108-39-4 / 106-44-5 | |
| Bis(2-ethylhexyl) phthalate | 117-81-7 | | Naphthalene | 91-20-3 | See also 8260, 8310 |
| 4-Bromophenyl phenyl ether | 101-55-3 | | 2-Nitroaniline | 88-74-4 | |
| Butyl benzyl phthalate | 85-68-7 | | 3-Nitroaniline | 99-09-2 | |
| Carbazole | 86-74-8 | | 4-Nitroaniline | 100-01-6 | |

Xylene.

[5] Hexachlorocyclopentadiene has often been included on DoD target analyte lists for Method 8270 in the past. The analyte is an intermediate product of pesticide manufacturing and would not be expected to be found on a DoD site. Therefore, it has purposely been removed from the target analyte list. If pesticide manufacturing has occurred at the site, the compound should be added back in on a project-specific basis.

[6] Poor performing analyte for the solid matrix. Must be in the calibration standard but data indicate it may not consistently produce quantitative data. See Section D.5 of Appendix DoD-D for further explanation.

## TABLE C-2. SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS TARGET ANALYTE LIST (CONTINUED) (BASED ON SW-846 METHOD 8270)

| Semivolatile Compound | CAS # | Comments | Semivolatile Compound | CAS # | Comments |
|---|---|---|---|---|---|
| ***4-Chloroaniline***[6] | 106-47-8 | | Nitrobenzene | 98-95-3 | See also 8330 |
| 4-Chloro-3-methylphenol | 59-50-7 | | 2-Nitrophenol | 88-75-5 | |
| 2-Chloronaphthalene | 91-58-7 | | ***4-Nitrophenol***[7] | 100-02-7 | |
| 2-Chlorophenol | 95-57-8 | | N-Nitrosodimethylamine | 62-75-9 | |
| 4-Chlorophenyl phenyl ether | 7005-72-3 | | N-Nitrosodiphenylamine | 86-30-6 | |
| Chrysene | 218-01-9 | See also 8310 | N-Nitrosodi-n-propylamine | 621-64-7 | |
| Dibenz[a,h]anthracene | 53-70-3 | See also 8310 | N-Nitrosopyrrolidine | 930-55-2 | |
| Dibenzofuran | 132-64-9 | | Pentachlorophenol | 87-86-5 | |
| Di-n-butyl phthalate | 84-74-2 | | Phenanthrene | 85-01-8 | See also 8310 |
| 1,2-Dichlorobenzene | 95-50-1 | See also 8260 | ***Phenol***[7] | 108-95-2 | |
| 1,3-Dichlorobenzene | 541-73-1 | See also 8260 | Pyrene | 129-00-0 | See also 8310 |
| 1,4-Dichlorobenzene | 106-46-7 | See also 8260 | 1,2,4-Trichlorobenzene | 120-82-1 | See also 8260 |
| ***3,3'-Dichlorobenzidine***[6] | 91-94-1 | | 2,4,5-Trichlorophenol | 95-95-4 | |
| 2,4-Dichlorophenol | 120-83-2 | | 2,4,6-Trichlorophenol | 88-06-2 | |
| 2,6-Dichlorophenol | 87-65-0 | | 2-Fluorophenol | 367-12-4 | Surrogate |
| Diethyl phthalate | 84-66-2 | | ***Phenol-$d_5$/$d_6$***[7] | 13127-88-3 | Surrogate |
| 2,4-Dimethylphenol | 105-67-9 | | Nitrobenzene-$d_5$ | 4165-60-0 | Surrogate |
| Dimethyl phthalate | 131-11-3 | | 2-Fluorobiphenyl | 321-60-8 | Surrogate |
| 4,6-Dintro-2-methylphenol | 534-52-1 | | 2,4,6-Tribromophenol | 118-79-6 | Surrogate |
| 2,4-Dinitrophenol | 51-28-5 | | Terphenyl-$d_{14}$ | 1718-51-0 | Surrogate |

[7] Poor performing analyte for water. Must be in the calibration standard but data indicate it may not consistently produce quantitative data. See Section D.5 of Appendix DoD-D for further explanation.

## TABLE C-3. DIOXINS/FURANS BY GC/MS TARGET ANALYTE LIST (BASED ON SW-846 METHODS 8280 AND 8290)

| Dioxin/Furan Compound | CAS # | Comments |
|---|---|---|
| 2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD) | 1746-01-6 | |
| 1,2,3,7,8-Pentachlorodibenzo-p-dioxin | 40321-76-4 | |
| 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin | 39227-28-6 | |
| 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin | 57653-85-7 | |
| 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin | 19408-74-3 | |
| 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin | 35822-46-9 | |
| 1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin | 3268-87-9 | |
| 2,3,7,8-Tetrachlorodibenzofuran | 51207-31-9 | |
| 1,2,3,7,8-Pentachlorodibenzofuran | 57117-41-6 | |
| 2,3,4,7,8-Pentachlorodibenzofuran | 57117-31-4 | |
| 1,2,3,4,7,8-Hexachlorodibenzofuran | 70648-26-9 | |
| 1,2,3,6,7,8-Hexachlorodibenzofuran | 57117-44-9 | |
| 1,2,3,7,8,9-Hexachlorodibenzofuran | 72918-21-9 | |
| 2,3,4,6,7,8-Hexachlorodibenzofuran | 60851-34-5 | |
| 1,2,3,4,6,7,8-Heptachlorodibenzofuran | 67562-39-4 | |
| 1,2,3,4,7,8,9-Heptachlorodibenzofuran | 55673-87-7 | |
| 1,2,3,4,6,7,8,9-Octachlorodibenzofurans | 39001-02-0 | |
| $^{13}C_{12}$-1,2,3,4-TCDD | | Recovery standard |
| $^{13}C_{12}$-1,2,3,7,8,9-HxCDD | | Recovery standard |
| $^{37}Cl_4$-2,3,7,8-TCDD | | Cleanup standard |

## TABLE C-4. ORGANOPHOSPHORUS PESTICIDES BY GC/FPD OR NPD TARGET ANALYTE LIST (BASED ON SW-846 METHOD 8141)

| Organophosphorus Pesticide Compound | CAS # | Comments |
|---|---|---|
| Azinphos-methyl | 86-50-0 | |
| Bolstar (Sulprofos) | 35400-43-2 | |
| Chlorpyrifos | 2921-88-2 | |
| Coumaphos | 56-72-4 | |
| Demeton-O | 298-03-3 | |
| Demeton-S | 126-75-0 | |
| Diazinon | 333-41-5 | |
| Dichlorvos (DDVP) | 62-73-7 | |
| Disulfoton | 298-04-4 | |
| Ethoprop | 13194-48-4 | |
| Fensulfothion | 115-90-2 | |
| Fenthion | 55-38-9 | |
| Merphos | 150-50-5 | |
| Naled | 300-76-5 | |
| Parathion, methyl | 298-00-0 | |
| Phorate | 298-02-2 | |
| Ronnel | 299-84-3 | |
| Stirophos (Tetrachlorovinphos) | 961-11-5 | |
| Tokuthion (Protothiofos) | 34643-46-4 | |
| Trichloronate | 327-98-0 | |
| 4-Chloro-3-nitrobenzo-trifluoride | 121-17-5 | Surrogate |
| Tributyl phosphate | 126-73-8 | Surrogate |
| Triphenyl phosphate | 115-86-6 | Surrogate |

## TABLE C-5. CHLORINATED HERBICIDES BY GC/ECD TARGET ANALYTE LIST (BASED ON SW-846 METHOD 8151)

| Chlorinated Herbicide Compound | CAS # | Comments |
|---|---|---|
| 2,4-D | 94-75-7 | |
| 2,4-DB | 94-82-6 | |
| 2,4,5-TP (Silvex) | 93-72-1 | |
| 2,4,5-T | 93-76-5 | |
| Dalapon | 75-99-0 | |
| Dicamba | 1918-00-9 | |
| Dichloroprop | 120-36-5 | |
| Dinoseb | 88-85-7 | |
| MCPA | 94-74-6 | |
| MCPP | 93-65-2 | |
| 2,4-Dichlorophenylacetic acid | 19719-28-9 | Surrogate |

## TABLE C-6. POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC TARGET ANALYTE LIST (BASED ON SW-846 METHOD 8310)

| Polynuclear Aromatic Hydrocarbon Compound | CAS # | Comments |
|---|---|---|
| Acenaphthene | 83-32-9 | See also 8270 |
| Acenaphthylene | 208-96-8 | See also 8270 |
| Anthracene | 120-12-7 | See also 8270 |
| Benz[a]anthracene | 56-55-3 | See also 8270 |
| Benzo[a]pyrene | 50-32-8 | See also 8270 |
| Benzo[b]fluoranthene | 205-99-2 | See also 8270 |
| Benzo[k]fluoranthene | 207-08-9 | See also 8270 |
| Benzo[g,h,i]perylene | 191-24-2 | See also 8270 |
| Chrysene | 218-01-9 | See also 8270 |
| Dibenzo[a,h]anthracene | 53-70-3 | See also 8270 |
| Fluoranthene | 206-44-0 | See also 8270 |
| Fluorene | 86-73-7 | See also 8270 |
| Indeno[1,2,3-cd]pyrene | 193-39-5 | See also 8270 |
| Naphthalene | 91-20-3 | See also 8270 |
| Phenanthrene | 85-01-8 | See also 8270 |
| Pyrene | 129-00-0 | See also 8270 |
| Decafluorobiphenyl | 434-90-2 | Surrogate |

## TABLE C-7. EXPLOSIVES BY HPLC TARGET ANALYTE LIST[8] (BASED ON SW-846 METHOD 8330)

| Explosive Compound | CAS # | Comments |
|---|---|---|
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | 2691-41-0 | |
| Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) | 121-82-4 | |
| 1,3,5-Trinitrobenzene | 99-35-4 | |
| 1,3-Dinitrobenzene | 99-65-0 | |
| Methyl-2,4,6-trinitrophenylnitramine (Tetryl) | 479-45-8 | |
| Nitrobenzene | 98-95-3 | See also 8260 and 8270 |
| 2,4,6-Trinitrotoluene (TNT) | 118-96-7 | |
| 4-Amino-2,6-dinitrotoluene | 19406-51-0 | |
| 2-Amino-4,6-dinitrotoluene | 35572-78-2 | |

[8] When surrogate compounds are not identified by the client, use an analyte from the method that is not expected to be present in the samples as the surrogate.

## TABLE C-7. EXPLOSIVES BY HPLC TARGET ANALYTE LIST (CONTINUED) (BASED ON SW-846 METHOD 8330)

| Explosive Compound | CAS # | Comments |
|---|---|---|
| 2,4-Dinitrotoluene | 121-14-2 | See also 8270 |
| 2,6-Dinitrotoluene | 606-20-2 | See also 8270 |
| 2-Nitrotoluene | 88-72-2 | |
| 3-Nitrotoluene | 99-08-1 | |
| 4-Nitrotoluene | 99-99-0 | |

## TABLE C-8. ORGANOCHLORINE PESTICIDES BY GC/ECD TARGET ANALYTE LIST (BASED ON SW-846 METHOD 8081)

| Organochlorine Pesticide Compound | CAS # | Comments |
|---|---|---|
| Aldrin | 309-00-2 | |
| alpha-BHC | 319-84-6 | |
| beta-BHC | 319-85-7 | |
| delta-BHC | 319-86-8 | |
| gamma-BHC (Lindane) | 58-89-9 | |
| alpha-Chlordane | 5103-71-9 | |
| gamma-Chlordane | 5103-74-2 | |
| Chlordane (not otherwise specified) | 57-74-9 | |
| 4,4'-DDD | 72-54-8 | |
| 4,4'-DDE | 72-55-9 | |
| 4,4'-DDT | 50-29-3 | |
| Dieldrin | 60-57-1 | |
| Endosulfan I | 959-98-8 | |
| Endosulfan II | 33213-65-9 | |
| Endosulfan sulfate | 1031-07-8 | |
| Endrin | 72-20-8 | |
| Endrin aldehyde | 7421-93-4 | |
| Endrin ketone | 53494-70-5 | |
| Heptachlor | 76-44-8 | |
| Heptachlor epoxide | 1024-57-3 | |
| Hexachlorobenzene | 118-74-1 | See also 8270 |
| Methoxychlor | 72-43-5 | |
| Toxaphene | 8001-35-2 | |
| 4-Chloro-3-nitrobenzo-trifluoride | 121-17-5 | Surrogate |
| Tetrachloro-m-xylene (TCMX) | 877-09-8 | Surrogate |
| Decachlorobiphenyl | 2051-24-3 | Surrogate |

## TABLE C-9. POLYCHLORINATED BIPHENYLS BY GC/ECD TARGET ANALYTE LIST (BASED ON SW-846 METHOD 8082)

| PCB Compound | CAS # | Comments |
|---|---|---|
| Aroclor 1016 | 12674-11-2 | |
| Aroclor 1221 | 11104-28-2 | |
| Aroclor 1232 | 11141-16-5 | |
| Aroclor 1242 | 53469-21-9 | |
| Aroclor 1248 | 12672-29-6 | |
| Aroclor 1254 | 11097-69-1 | |
| Aroclor 1260 | 11096-82-5 | |

## TABLE C-9. POLYCHLORINATED BIPHENYLS BY GC/ECD TARGET ANALYTE LIST (CONTINUED) (BASED ON SW-846 METHOD 8082)

| PCB Compound | CAS # | Comments |
|---|---|---|
| Aroclor 1262 | 37324-23-5 | |
| Aroclor 1268 | 11100-14-4 | |
| Decachlorobiphenyl | 2051-24-3 | Surrogate |
| Tetrachloro-m-xylene (TCMX) | 877-09-8 | Surrogate |
| 2,2',4,4',5,5'-Hexabromobiphenyl | 59080-40-9 | Surrogate 8082 |

## TABLE C-10. METALS BY ICP, ICP/MS, GFAA, AND CVAA TARGET ANALYTE LIST (BASED ON SW-846 METHODS 6010, 6020, AND 7000 SERIES)

| Metal | CAS # | Comments |
|---|---|---|
| Aluminum | 7429-90-5 | 6010/6020 |
| Antimony | 7440-36-0 | 6010//6020/7010 |
| Arsenic | 7440-38-2 | 6010/6020/7010/7061/7062 |
| Barium | 7440-39-3 | 6010/6020/7010 |
| Beryllium | 7440-41-7 | 6010/6020/7010 |
| Cadmium | 7440-43-9 | 6010/6020/7010 |
| Calcium | 7440-70-2 | 6010/6020 |
| Chromium | 7440-47-3 | 6010/6020/7010 |
| Cobalt | 7440-48-4 | 6010/60207010 |
| Copper | 7440-50-8 | 6010/6020/7010 |
| Iron | 7439-89-6 | 6010/6020/7010 |
| Lead | 7439-92-1 | 6010/6020/7010 |
| Magnesium | 7439-95-4 | 6010/6020 |
| Manganese | 7439-96-5 | 6010/6020/7010 |
| Mercury | 7439-97-6 | 6020/7470/7471/7472/7473 |
| Molybdenum | 7439-98-7 | 6010/6020/7010 |
| Nickel | 7440-02-0 | 6010/60207010 |
| Potassium | 7440-09-7 | 6010/6020 |
| Selenium | 7782-49-2 | 6010/6020/7010/7741/7742 |
| Silver | 7440-22-4 | 6010/6020/7010 |
| Sodium | 7440-23-5 | 6010/6020 |
| Thallium | 7440-28-0 | 6010/6020/7010 |
| Vanadium | 7440-62-2 | 6010/6020/7010 |
| Zinc | 7440-66-6 | 6010/6020/7010 |

## TABLE C-11. OTHER INORGANICS TARGET ANALYTE LIST[9] (BASED ON SW-846 METHODS 7000 SERIES, 9010, 9012, AND 9056)

| Inorganic Compound | CAS # | Comments |
|---|---|---|
| Bromide | 24959-67-9 | 9056 |
| Chloride | 16887-00-6 | 9056 |
| Chromium, hexavalent | 18540-29-9 | 7195/7196/7197/7198/7199 |
| Cyanide | 57-12-5 | 9010/9012/9013/9014 |
| Fluoride | 16984-48-8 | 9056 |
| Nitrate | 14797-55-8 | 9056 |
| Nitrite | 14979-65-0 | 9056 |
| Phosphate | 14265-44-2 | 9056 |
| Sulfate | 14808-79-8 | 9056 |

[9] Hexavalent chromium, cyanide, and anions will most likely be requested separately from each other.

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# APPENDIX DOD-D – LCS CONTROL LIMITS

DoD conducted a study to establish control limits for laboratory control samples using data collected from environmental laboratories that analyze samples for DoD. LCS recoveries for all the analytes on the target analyte lists were pooled, and statistical analyses (such as outlier tests and analysis of variance) were performed on the data before generating the final LCS control limits (LCS-CL). A complete description of the methodology and findings for Method 8270 can be found in the Laboratory Control Sample Pilot Study (DoD, 2000).

Environmental testing laboratories that perform work for DoD must utilize the DoD-specified LCS control limits when assessing batch acceptance whenever they are available. This appendix presents the control limits generated by the LCS study and the methodology for applying the limits to LCS data. All analytes spiked in the LCS shall meet the DoD-generated LCS control limits. As described in Section D.1.1.2.1.e of NELAC Appendix D, a number of sporadic marginal exceedances are allowed. Depending on the length of the list of analytes, a specified small number of analytes may exceed the generated control limit. Upper and lower marginal exceedance (ME) limits, calculated at 4 standard deviations around the mean, are established to mark the boundaries of marginal exceedances. If more analytes exceed the LCS-CLs than are allowed, or if any one analyte exceeds the ME limits, then the LCS has failed.

**DoD LCS Control Limits Policy**

- The laboratory shall use project-specific control limits based on data quality objectives (DQOs), if available. If not, DoD-generated LCS-CLs shall be used, if available. Otherwise, the laboratory's own in-house control limits shall be used.
- The LCS-CLs are based on the promulgated versions of SW-846 methods at the time of the study (2000). They should be used as a benchmark to evaluate acceptability even as methods are updated or alternative methods for the same class of compounds become available.
- The fact that the LCS-CLs are based on certain SW-846 methods should not limit the use of alternative analytical methods, as appropriate. If an alternative method is used, however, it should be capable of producing LCS recoveries that are at least as good as the DoD-generated LCS-CLs, unless project-specific DQOs allow less stringent criteria.
- The LCS study shows that preparatory methods may have a significant influence on a laboratory's ability to achieve certain LCS-CLs. If a laboratory is unable to achieve the LCS-CLs presented in this appendix, it should investigate the use of alternative preparatory methods as a means to improve precision and accuracy.

## D.1 Generated LCS Control Limits

As mentioned above, DoD compiled LCS data from multiple laboratories, performing statistical analyses on the data sets before generating control limits. The control limits were set at 3 standard deviations around the mean for all methods except 8151 (see below for further explanation). Limits were then rounded to the nearest 5 for ease of use. The ME limits were set at 4 standard deviations around the mean. The lower ME limit was then raised to 10% for those analytes in which 4 standard deviations falls below that level. Tables D-4 through D-19 at the end of this appendix present the mean or median, standard deviation, lower control limit, upper control limit, lower ME limit, and upper ME limit, as applicable, for each analyte in Methods 8260, 8270, 8151, 8310, 8330, 8081, 8082, 6010, and 7470/7471, for the water and solid matrices. The lower and upper ME limits are not presented for Methods 8151, 8082, and 7470/7471, since those methods have fewer than 11 analytes and are therefore not capable of utilizing the sporadic marginal exceedance allowance. The analytes for Method 8270 are grouped by compound class.

The control limits for explosives Method 8330 in the water matrix were generated using data that were extracted with solid phase extraction (SPE) using acetonitrile only. Analysis of the data received from the LCS study showed that the extraction method produced recoveries with higher means and lower standard deviations than the salting out extraction method. This results in significantly narrower control limits. Since SPE (acetonitrile) is less expensive, cumbersome, and time and labor intensive, the LCS control

limits for Method 8330 in water were set with data using only that method. A limited amount of data were received that used SPE/acetonitrile, therefore, no outliers were removed during the statistical analysis. This ensures that a representative data set was used to generate the control limits (see Table D-12). Note: Laboratories may use any extraction method they feel is appropriate; however, the LCS recoveries must fall within the LCS-CLs presented in Table D-12.

Control limits for chlorinated herbicides Method 8151 were generated using a non-parametric statistical approach. This is a different approach than for the other methods in the LCS study due to the large amount of intralaboratory variability in recoveries for all analytes in the method. The control limits for Method 8151, both solid and water matrices, were set at the 5$^{th}$ and 95$^{th}$ percentile of all data received in the study (no outliers were removed). Tables D-8 and D-9 present the median, lower control limit, and upper control limit for each analyte. LCS failure is assessed and corrective action applied the same way for all methods with control limits in this appendix (see Sections D.3 and D.4). (Note: These data represent the current capability of the SW-846 analytical and preparatory methods. Use of alternative preparatory procedures and/or improvements through PBMS is encouraged. Project-specific control limits can supersede these DoD limits.) If limits are not available for a project-specific analyte, the laboratory shall discuss with the client appropriate limits considering the project-specific DQOs.

Control limits for metals Method 6010, and mercury Method 7470/7471 were set at 80 to 120% even though generated limits were within these numbers. This reflects the allowable uncertainty in the calibration of the instrument. In one case the generated limit (silver in solid) was outside 80 to 120%, and therefore the generated limit was used.

## D.2 Marginal Exceedance

As described in Section D.1.1.2.1.e of NELAC Appendix D, a number of sporadic marginal exceedances of the LCS-CLs will be allowed. The number of exceedances is based on the total number of analytes spiked in the LCS. As the number of analytes in the LCS increases, more marginal exceedances are allowed. Table D-1 presents the allowable number of marginal exceedances for a given number of analytes in the LCS (as presented in NELAC Appendix D).

**TABLE D-1. NUMBER OF MARGINAL EXCEEDANCES**

| Number of Analytes in LCS | Allowable Number of Marginal Exceedances of LCS-CLs |
|---|---|
| > 90 | 5 |
| 71 – 90 | 4 |
| 51 – 70 | 3 |
| 31 – 50 | 2 |
| 11 – 30 | 1 |
| < 11 | 0 |

A *marginal* exceedance is defined as beyond the LCS-CL but still within the marginal exceedance limits (set at 4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing.

NELAC requires that the marginal exceedances be sporadic (i.e., random). As defined by DoD, if the same analyte exceeds the LCS-CL repeatedly (e.g., 2 out of 3 consecutive LCS), that is an indication that the problem is systematic and something is wrong with the measurement system. The source of error should be located and the appropriate corrective action taken. Laboratories must monitor through QA channels the application of the sporadic marginal exceedance allowance to the LCS results to ensure random behavior. Effective implementation of the marginal exceedance allowance requires cooperation from the laboratory. If the laboratory fails to implement the policy properly, the privilege of using the marginal exceedance option will be revoked. Oversight and appropriate corrective action will be a focus of DoD laboratory assessments in the future.

## D.3 LCS Failure

Each LCS must be evaluated against the appropriate control limits and ME limits before being accepted. The laboratory shall use project-specific control limits, if available. If not, DoD generated LCS-CLs shall be used, if available (see Tables D-4 through D-19). Otherwise, the laboratory's own in-house control limits shall be used. First, the recoveries for the analytes spiked in the LCS should be compared with the LCS control limits. If a recovery is less than the lower control limit or greater than the upper control limit, that is an exceedance. The laboratory should note which analytes exceeded the control limits and make a comparison to the list of project-specific analytes of concern. **If a project-specific analyte of concern exceeds its LCS-CL, the LCS has failed.** Next, the laboratory should add up the total number of exceedances for the LCS. Based on the number of analytes spiked in the LCS, the total number of exceedances should be compared with the allowable number from Table D-1. (The allowable number of marginal exceedances depends on the total number of analytes spiked in the LCS, even if DoD-generated control limits are not available for all analytes.) **If a LCS has more than the allowable number of marginal exceedances, the LCS has failed.** Finally, the recoveries for those analytes that exceeded the LCS-CL should be compared to the ME limits from Tables D-4 to D-7, D-10 to D-15, or D-18 to D-19. **If a single analyte exceeds its marginal exceedance limit, the LCS has failed.** (This applies only to methods with greater than 10 analytes.)

Note: For the purposes of this section, the target analyte lists from Appendix DoD-C or other general lists should not be considered project-specific analytes of concern. A requirement to analyze all compounds on a general target analyte list does not define a project-specific analyte of concern for LCS batch acceptance.

In summary, failure of the LCS can occur several ways:

- Exceedance of a LCS-CL by any project-specific analyte of concern
- Marginal exceedance of the LCS-CLs by more than the allowable number of analytes
- Exceedance of the ME limits by one or more analytes

Once a LCS has failed, corrective action is required (see section D.4).

## D.4 Corrective Action

If a sample fails based on any of the criteria in section D.3, corrective action is required. The corrective action requirement applies to all analytes that exceeded the LCS-CLs, even if one specific analyte's exceedance was not the trigger of LCS failure (see example in text box). **All exceedances of the LCS-CLs, marginal or otherwise, are subject to corrective action.**

**Example of Applying Corrective Action**

- In a single LCS, anthracene has a recovery of 30%.
- The lower ME limit for anthracene is 45, therefore the LCS has failed.
- In the same LCS three other analytes exceeded their LCS-CLs but were within their ME limits.
- The LCS was spiked with 74 analytes; therefore, according to Table D-1, four marginal exceedances are allowed.
- The four total exceedances (anthracene plus the three other analytes) are within the allowable number for that analyte list size.

*Corrective action is triggered for the LCS because the anthracene recovery exceeded its ME limit, but it is required for all four analytes that exceeded the LCS-CLs.*

If a LCS fails, an attempt must be made to determine the source of error and find a solution. All the findings and corrective action should be documented. DoD then requires that the analytes subject to corrective action in the LCS and all the samples in the batch be reprepped and reanalyzed or the batch rerun with a new LCS. The corrective action applied shall be based on professional judgment in the review of other QC measures (i.e., surrogates). If an analyte falls outside the LCS-CL a second time or if there is not sufficient sample material available to be reanalyzed,

then all the results in the associated batch for that analyte must be flagged with a Q (see DoD clarification box 20). The recoveries of those analytes subject to corrective action must be documented in the case narrative, whether flagging is needed or not.

### D.5 Poor Performing Analytes

On the basis of results from the LCS study, DoD identified certain compounds that do not perform well with specific methods. These compounds produce low mean recoveries and high standard deviations, resulting in wide LCS control limits with particularly low lower control limits (sometimes negative values). The performance of these compounds reflects routine implementation of the method in many laboratories. DoD has defined a poor performing analyte as having a lower control limit of 10% or less. DoD does not feel it is appropriate to control batch acceptance on these compounds because there is a high level of uncertainty in their recovery. The data may be used; however, routine performance of the method on these compounds can result in being able to identify only a small percentage of the analyte.

The laboratory should include all target analytes in the calibration standard, including the poor performing analytes. If one of the poor performing analytes identified below is a project-specific analyte of concern *or* if it is detected in the project samples, the laboratory should contact the client (DoD), who will then work with the laboratory on an appropriate course of action. Ideally DoD and the laboratory would use an alternative method to test for the analyte (one that is known to produce higher recoveries) or else modify the original method to optimize conditions for the poor performing analyte.

Poor performing analytes were only identified in SW-846 Methods 8270, 8151, and 8330. These analytes, along with the mean, standard deviation, lower control limit, upper control limit, lower ME limit, and upper ME limit (as generated by the LCS study) are presented in Table D-2.

**TABLE D-2. POOR PERFORMING ANALYTES[10]**

| Analyte | Mean/ Median | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| **8270 Water:** | | | | | | |
| 4-Nitrophenol | 54 | 23 | 0 | 125 | 0 | 145 |
| Benzoic acid | 54 | 24 | 0 | 125 | 0 | 150 |
| Phenol | 55 | 19 | 0 | 115 | 0 | 135 |
| Phenol-$d_5$/$d_6$ (surrogate) | 62 | 18 | 10 | 115 | 0 | 135 |
| **8270 Solid:** | | | | | | |
| 3,3'Dichlorobenzidine | 68 | 19 | 10 | 130 | 0 | 145 |
| 4-Chloroaniline | 51 | 14 | 10 | 95 | 0 | 110 |
| Benzoic acid | 55 | 18 | 0 | 110 | 0 | 130 |
| **8151 Solid:** | | | | | | |
| Dinoseb | 72 | | 5 | 130 | | |
| **8330 Solid:** | | | | | | |
| Methyl-2,4,6-trinitrophenylnitramine (Tetryl) | 80 | 23 | 10 | 150 | 0 | 172 |

Note: Lower limits calculated as negative values were raised to zero.

The LCS control limits generated by the study for the poor performing analytes are provided as a benchmark against which laboratories may measure the effectiveness of alternative methods or modifications to the current methods. Batch acceptance should not be evaluated using these limits. When choosing alternative or modified methods, laboratories should strive to raise the mean recoveries and lower the standard deviations in comparison with the performance of the analytes presented in Table D-2.

[10] Control limits for method 8151 were generated using non-parametric statistics; therefore, the median and no standard deviation is presented (see Section D.1 for further explanation). ME limits are not used for method 8151 since the target analyte list has less than 11 analytes.

The lower control limit generated for alternative or modified methods must be greater than 10% to be considered acceptable.

## D.6 Surrogates

The surrogate compounds for each method are added to all samples, standards, and blanks to assess the ability of the method to recover specific non-target analytes from a given matrix and to monitor sample-specific recovery. Control limits for these compounds were calculated in the same study as the other analytes on the target analyte lists. Below are the limits for some of the surrogates of Methods 8260, 8270, 8081, and 8082, based on 3 standard deviations around the mean (Table D-3). Control limits are not available for some surrogates that appear on the target analyte lists in Appendix DoD-C. Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. No ME limits are presented as marginal exceedances are not acceptable for surrogate spikes. Note: DoD prefers the use of those surrogates not identified as poor performing analytes in Table D-2 above.

**TABLE D-3. SURROGATES**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|---|---|---|---|---|
| **8260 Water:** | | | | |
| 1,2-Dichloroethane-$d_4$ | 95 | 8 | 70 | 120 |
| 4-Bromofluorobenzene | 98 | 7 | 75 | 120 |
| Dibromofluoromethane | 100 | 5 | 85 | 115 |
| Toluene-$d_8$ | 102 | 6 | 85 | 120 |
| **8260 Solid:** | | | | |
| 4-Bromofluorobenzene | 101 | 6 | 85 | 120 |
| Toluene-$d_8$ | 100 | 5 | 85 | 115 |
| **8270 Water:** | | | | |
| 2-Fluorobiphenyl | 79 | 10 | 50 | 110 |
| Terphenyl-$d_{14}$ | 92 | 14 | 50 | 135 |
| 2,4,6-Tribromophenol | 82 | 13 | 40 | 125 |
| 2-Fluorophenol | 63 | 14 | 20 | 110 |
| Nitrobenzene-$d_5$ | 76 | 11 | 40 | 110 |
| **8270 Solid:** | | | | |
| 2-Fluorobiphenyl | 72 | 10 | 45 | 105 |
| Terphenyl-$d_{14}$ | 78 | 15 | 30 | 125 |
| 2,4,6-Tribromophenol | 80 | 15 | 35 | 125 |
| 2-Fluorophenol | 70 | 11 | 35 | 105 |
| Phenol-$d_5$/$d_6$ | 71 | 10 | 40 | 100 |
| Nitrobenzene-$d_5$ | 69 | 10 | 35 | 100 |
| **8081 Water:** | | | | |
| Decachlorobiphenyl | 83 | 17 | 30 | 135 |
| TCMX | 81 | 19 | 25 | 140 |
| **8081 Solid:** | | | | |
| Decachlorobiphenyl | 94 | 13 | 55 | 130 |
| TCMX | 97 | 9 | 70 | 125 |
| **8082 Water:** | | | | |
| Decachlorobiphenyl | 88 | 15 | 40 | 135 |
| **8082 Solid:** | | | | |
| Decachlorobiphenyl | 91 | 11 | 60 | 125 |

## D.7 In-House LCS Control Limits

The acceptability of LCS results within any preparatory batch shall be based on project specified limits or the following DoD-specified LCS control limits, if project-specific limits are not available. If DoD limits are not available, the laboratory must use its in-house limits for batch acceptance.

DoD strongly believes that it is important for laboratories to maintain their own in-house LCS limits. These in-house limits must be consistent with the DoD limits (project-specific, if available; otherwise the following LCS-CLs). The laboratory in-house limits shall be calculated from the laboratory's historical LCS data in accordance with a documented procedure (e.g., SOP) that is consistent with good laboratory practice. That document must describe the process for establishing and maintaining LCS limits and the use of control charts.

The laboratory in-house limits are to be used for several purposes:

- Laboratories are expected to utilize their in-house limits as part of their quality control system, and to evaluate trends and monitor and improve performance.

- When laboratories' in-house limits are outside the DoD control limits (upper and/or lower), they must report their in-house limits in the laboratory report (see Appendix DoD-A) even if the LCS associated with the batch in fact fell within the DoD limits. In this manner, DoD will be able to evaluate how laboratory performance affects the quality of the environmental data.

- DoD may review the laboratory in-house limits and associated trends, as reflected in control charts, to determine whether the laboratory's overall performance is acceptable. If deemed unacceptable, this may be a basis on which DoD makes a decision to not use the laboratory again until substantial improvement has occurred.

**TABLE D-4. LCS CONTROL LIMITS FOR VOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8260 WATER MATRIX[11]**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| 1,1,1,2-Tetrachloroethane | 105 | 8 | 80 | 130 | 75 | 135 |
| 1,1,1-Trichloroethane | 100 | 11 | 65 | 130 | 55 | 145 |
| 1,1,2,2-Tetrachloroethane | 96 | 11 | 65 | 130 | 55 | 140 |
| 1,1,2-Trichloroethane | 100 | 8 | 75 | 125 | 65 | 135 |
| 1,1-Dichloroethane | 101 | 11 | 70 | 135 | 60 | 145 |
| 1,1-Dichloroethene | 99 | 10 | 70 | 130 | 55 | 140 |
| 1,1-Dichloropropene | 102 | 10 | 75 | 130 | 65 | 140 |
| 1,2,3-Trichlorobenzene | 99 | 14 | 55 | 140 | 45 | 155 |
| 1,2,3-Trichloropropane | 98 | 9 | 75 | 125 | 65 | 130 |
| 1,2,4-Trichlorobenzene | 100 | 11 | 65 | 135 | 55 | 145 |
| 1,2,4-Trimethylbenzene | 103 | 10 | 75 | 130 | 65 | 140 |
| 1,2-Dibromo-3-chloropropane | 91 | 14 | 50 | 130 | 35 | 145 |
| 1,2-Dibromoethane | 100 | 7 | 80 | 120 | 75 | 125 |
| 1,2-Dichlorobenzene | 96 | 9 | 70 | 120 | 60 | 130 |
| 1,2-Dichloroethane | 100 | 10 | 70 | 130 | 60 | 140 |
| 1,2-Dichloropropane | 100 | 8 | 75 | 125 | 65 | 135 |

[11] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Total Xylene. Xylene may be reported on a project-specific basis as a total number; however, for the purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section D.5 and for surrogate compounds in section D.6.

## TABLE D-4. LCS CONTROL LIMITS FOR VOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8260 WATER MATRIX[11]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| 1,3,5-Trimethylbenzene | 102 | 10 | 75 | 130 | 65 | 140 |
| 1,3-Dichlorobenzene | 100 | 8 | 75 | 125 | 65 | 130 |
| 1,3-Dichloropropane | 100 | 9 | 75 | 125 | 65 | 135 |
| 1,4-Dichlorobenzene | 99 | 8 | 75 | 125 | 65 | 130 |
| 2,2-Dichloropropane | 103 | 11 | 70 | 135 | 60 | 150 |
| 2-Butanone | 91 | 20 | 30 | 150 | 10 | 170 |
| 2-Chlorotoluene | 100 | 9 | 75 | 125 | 65 | 135 |
| 2-Hexanone | 92 | 12 | 55 | 130 | 45 | 140 |
| 4-Chlorotoluene | 101 | 9 | 75 | 130 | 65 | 135 |
| 4-Methyl-2-pentanone | 96 | 13 | 60 | 135 | 45 | 145 |
| Acetone | 91 | 17 | 40 | 140 | 20 | 160 |
| Benzene | 102 | 7 | 80 | 120 | 75 | 130 |
| Bromobenzene | 100 | 8 | 75 | 125 | 70 | 130 |
| Bromochloromethane | 97 | 11 | 65 | 130 | 55 | 140 |
| Bromodichloromethane | 98 | 8 | 75 | 120 | 70 | 130 |
| Bromoform | 99 | 10 | 70 | 130 | 60 | 140 |
| Bromomethane | 88 | 19 | 30 | 145 | 10 | 165 |
| Carbon disulfide | 100 | 21 | 35 | 160 | 15 | 185 |
| Carbon tetrachloride | 102 | 12 | 65 | 140 | 55 | 150 |
| Chlorobenzene | 102 | 7 | 80 | 120 | 75 | 130 |
| Chlorodibromomethane | 96 | 13 | 60 | 135 | 45 | 145 |
| Chloroethane | 99 | 12 | 60 | 135 | 50 | 145 |
| Chloroform | 100 | 12 | 65 | 135 | 50 | 150 |
| Chloromethane | 83 | 15 | 40 | 125 | 25 | 140 |
| cis-1,2-Dichloroethene | 99 | 9 | 70 | 125 | 60 | 135 |
| cis-1,3-Dichloropropene | 100 | 10 | 70 | 130 | 60 | 140 |
| Dibromomethane | 101 | 8 | 75 | 125 | 65 | 135 |
| Dichlorodifluoromethane | 93 | 21 | 30 | 155 | 10 | 175 |
| Ethylbenzene | 100 | 9 | 75 | 125 | 65 | 135 |
| Hexachlorobutadiene | 97 | 15 | 50 | 140 | 35 | 160 |
| Isopropylbenzene | 101 | 9 | 75 | 125 | 65 | 135 |
| m,p-Xylene | 102 | 9 | 75 | 130 | 65 | 135 |
| Methyl tert-butyl ether | 94 | 10 | 65 | 125 | 55 | 135 |
| Methylene chloride | 96 | 14 | 55 | 140 | 40 | 155 |
| Naphthalene | 96 | 14 | 55 | 140 | 40 | 150 |
| n-Butylbenzene | 103 | 11 | 70 | 135 | 55 | 150 |
| n-Propylbenzene | 101 | 9 | 70 | 130 | 65 | 140 |
| o-Xylene | 100 | 7 | 80 | 120 | 75 | 130 |
| p-Isopropyltoluene | 102 | 10 | 75 | 130 | 65 | 140 |
| sec-Butylbenzene | 100 | 9 | 70 | 125 | 65 | 135 |
| Styrene | 100 | 11 | 65 | 135 | 55 | 145 |
| tert-Butylbenzene | 99 | 10 | 70 | 130 | 60 | 140 |
| Tetrachloroethene | 96 | 18 | 45 | 150 | 25 | 165 |
| Toluene | 100 | 7 | 75 | 120 | 70 | 130 |
| trans-1,2-Dichloroethene | 99 | 13 | 60 | 140 | 45 | 150 |
| trans-1,3-Dichloropropene | 98 | 15 | 55 | 140 | 40 | 155 |
| Trichloroethene | 99 | 9 | 70 | 125 | 60 | 135 |
| Trichlorofluoromethane | 103 | 15 | 60 | 145 | 45 | 160 |
| Vinyl chloride | 99 | 16 | 50 | 145 | 35 | 165 |

**TABLE D-5. LCS CONTROL LIMITS FOR VOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8260 SOLID MATRIX[12]**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| 1,1,1,2-Tetrachloroethane | 100 | 9 | 75 | 125 | 65 | 135 |
| 1,1,1-Trichloroethane | 101 | 11 | 70 | 135 | 55 | 145 |
| 1,1,2,2-Tetrachloroethane | 93 | 13 | 55 | 130 | 40 | 145 |
| 1,1,2-Trichloroethane | 95 | 11 | 60 | 125 | 50 | 140 |
| 1,1-Dichloroethane | 99 | 9 | 75 | 125 | 65 | 135 |
| 1,1-Dichloroethene | 100 | 12 | 65 | 135 | 55 | 150 |
| 1,1-Dichloropropene | 102 | 11 | 70 | 135 | 60 | 145 |
| 1,2,3-Trichlorobenzene | 97 | 12 | 60 | 135 | 50 | 145 |
| 1,2,3-Trichloropropane | 97 | 11 | 65 | 130 | 50 | 140 |
| 1,2,4-Trichlorobenzene | 98 | 11 | 65 | 130 | 55 | 140 |
| 1,2,4-Trimethylbenzene | 100 | 12 | 65 | 135 | 55 | 145 |
| 1,2-Dibromo-3-chloropropane | 87 | 16 | 40 | 135 | 25 | 150 |
| 1,2-Dibromoethane | 97 | 9 | 70 | 125 | 60 | 135 |
| 1,2-Dichlorobenzene | 97 | 7 | 75 | 120 | 65 | 125 |
| 1,2-Dichloroethane | 104 | 11 | 70 | 135 | 60 | 145 |
| 1,2-Dichloropropane | 95 | 8 | 70 | 120 | 65 | 125 |
| 1,3,5-Trimethylbenzene | 99 | 11 | 65 | 135 | 55 | 145 |
| 1,3-Dichlorobenzene | 98 | 9 | 70 | 125 | 65 | 135 |
| 1,3-Dichloropropane | 100 | 8 | 75 | 125 | 70 | 130 |
| 1,4-Dichlorobenzene | 98 | 9 | 70 | 125 | 65 | 135 |
| 2,2-Dichloropropane | 101 | 11 | 65 | 135 | 55 | 145 |
| 2-Butanone | 94 | 22 | 30 | 160 | 10 | 180 |
| 2-Chlorotoluene | 98 | 10 | 70 | 130 | 60 | 140 |
| 2-Hexanone | 97 | 16 | 45 | 145 | 30 | 160 |
| 4-Chlorotoluene | 100 | 9 | 75 | 125 | 65 | 135 |
| 4-Methyl-2-pentanone | 97 | 17 | 45 | 145 | 30 | 165 |
| Acetone | 88 | 23 | 20 | 160 | 10 | 180 |
| Benzene | 99 | 9 | 75 | 125 | 65 | 135 |
| Bromobenzene[13] | 93 | 9 | 65 | 120 | 55 | 130 |
| Bromochloromethane | 99 | 9 | 70 | 125 | 60 | 135 |
| Bromodichloromethane | 100 | 9 | 70 | 130 | 60 | 135 |
| Bromoform | 96 | 13 | 55 | 135 | 45 | 150 |
| Bromomethane | 95 | 21 | 30 | 160 | 10 | 180 |
| Carbon disulfide | 103 | 19 | 45 | 160 | 30 | 180 |
| Carbon tetrachloride | 100 | 11 | 65 | 135 | 55 | 145 |
| Chlorobenzene | 99 | 8 | 75 | 125 | 65 | 130 |
| Chlorodibromomethane | 98 | 11 | 65 | 130 | 55 | 140 |
| Chloroethane | 98 | 20 | 40 | 155 | 20 | 175 |
| Chloroform | 98 | 9 | 70 | 125 | 65 | 135 |
| Chloromethane | 90 | 13 | 50 | 130 | 40 | 140 |

[12] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Methyl tert-butyl ether and Total Xylene although those compounds do appear on the target analyte list for method 8260 (Table C-1 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for MTBE during the LCS study. Xylene may be reported on a project-specific basis as a total number; however, for the purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section D.5 and for surrogate compounds in section D.6.

[13] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from fewer than four laboratories. Limits may be adjusted in the future as additional data become available.

## TABLE D-5. LCS CONTROL LIMITS FOR VOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8260 SOLID MATRIX[12]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| cis-1,2-Dichloroethene | 96 | 10 | 65 | 125 | 55 | 135 |
| cis-1,3-Dichloropropene | 99 | 9 | 70 | 125 | 65 | 135 |
| Dibromomethane | 100 | 9 | 75 | 130 | 65 | 135 |
| Dichlorodifluoromethane[13] | 85 | 17 | 35 | 135 | 15 | 155 |
| Ethylbenzene | 101 | 9 | 75 | 125 | 65 | 135 |
| Hexachlorobutadiene | 98 | 15 | 55 | 140 | 40 | 155 |
| Isopropylbenzene | 103 | 9 | 75 | 130 | 70 | 140 |
| m,p-Xylene | 102 | 8 | 80 | 125 | 70 | 135 |
| Methylene chloride | 97 | 14 | 55 | 140 | 40 | 155 |
| Naphthalene | 84 | 14 | 40 | 125 | 25 | 140 |
| n-Butylbenzene | 101 | 12 | 65 | 140 | 50 | 150 |
| n-Propylbenzene | 99 | 12 | 65 | 135 | 50 | 145 |
| o-Xylene | 101 | 8 | 75 | 125 | 70 | 135 |
| p-Isopropyltoluene | 104 | 10 | 75 | 135 | 65 | 140 |
| sec-Butylbenzene | 97 | 11 | 65 | 130 | 50 | 145 |
| Styrene | 101 | 9 | 75 | 125 | 65 | 135 |
| tert-Butylbenzene | 99 | 11 | 65 | 130 | 55 | 145 |
| Tetrachloroethene | 103 | 12 | 65 | 140 | 55 | 150 |
| Toluene | 99 | 9 | 70 | 125 | 60 | 135 |
| trans-1,2-Dichloroethene | 100 | 11 | 65 | 135 | 55 | 145 |
| trans-1,3-Dichloropropene | 96 | 10 | 65 | 125 | 55 | 140 |
| Trichloroethene | 101 | 8 | 75 | 125 | 70 | 130 |
| Trichlorofluoromethane | 106 | 27 | 25 | 185 | 10 | 215 |
| Vinyl chloride | 92 | 11 | 60 | 125 | 45 | 140 |

## TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| **Polynuclear Aromatics** | | | | | | |
| 2-Methylnaphthalene | 75.0 | 9.5 | 45 | 105 | 35 | 115 |
| Acenaphthene | 77.6 | 10.1 | 45 | 110 | 35 | 120 |
| Acenaphthylene | 78.5 | 9.4 | 50 | 105 | 40 | 115 |
| Anthracene | 83.0 | 9.7 | 55 | 110 | 45 | 120 |
| Benz[a]anthracene | 82.7 | 8.9 | 55 | 110 | 45 | 120 |
| Benzo[a]pyrene | 81.3 | 9.5 | 55 | 110 | 45 | 120 |
| Benzo[b]fluoranthene | 81.8 | 12.1 | 45 | 120 | 35 | 130 |
| Benzo[k]fluoranthene | 84.6 | 13.2 | 45 | 125 | 30 | 135 |

[14] A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

## TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| Benzo[g,h,i]perylene | 80.5 | 14.1 | 40 | 125 | 25 | 135 |
| Chrysene | 82.1 | 8.9 | 55 | 110 | 45 | 120 |
| Dibenz[a,h]anthracene | 84.7 | 14.1 | 40 | 125 | 30 | 140 |
| Fluoranthene | 85.2 | 10.4 | 55 | 115 | 45 | 125 |
| Fluorene | 80.6 | 10.3 | 50 | 110 | 40 | 120 |
| Indeno[1,2,3-cd]pyrene | 84.3 | 13.6 | 45 | 125 | 30 | 140 |
| Naphthalene | 70.8 | 10.5 | 40 | 100 | 30 | 115 |
| Phenanthrene | 84.0 | 11.0 | 50 | 115 | 40 | 130 |
| Pyrene | 88.6 | 13.2 | 50 | 130 | 35 | 140 |
| **Phenolic/Acidic** | | | | | | |
| 2,4,5-Trichlorophenol | 79.7 | 10.3 | 50 | 110 | 40 | 120 |
| 2,4,6-Trichlorophenol | 80.7 | 10.7 | 50 | 115 | 40 | 125 |
| 2,4-Dichlorophenol | 76.3 | 9.6 | 50 | 105 | 40 | 115 |
| 2,4-Dimethylphenol | 68.8 | 13.5 | 30 | 110 | 15 | 125 |
| 2,4-Dinitrophenol | 75.8 | 20.6 | 15 | 140 | 10 | 160 |
| 2-Chlorophenol | 71.3 | 11.4 | 35 | 105 | 25 | 115 |
| 2-Methylphenol | 73.3 | 11.7 | 40 | 110 | 25 | 120 |
| 2-Nitrophenol | 75.8 | 12.4 | 40 | 115 | 25 | 125 |
| 3-Methylphenol/4-Methylphenol | 71.3 | 13.0 | 30 | 110 | 20 | 125 |
| 4,6-Dinitro-2-methylphenol | 84.9 | 15.0 | 40 | 130 | 25 | 145 |
| 4-Chloro-3-methylphenol | 78.6 | 10.7 | 45 | 110 | 35 | 120 |
| Pentachlorophenol | 77.6 | 13.3 | 40 | 115 | 25 | 130 |
| **Basic** | | | | | | |
| 3,3'-Dichlorobenzidine | 65.2 | 15.3 | 20 | 110 | 10 | 125 |
| 4-Chloroaniline | 62.2 | 15.6 | 15 | 110 | 10 | 125 |
| **Phthalate Esters** | | | | | | |
| Bis(2-ethylhexyl) phthalate | 84.2 | 14.0 | 40 | 125 | 30 | 140 |
| Butyl benzyl phthalate | 81.1 | 11.7 | 45 | 115 | 35 | 130 |
| Di-n-butyl phthalate | 84.8 | 10.3 | 55 | 115 | 45 | 125 |
| Di-n-octyl phthalate | 87.4 | 16.6 | 35 | 135 | 20 | 155 |
| Diethyl phthalate | 79.2 | 12.9 | 40 | 120 | 30 | 130 |
| Dimethyl phthalate | 75.9 | 16.9 | 25 | 125 | 10 | 145 |
| **Nitrosoamines** | | | | | | |
| N-Nitrosodi-n-propylamine | 80.9 | 15.7 | 35 | 130 | 20 | 145 |
| N-Nitrosodimethylamine | 67.9 | 14.1 | 25 | 110 | 10 | 125 |
| N-Nitrosodiphenylamine | 79.6 | 10.6 | 50 | 110 | 35 | 120 |
| **Chlorinated Aliphatics** | | | | | | |
| Bis(2-chlorethoxy)methane | 76.2 | 10.2 | 45 | 105 | 35 | 115 |
| Bis(2-chloroethyl) ether | 73.3 | 12.3 | 35 | 110 | 25 | 120 |
| Bis(2-chloroisopropyl) ether | 78.2 | 17.5 | 25 | 130 | 10 | 150 |
| Hexachlorobutadiene | 65.2 | 12.6 | 25 | 105 | 15 | 115 |
| Hexachloroethane | 60.9 | 11.1 | 30 | 95 | 15 | 105 |

## TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| **Halogenated Aromatics** | | | | | | |
| 1,2,4-Trichlorobenzene | 71.7 | 11.6 | 35 | 105 | 25 | 120 |
| 1,2-Dichlorobenzene | 67.3 | 11.4 | 35 | 100 | 20 | 115 |
| 1,3-Dichlorobenzene | 64.8 | 10.9 | 30 | 100 | 20 | 110 |
| 1,4-Dichlorobenzene | 64.8 | 10.9 | 30 | 100 | 20 | 110 |
| 2-Chloronaphthalene | 76.5 | 9.3 | 50 | 105 | 40 | 115 |
| 4-Bromophenyl phenyl ether | 82.9 | 10.2 | 50 | 115 | 40 | 125 |
| 4-Chlorophenyl phenyl ether | 80.6 | 10.3 | 50 | 110 | 40 | 120 |
| Hexachlorobenzene | 82.3 | 10.0 | 50 | 110 | 40 | 120 |
| **Nitroaromatics** | | | | | | |
| 2,4-Dinitrotoluene | 84.3 | 11.2 | 50 | 120 | 40 | 130 |
| 2,6-Dinitrotoluene | 82.7 | 11.3 | 50 | 115 | 35 | 130 |
| 2-Nitroaniline | 81.8 | 11.2 | 50 | 115 | 35 | 125 |
| 3-Nitroaniline | 72.6 | 17.7 | 20 | 125 | 10 | 145 |
| 4-Nitroaniline | 77.2 | 13.7 | 35 | 120 | 20 | 130 |
| Nitrobenzene | 76.8 | 10.8 | 45 | 110 | 35 | 120 |
| **Neutral Aromatics** | | | | | | |
| Carbazole | 82.5 | 11.4 | 50 | 115 | 35 | 130 |
| Dibenzofuran | 80.3 | 8.8 | 55 | 105 | 45 | 115 |
| **Others** | | | | | | |
| 1,2-Diphenylhydrazine | 84.8 | 9.4 | 55 | 115 | 45 | 120 |
| Benzyl alcohol | 71.0 | 13.8 | 30 | 110 | 15 | 125 |
| Isophorone | 81.0 | 10.5 | 50 | 110 | 40 | 125 |

## TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 SOLID MATRIX[15]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| **Polynuclear Aromatics** | | | | | | |
| 2-Methylnaphthalene | 77.3 | 10.0 | 45 | 105 | 35 | 115 |
| Acenaphthene | 77.3 | 10.3 | 45 | 110 | 35 | 120 |
| Acenaphthylene | 75.7 | 10.4 | 45 | 105 | 35 | 115 |
| Anthracene | 79.9 | 9.0 | 55 | 105 | 45 | 115 |

[15] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spike in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, 1,2-Diphenylhydrazine, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

## TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 SOLID MATRIX[15]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| Benz[a]anthracene | 81.6 | 9.8 | 50 | 110 | 40 | 120 |
| Benzo[a]pyrene | 80.7 | 10.3 | 50 | 110 | 40 | 120 |
| Benzo[b]fluoranthene | 79.7 | 11.4 | 45 | 115 | 35 | 125 |
| Benzo[k]fluoranthene | 83.8 | 12.9 | 45 | 125 | 30 | 135 |
| Benzo[g,h,i]perylene | 81.8 | 14.7 | 40 | 125 | 25 | 140 |
| Chrysene | 82.6 | 9.9 | 55 | 110 | 45 | 120 |
| Dibenz[a,h]anthracene | 82.9 | 13.9 | 40 | 125 | 25 | 140 |
| Fluoranthene | 83.9 | 10.1 | 55 | 115 | 45 | 125 |
| Fluorene | 78.3 | 9.8 | 50 | 110 | 40 | 115 |
| Indeno[1,2,3-cd]pyrene | 79.7 | 13.8 | 40 | 120 | 25 | 135 |
| Naphthalene | 73.4 | 11.1 | 40 | 105 | 30 | 120 |
| Phenanthrene | 80.1 | 10.0 | 50 | 110 | 40 | 120 |
| Pyrene | 84.4 | 12.8 | 45 | 125 | 35 | 135 |
| **Phenolic/Acidic** | | | | | | |
| 2,4,5-Trichlorophenol | 80.1 | 10.4 | 50 | 110 | 40 | 120 |
| 2,4,6-Trichlorophenol | 76.3 | 11.0 | 45 | 110 | 30 | 120 |
| 2,4-Dichlorophenol | 77.2 | 10.9 | 45 | 110 | 35 | 120 |
| 2,4-Dimethylphenol | 67.3 | 11.9 | 30 | 105 | 20 | 115 |
| 2,4-Dinitrophenol | 72.6 | 20.0 | 15 | 130 | 10 | 150 |
| 2-Chlorophenol | 74.7 | 10.3 | 45 | 105 | 35 | 115 |
| 2-Methylphenol | 71.7 | 10.6 | 40 | 105 | 30 | 115 |
| 2-Nitrophenol | 76.2 | 11.5 | 40 | 110 | 30 | 120 |
| 3-Methylphenol/4-Methylphenol | 73.9 | 10.9 | 40 | 105 | 30 | 120 |
| 4,6-Dinitro-2-methylphenol | 83.1 | 18.0 | 30 | 135 | 10 | 155 |
| 4-Chloro-3-methylphenol | 79.5 | 11.1 | 45 | 115 | 35 | 125 |
| 4-Nitrophenol | 77.0 | 20.2 | 15 | 140 | 10 | 160 |
| Pentachlorophenol | 71.9 | 15.6 | 25 | 120 | 10 | 135 |
| Phenol | 69.7 | 10.2 | 40 | 100 | 30 | 110 |
| **Phthalate Esters** | | | | | | |
| Bis(2-ethylhexyl) phthalate | 87.4 | 13.3 | 45 | 125 | 35 | 140 |
| Butyl benzyl phthalate | 86.4 | 12.3 | 50 | 125 | 35 | 135 |
| Di-n-butyl phthalate | 83.2 | 9.1 | 55 | 110 | 45 | 120 |
| Di-n-octyl phthalate | 86.4 | 15.2 | 40 | 130 | 25 | 145 |
| Diethyl phthalate | 82.2 | 10.6 | 50 | 115 | 40 | 125 |
| Dimethyl phthalate | 79.6 | 10.2 | 50 | 110 | 40 | 120 |
| **Nitrosoamines** | | | | | | |
| N-Nitrosodi-n-propylamine | 76.8 | 12.3 | 40 | 115 | 30 | 125 |
| N-Nitrosodimethylamine | 66.1 | 15.9 | 20 | 115 | 10 | 130 |
| N-Nitrosodiphenylamine | 82.4 | 11.1 | 50 | 115 | 40 | 125 |
| **Chlorinated Aliphatics** | | | | | | |
| Bis(2-chlorethoxy)methane | 75.5 | 10.9 | 45 | 110 | 30 | 120 |
| Bis(2-chloroethyl) ether | 71.1 | 11.2 | 40 | 105 | 25 | 115 |
| Bis(2-chloroisopropyl) ether | 68.4 | 15.7 | 20 | 115 | 10 | 130 |

## TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 SOLID MATRIX[15]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| Hexachlorobutadiene | 78.2 | 12.9 | 40 | 115 | 25 | 130 |
| Hexachloroethane | 71.9 | 12.6 | 35 | 110 | 20 | 120 |
| **Halogenated Aromatics** | | | | | | |
| 1,2,4-Trichlorobenzene | 77.4 | 11.2 | 45 | 110 | 30 | 120 |
| 1,2-Dichlorobenzene | 70.9 | 8.7 | 45 | 95 | 35 | 105 |
| 1,3-Dichlorobenzene | 69.7 | 10.3 | 40 | 100 | 30 | 110 |
| 1,4-Dichlorobenzene | 69.0 | 11.4 | 35 | 105 | 25 | 115 |
| 2-Chloronaphthalene | 75.2 | 9.9 | 45 | 105 | 35 | 115 |
| 4-Bromophenyl phenyl ether | 81.7 | 11.8 | 45 | 115 | 35 | 130 |
| 4-Chlorophenyl phenyl ether | 79.6 | 10.7 | 45 | 110 | 35 | 120 |
| Hexachlorobenzene | 82.5 | 11.7 | 45 | 120 | 35 | 130 |
| **Nitroaromatics** | | | | | | |
| 2,4-Dinitrotoluene | 82.0 | 11.4 | 50 | 115 | 35 | 130 |
| 2,6-Dinitrotoluene | 80.2 | 10.7 | 50 | 110 | 35 | 125 |
| 2-Nitroaniline | 81.0 | 12.2 | 45 | 120 | 30 | 130 |
| 3-Nitroaniline | 68.8 | 13.8 | 25 | 110 | 15 | 125 |
| 4-Nitroaniline | 73.6 | 13.1 | 35 | 115 | 20 | 125 |
| Nitrobenzene | 77.2 | 11.9 | 40 | 115 | 30 | 125 |
| **Neutral Aromatics** | | | | | | |
| Carbazole | 80.4 | 12.3 | 45 | 115 | 30 | 130 |
| Dibenzofuran | 77.1 | 8.8 | 50 | 105 | 40 | 110 |
| **Others** | | | | | | |
| Benzyl alcohol | 70.9 | 17.4 | 20 | 125 | 10 | 140 |
| Isophorone | 77.0 | 11.4 | 45 | 110 | 30 | 125 |

## TABLE D-8. LCS CONTROL LIMITS FOR CHLORINATED HERBICIDES SW-846 METHOD 8151 WATER MATRIX[16]

| Analyte | Median | Lower Control Limit | Upper Control Limit |
|---|---|---|---|
| 2,4-D | 88 | 35 | 115 |
| 2,4-DB | 99 | 45 | 130 |
| 2,4,5-T | 83 | 35 | 110 |
| 2,4,5-TP (Silvex) | 87 | 50 | 115 |
| Dalapon | 62 | 40 | 110 |
| Dicamba | 86 | 60 | 110 |
| Dichloroprop | 91 | 70 | 120 |
| Dinoseb | 65 | 20 | 95 |
| MCPA | 93 | 60 | 145 |

[16] LCS control limits were generated using non-parametric statistics (see section D.1 for further explanation). LCS control limits are not available for MCPP, although the compound does appear on the target analyte list for method 8151 (Table C-5 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for the analyte during the LCS study.

**TABLE D-9. LCS CONTROL LIMITS FOR CHLORINATED HERBICIDES SW-846 METHOD 8151 SOLID MATRIX[17]**

| Analyte | Median | Lower Control Limit | Upper Control Limit |
|---|---|---|---|
| 2,4-D | 88 | 35 | 145 |
| 2,4-DB | 108 | 50 | 155 |
| 2,4,5-T | 86 | 45 | 135 |
| 2,4,5-TP (Silvex) | 90 | 45 | 125 |
| Dicamba | 90 | 55 | 110 |
| Dichloroprop | 99 | 75 | 140 |

[17] LCS control limits were generated using non-parametric statistics (see section D.1 for further explanation). LCS control limits are not available for Dalapon, MCPA, and MCPP, although those compounds do appear on the target analyte list for method 8151 (Table C-5 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

## TABLE D-10. LCS CONTROL LIMITS FOR POLYNUCLEAR AROMATIC HYDROCARBONS SW-846 METHOD 8310 WATER MATRIX[18]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| Acenaphthene | 70 | 11 | 35 | 105 | 25 | 115 |
| Acenaphthylene | 74 | 13 | 35 | 115 | 20 | 125 |
| Anthracene | 77 | 12 | 40 | 110 | 30 | 125 |
| Benz[a]anthracene | 81 | 11 | 50 | 110 | 40 | 125 |
| Benzo[a]pyrene | 79 | 11 | 45 | 115 | 35 | 125 |
| Benzo[b]fluoranthene | 82 | 10 | 50 | 110 | 40 | 125 |
| Benzo[k]fluoranthene | 79 | 10 | 50 | 110 | 40 | 120 |
| Benzo[g,h,i]perylene | 77 | 14 | 35 | 120 | 20 | 135 |
| Chrysene | 83 | 11 | 50 | 115 | 40 | 125 |
| Dibenz[a,h]anthracene | 64 | 15 | 20 | 110 | 10 | 125 |
| Fluoranthene | 82 | 11 | 50 | 115 | 35 | 125 |
| Fluorene | 69 | 11 | 35 | 105 | 25 | 115 |
| Indeno[1,2,3-cd]pyrene | 80 | 11 | 45 | 110 | 35 | 125 |
| Naphthalene | 68 | 12 | 35 | 105 | 20 | 115 |
| Phenanthrene | 80 | 13 | 40 | 120 | 25 | 135 |
| Pyrene | 80 | 9 | 50 | 110 | 45 | 115 |

## TABLE D-11. LCS CONTROL LIMITS FOR POLYNUCLEAR AROMATIC HYDROCARBONS SW-846 METHOD 8310 SOLID MATRIX[19]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| Acenaphthene | 71 | 12 | 35 | 110 | 20 | 120 |
| Acenaphthylene | 73 | 13 | 35 | 115 | 20 | 125 |
| Anthracene | 86 | 13 | 45 | 125 | 35 | 140 |
| Benz[a]anthracene | 78 | 9 | 50 | 105 | 40 | 115 |
| Benzo[a]pyrene | 86 | 15 | 40 | 135 | 25 | 150 |
| Benzo[b]fluoranthene | 89 | 11 | 55 | 120 | 45 | 130 |
| Benzo[k]fluoranthene | 84 | 12 | 50 | 120 | 35 | 135 |
| Benzo[g,h,i]perylene[20] | 85 | 10 | 55 | 115 | 45 | 125 |
| Chrysene | 87 | 11 | 55 | 120 | 45 | 130 |
| Dibenz[a,h]anthracene | 81 | 11 | 45 | 115 | 35 | 125 |
| Fluoranthene | 88 | 16 | 40 | 135 | 25 | 150 |
| Fluorene | 76 | 10 | 45 | 105 | 35 | 115 |
| Indeno[1,2,3-cd]pyrene | 95 | 13 | 55 | 135 | 45 | 145 |
| Naphthalene | 80 | 11 | 50 | 110 | 40 | 120 |
| Phenanthrene | 91 | 12 | 55 | 125 | 45 | 135 |
| Pyrene | 82 | 11 | 50 | 115 | 40 | 125 |

[18] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

[19] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

[20] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from fewer than four laboratories. Limits may be adjusted in the future as additional data become available.

## TABLE D-12. LCS CONTROL LIMITS FOR EXPLOSIVES SW-846 METHOD 8330 WATER MATRIX[21]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| 1,3,5-Trinitrobenzene | 102 | 13 | 65 | 140 | 50 | 150 |
| 1,3-Dinitrobenzene | 103 | 18 | 45 | 160 | 30 | 175 |
| 2,4-Dinitrotoluene | 98 | 12 | 60 | 135 | 50 | 145 |
| 2,6-Dinitrotoluene | 99 | 13 | 60 | 135 | 50 | 150 |
| 2,4,6-Trinitrotoluene (TNT) | 98 | 15 | 50 | 145 | 35 | 160 |
| 2-Amino-4,6-dinitrotoluene[22] | 101 | 17 | 50 | 155 | 35 | 170 |
| 2-Nitrotoluene | 88 | 15 | 45 | 135 | 30 | 150 |
| 3-Nitrotoluene | 90 | 14 | 50 | 130 | 35 | 145 |
| 4-Amino-2,6-dinitrotoluene[22] | 104 | 16 | 55 | 155 | 40 | 170 |
| 4-Nitrotoluene | 90 | 14 | 50 | 130 | 35 | 145 |
| Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) | 106 | 18 | 50 | 160 | 35 | 180 |
| Methyl-2,4,6-trinitrophenylnitramine (Tetryl)[22] | 98 | 25 | 20 | 175 | 10 | 200 |
| Nitrobenzene | 94 | 15 | 50 | 140 | 35 | 155 |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | 99 | 6 | 80 | 115 | 75 | 120 |

## TABLE D-13. LCS CONTROL LIMITS FOR EXPLOSIVES SW-846 METHOD 8330 SOLID MATRIX[23]

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| 1,3,5-Trinitrobenzene | 99 | 9 | 75 | 125 | 65 | 135 |
| 1,3-Dinitrobenzene | 102 | 8 | 80 | 125 | 70 | 135 |
| 2,4-Dinitrotoluene | 102 | 7 | 80 | 125 | 75 | 130 |
| 2,6-Dinitrotoluene | 100 | 7 | 80 | 120 | 70 | 130 |
| 2,4,6-Trinitrotoluene (TNT) | 99 | 14 | 55 | 140 | 45 | 155 |
| 2-Amino-4,6-dinitrotoluene | 102 | 7 | 80 | 125 | 75 | 130 |
| 2-Nitrotoluene | 101 | 7 | 80 | 125 | 70 | 130 |
| 3-Nitrotoluene | 100 | 7 | 75 | 120 | 70 | 130 |
| 4-Amino-2,6-dinitrotoluene | 101 | 7 | 80 | 125 | 75 | 130 |
| 4-Nitrotoluene | 101 | 8 | 75 | 125 | 70 | 135 |
| Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) | 103 | 10 | 70 | 135 | 65 | 145 |
| Nitrobenzene | 100 | 8 | 75 | 125 | 70 | 130 |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | 100 | 9 | 75 | 125 | 65 | 135 |

[21] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits were generated with data using solid phase extraction with acetonitrile only, without removing outliers from the data set (see section D.1 for further explanation).

[22] Provisional limits – LCS-CLs were generated with data from fewer than four laboratories. Limits may be adjusted in the future as additional data become available.

[23] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. Additional limits for poor performing compounds can be found in section D.5.

**TABLE D-14. LCS CONTROL LIMITS FOR ORGANOCHLORINE PESTICIDES SW-846 METHOD 8081 WATER MATRIX[24]**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| 4,4'-DDD | 88 | 20 | 25 | 150 | 10 | 170 |
| 4,4'-DDE | 87 | 18 | 35 | 140 | 15 | 160 |
| 4,4'-DDT | 92 | 15 | 45 | 140 | 30 | 155 |
| Aldrin | 83 | 19 | 25 | 140 | 10 | 155 |
| alpha-BHC | 94 | 11 | 60 | 130 | 50 | 140 |
| alpha-Chlordane | 93 | 10 | 65 | 125 | 55 | 135 |
| beta-BHC | 96 | 10 | 65 | 125 | 55 | 135 |
| delta-BHC | 91 | 15 | 45 | 135 | 30 | 150 |
| Dieldrin | 95 | 11 | 60 | 130 | 50 | 140 |
| Endosulfan I[25] | 80 | 10 | 50 | 110 | 40 | 120 |
| Endosulfan II | 79 | 17 | 30 | 130 | 10 | 150 |
| Endosulfan sulfate | 96 | 14 | 55 | 135 | 40 | 150 |
| Endrin | 95 | 13 | 55 | 135 | 45 | 145 |
| Endrin aldehyde | 96 | 14 | 55 | 135 | 40 | 150 |
| Endrin ketone | 102 | 8 | 75 | 125 | 70 | 135 |
| gamma-BHC | 82 | 18 | 25 | 135 | 10 | 155 |
| gamma-Chlordane | 94 | 11 | 60 | 125 | 50 | 135 |
| Heptachlor | 87 | 15 | 40 | 130 | 30 | 145 |
| Heptachlor epoxide | 96 | 11 | 60 | 130 | 50 | 140 |
| Methoxychlor | 103 | 16 | 55 | 150 | 40 | 165 |

[24] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Hexachlorobenzene and Toxaphene, although those compounds do appear on the target analyte list for method 8081 (Table C-8 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

[25] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from fewer than four laboratories. Limits may be adjusted in the future as additional data becomes available.

**TABLE D-15. LCS CONTROL LIMITS FOR ORGANOCHLORINE PESTICIDES SW-846 METHOD 8081 SOLID MATRIX[26]**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| 4,4'-DDD | 81 | 18 | 30 | 135 | 10 | 155 |
| 4,4'-DDE | 97 | 10 | 70 | 125 | 60 | 135 |
| 4,4'-DDT | 92 | 16 | 45 | 140 | 30 | 155 |
| Aldrin | 93 | 16 | 45 | 140 | 30 | 155 |
| alpha-BHC | 93 | 10 | 60 | 125 | 50 | 135 |
| alpha-Chlordane | 92 | 10 | 65 | 120 | 55 | 130 |
| beta-BHC | 95 | 11 | 60 | 125 | 50 | 135 |
| delta-BHC | 94 | 12 | 55 | 130 | 45 | 145 |
| Dieldrin | 96 | 10 | 65 | 125 | 55 | 135 |
| Endosulfan I | 74 | 20 | 15 | 135 | 10 | 155 |
| Endosulfan II | 89 | 17 | 35 | 140 | 20 | 160 |
| Endosulfan sulfate | 99 | 12 | 60 | 135 | 50 | 145 |
| Endrin | 97 | 12 | 60 | 135 | 50 | 145 |
| Endrin aldehyde | 92 | 18 | 35 | 145 | 20 | 165 |
| Endrin ketone | 100 | 11 | 65 | 135 | 55 | 145 |
| gamma-BHC | 91 | 11 | 60 | 125 | 50 | 135 |
| gamma-Chlordane | 96 | 10 | 65 | 125 | 55 | 135 |
| Heptachlor | 96 | 15 | 50 | 140 | 35 | 155 |
| Heptachlor epoxide | 98 | 11 | 65 | 130 | 55 | 140 |
| Methoxychlor | 100 | 14 | 55 | 145 | 45 | 155 |

**TABLE D-16. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846 METHOD 8082 WATER MATRIX[27]**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|---|---|---|---|---|
| Aroclor 1016 | 85 | 20 | 25 | 145 |
| Aroclor 1260 | 87 | 19 | 30 | 145 |

**TABLE D-17. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846 METHOD 8082 SOLID MATRIX[27]**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|---|---|---|---|---|
| Aroclor 1016 | 90 | 16 | 40 | 140 |
| Aroclor 1260 | 96 | 12 | 60 | 130 |

[26] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Hexachlorobenzene, Hexachlorocyclopentadiene, and Toxaphene, although these compounds do appear on the target analyte list for method 8081 (Table C-8 in Appendix DoD-C). Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. Additional limits for surrogate compounds can be found in section D.6.

[27] LCS control limits are not available for Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268, although those compounds do appear on the target analyte list for method 8082 (Table C-9 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

**TABLE D-18. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7470 WATER MATRIX[28]**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| Aluminum | 97 | 5 | 80 | 120 | 80 | 120 |
| Antimony | 98 | 4 | 80 | 120 | 80 | 120 |
| Arsenic | 98 | 4 | 80 | 120 | 80 | 120 |
| Barium | 99 | 4 | 80 | 120 | 80 | 120 |
| Beryllium | 99 | 4 | 80 | 120 | 80 | 120 |
| Cadmium | 100 | 4 | 80 | 120 | 80 | 120 |
| Calcium | 98 | 4 | 80 | 120 | 80 | 120 |
| Chromium | 100 | 4 | 80 | 120 | 80 | 120 |
| Cobalt | 99 | 3 | 80 | 120 | 80 | 120 |
| Copper | 99 | 3 | 80 | 120 | 80 | 120 |
| Iron | 102 | 4 | 80 | 120 | 80 | 120 |
| Lead | 99 | 4 | 80 | 120 | 80 | 120 |
| Magnesium | 98 | 4 | 80 | 120 | 80 | 120 |
| Manganese | 100 | 4 | 80 | 120 | 80 | 120 |
| Mercury | 100 | 5 | 80 | 120 | No ME | No ME |
| Molybdenum | 95 | 5 | 80 | 120 | 75 | 120 |
| Nickel | 100 | 4 | 80 | 120 | 80 | 120 |
| Potassium | 98 | 4 | 80 | 120 | 80 | 120 |
| Selenium | 98 | 6 | 80 | 120 | 75 | 120 |
| Silver | 97 | 5 | 80 | 120 | 75 | 120 |
| Sodium | 99 | 4 | 80 | 120 | 80 | 120 |
| Thallium | 97 | 4 | 80 | 120 | 80 | 120 |
| Vanadium | 99 | 4 | 80 | 120 | 80 | 120 |
| Zinc | 100 | 4 | 80 | 120 | 80 | 120 |

[28] The as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

**TABLE D-19. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7471 SOLID MATRIX[29]**

| Analyte | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit |
|---|---|---|---|---|---|---|
| Aluminum | 95 | 5 | 80 | 120 | 75 | 120 |
| Antimony | 96 | 5 | 80 | 120 | 75 | 120 |
| Arsenic | 95 | 4 | 80 | 120 | 80 | 120 |
| Barium | 98 | 3 | 80 | 120 | 80 | 120 |
| Beryllium | 99 | 4 | 80 | 120 | 80 | 120 |
| Cadmium | 97 | 4 | 80 | 120 | 80 | 120 |
| Calcium | 97 | 4 | 80 | 120 | 80 | 120 |
| Chromium | 99 | 5 | 80 | 120 | 80 | 120 |
| Cobalt | 98 | 4 | 80 | 120 | 80 | 120 |
| Copper | 97 | 3 | 80 | 120 | 80 | 120 |
| Iron | 100 | 4 | 80 | 120 | 80 | 120 |
| Lead | 95 | 4 | 80 | 120 | 80 | 120 |
| Magnesium | 96 | 3 | 80 | 120 | 80 | 120 |
| Manganese | 97 | 4 | 80 | 120 | 80 | 120 |
| Mercury | 100 | 6 | 80 | 120 | No ME | No ME |
| Molybdenum | 96 | 5 | 80 | 120 | 75 | 120 |
| Nickel | 97 | 4 | 80 | 120 | 80 | 120 |
| Potassium | 96 | 4 | 80 | 120 | 80 | 120 |
| Selenium | 93 | 4 | 80 | 120 | 75 | 120 |
| Silver | 96 | 7 | 75 | 120 | 70 | 125 |
| Sodium | 96 | 4 | 80 | 120 | 80 | 120 |
| Thallium | 94 | 4 | 80 | 120 | 80 | 120 |
| Vanadium | 99 | 3 | 80 | 120 | 80 | 120 |
| Zinc | 95 | 5 | 80 | 120 | 75 | 120 |

[29] Some as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

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