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Subchapter A. GENERAL PROVISIONS

252.1. Definitions: To be added as chapter develops.

252.2. Purpose.

The purpose of this chapter is to establish an accreditation program for environmental laboratories and to protect the public health, safety and the environment by ensuring the accuracy, precision and reliability of data generated by environmental laboratories.

252.3. Scope.

This chapter applies to environmental laboratories that test or analyze environmental samples taken for the purpose of testing or analysis as required by an environmental statute administered by the Department. The following fields of accreditation are included:

(1) Drinking Water.

(2) Wastewater or Non-Potable Water.

(3) Whole Effluent Toxicity Testing.

(4) Solid or Chemical Materials, including soils, sediments, and sludges.

252.4. General Requirements.

(a) Testing or analysis of environmental samples within the fields of accreditation identified in § 252.3 (relating to scope) shall be performed by an environmental laboratory accredited under this chapter.

(b) An environmental laboratory testing or analyzing environmental samples within the fields of accreditation identified in § 252.3 shall be accredited under this chapter and be in compliance with this chapter in order to generate data and perform analysis used to comply with an environmental statute.
252.5. NELAP Equivalency.

(a) An environmental laboratory may apply to the Department for NELAP accreditation in lieu of accreditation under this chapter for fields of accreditation comparable to those identified in § 252.3 (relating to scope).

(b) An environmental laboratory seeking NELAP accreditation shall:

(1) Submit a complete application as provided in Subchapter B (relating to application, fees, and supporting documents).

(2) Comply with Subchapter G (relating to miscellaneous provisions).

252.6. Accreditation-by-Rule.

(a) Purpose. Environmental laboratories performing testing or analysis described in this section shall be deemed to have accreditation-by-rule if the following general requirements are met:

(1) The environmental laboratory performs the testing or analysis in conformance with mandated methods.

(2) The environmental laboratory assures that samples for analysis are properly preserved, are in proper containers, do not exceed maximum holding times between collection and analysis and are handled in accordance with guidelines governing quality control established by the Department.

(3) The environmental laboratory has the other necessary permits under the applicable environmental protection acts and is operating under the acts and regulations promulgated thereunder and the terms and conditions of permits.

(4) Records of the testing or analysis are retained on-site and available to the Department upon request.

(b) Inappropriate activity. The Department may require an environmental laboratory deemed to have accreditation-by-rule to apply for, and obtain, environmental laboratory accreditation pursuant to Subchapter B (relating to application, fees, and supporting documents), or take other appropriate action, when the environmental laboratory is not in compliance with the conditions of the accreditation-by-rule or is conducting an activity that harms or presents a threat of harm to the
health, safety or welfare of the people or the environment of this Commonwealth.

(c) Public water suppliers. Measurements for turbidity, fluoridation operation, residual disinfectant concentration, temperature, pH, alkalinity, orthophosphates, silica, calcium and conductivity shall be performed by a person meeting the requirements of § 109.704 (relating to operator certification).

(d) Waste water facilities. Measurements for pH, chlorine, dissolved oxygen, sulfite, temperature, or turbidity may be performed by a person meeting the requirements of section ????? (relating to operator certification).

(e) Other facilities: Measurements for pH, chlorine, dissolved oxygen, sulfite, temperature, or turbidity shall be performed in accordance with guidelines established by the Department.
Subchapter B. APPLICATION, FEES, AND SUPPORTING DOCUMENTS

252.201. Application and Supporting Documents.

(a) An environmental laboratory seeking accreditation for one or more fields of accreditation described in § 252.3 (relating to scope), or that seeks to add a field of accreditation, shall apply to the Department for accreditation in writing on forms provided by the Department. The applicant laboratory shall provide other relevant material requested by the Department.

(b) An application for accreditation shall include the appropriate application fee and all requested information including at least one satisfactory round of the most recent department specified proficiency test sample results or an analytical data package for test categories where no accessible proficiency tests exist.

(c) Environmental laboratories maintained on separate premises, even though operated under the same management, shall be required to maintain distinct accreditation. Separate accreditation is not required for buildings on the same or adjoining grounds. If a mobile laboratory is operating independently within the state, separate accreditation is required.


(a) This section applies to a change in ownership that involves the purchase or lease of equipment and where greater than or equal to 75% of the analytical staff is retained. A change in ownership that involves the purchase or lease of equipment and where less than 75% of the analytical staff are retained shall be treated as a new laboratory and require the submission of a complete application.

(b) Within 10 days following a change in laboratory ownership, the new owner of an accredited laboratory shall notify the Department in writing about the change. Within 30 days following the change in laboratory ownership, an accredited laboratory shall do the following:

(1) Submit an accreditation transfer application, indicating any changes in the equipment, methodology and staffing.

(2) Pay the transfer application fee.
(3) In the event that outstanding enforcement actions against the laboratory exist at the time of sale or transfer, agree to correct the outstanding violations in accordance with a schedule that is acceptable to the Department.

(c) All open or pending enforcement actions shall be transferred with the accreditation.

(d) Failure to meet the conditions of this section will cause the previous accreditation to expire.

(e) The laboratory may operate under the previous accreditation until the Department makes a final decision on the transfer application. If the Department denies the transfer application, the laboratory is no longer accredited and the new owner shall submit an application under §252.201 (relating to application and supporting documents).

252.203. General Fee Structure. – To be developed

252.204. Out-of-state Laboratories.

(a) Out-of-state laboratories may apply for primary accreditation or secondary accreditation from the Department.

(1) **Primary Accreditation**: Out-of-state laboratories may apply to the Department for primary accreditation under the provisions of NELAP equivalency in Section 252.5 (relating to NELAP equivalency) or under this chapter.

(2) **Secondary Accreditation**:

(i) The Department will recognize accreditation granted by any primary NELAP-approved accrediting authority for the same fields of accreditation for which the Department is a primary NELAP accrediting authority.

(ii) The Department may recognize the accreditation of an environmental laboratory by another state if the standards for accreditation are substantially equivalent to those established under this chapter and the laboratory is physically located within the state granting accreditation.

(iii) An environmental laboratory seeking secondary accreditation from the Department shall:

(A) Submit a properly completed application.
(B) Submit a quality manual.

(C) Pay the appropriate fee.

(D) Submit a copy of a valid accreditation certificate from the primary accrediting authority.

(E) Submit a copy of all on-site assessments conducted by the primary accrediting authority in the last three years.

(F) Submit copies of all proficiency test sample results reported within the past 12 months.

(b) The Department may conduct an on-site evaluation or issue proficiency test samples to an out-of-state environmental laboratory seeking secondary accreditation for reasons which may include, but are not limited to, addressing complaints from the public, requests from Department personnel, discrepancies with sample results, on-site deficiencies, frequent errors in reporting data to the Department, and suspicions of fraud regarding data quality. The laboratory shall pay for travel costs in accordance with § 252.205 (relating to out-of-state reimbursement).

(c) If the out-of-state laboratory’s accreditation is revoked or suspended by the primary accrediting authority, the laboratory’s authorization to perform testing or analysis is automatically revoked. The environmental laboratory shall notify the Department of the revocation or suspension within 48 hours of notification by the primary accrediting authority.


In addition to the nonrefundable application fee, an out-of-state laboratory shall reimburse the Department for the following costs associated with on-site assessments necessitated by accreditation:

(1) Transportation costs, including but not limited to airfare, mileage, tolls, car rental, public transportation, and parking.

(2) Meals and lodging.

(3) Travel time for each assessor at an hourly rate of $50 per hour.
252.301. Laboratory Supervisor.

(a) Testing, analysis and reporting of data by an environmental laboratory shall be under the direct supervision of a laboratory supervisor. The laboratory supervisor shall certify that each test or analysis is accurate and valid and the test or analysis was performed in accordance with all conditions of accreditation. The Department may disqualify a laboratory supervisor who is responsible for the submission of inaccurate test or analysis results.

(b) A laboratory may appoint one or more laboratory supervisors for the appropriate fields of accreditation for which they are seeking accreditation.

(c) An individual shall not be the laboratory supervisor of more than one accredited environmental laboratory without authorization from the Department. Circumstances to be considered in the decision to grant such authorization shall include, but not be limited to the following:

1. The extent to which operating hours of the laboratories to be directed overlap.
2. The adequacy of supervision in each laboratory.
3. The availability of environmental laboratory services in the area served.

(d) A laboratory supervisor who is absent for a period of time exceeding 15 consecutive calendar days shall designate another staff member meeting the qualifications of a laboratory supervisor to temporarily perform this function. If this absence exceeds 65 consecutive calendar days, the laboratory shall notify the Department in writing.

252.302. Qualifications of the Laboratory Supervisor.

(a) A laboratory supervisor of an environmental laboratory engaged in chemical analysis shall have the following qualifications:

1. A bachelor’s degree in chemistry, physics, environmental science, physical sciences, or engineering.
2. At least 24 college semester credit hours in chemistry.
(3) At least two years of experience in the testing or analysis of
environmental samples in representative inorganic and organic
fields of accreditation for which the laboratory seeks to obtain or to
maintain accreditation. A master’s or doctoral degree in chemistry,
physics, environmental science, physical sciences, or engineering
may be substituted for one year of experience.

(b) A laboratory supervisor of an environmental laboratory limited to
inorganic chemical analysis, other than metals analysis, shall have the
following qualifications:

(1) At least an earned associate’s degree in the chemistry, physics,
environmental science, physical sciences, or engineering, or two
years of equivalent and successful college education.

(2) At least 16 college semester credit hours in chemistry.

(3) At least two years of experience in the testing or analysis of
environmental samples in representative fields of accreditation for
which the laboratory seeks to obtain or to maintain accreditation.

(c) A laboratory supervisor of an environmental laboratory engaged in
microbiological or biological analysis shall have the following
qualifications:

(1) A bachelor’s degree in microbiology, biology, chemistry,
environmental sciences, physical sciences or engineering.

(2) At least 16 college semester credit hours in general microbiology
and biology.

(3) At least two years of experience in the testing or analysis of
environmental samples in representative microbiological or
biological fields of accreditation for which the laboratory seeks to
obtain or to maintain accreditation. A master’s or doctoral degree
in microbiology, biology, chemistry, environmental sciences,
physical sciences or engineering may be substituted for one year of
experience.

(d) A laboratory supervisor of an environmental laboratory engaged in
microbiological analysis limited to fecal coliform, total coliform and
standard plate count shall have the following qualifications:

(1) An associate’s degree in microbiology, biology, chemistry,
environmental sciences, physical sciences or engineering.

(2) A minimum of four college semester credit hours in general
microbiology.
(3) Two years of equivalent and successful college education, including the microbiology requirement, may be substituted for the associate’s degree.

(4) One year of experience in the testing or analysis of environmental samples in representative fields of accreditation for which the laboratory seeks to obtain or to maintain accreditation.

(e) A laboratory supervisor of an environmental laboratory engaged in radiological analysis shall have the following qualifications:

(1) A bachelor’s degree in chemistry, physics or engineering.

(2) At least 24 college semester credit hours in chemistry.

(3) Two or more years of experience in the testing or analysis of environmental samples in representative fields of accreditation for which the laboratory seeks to obtain or to maintain accreditation. A master’s or doctoral degree in chemistry, physics or engineering may be substituted for one year experience.

(f) A laboratory supervisor of an environmental laboratory engaged in microscopic examination of asbestos or airborne fibers shall have the following qualifications:

(1) 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.

(2) 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.

(3) 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.

(g) Notwithstanding any other provision of this section, a full-time employee of a drinking water or sewage treatment facility who holds a valid treatment plant operator’s certificate appropriate to the nature and size of such facility shall be deemed to meet the educational and
experience requirements of laboratory supervisor for an environmental laboratory devoted exclusively to the examination of environmental samples taken within such facility system. Accreditation for such a water treatment facility or a sewage treatment facility shall be limited to the scope of that facility’s regulatory permit.

(h) A full-time employee of an industrial waste treatment facility with a minimum of one year of experience in environmental analysis shall be deemed to meet the requirements of laboratory supervisor for an environmental laboratory devoted exclusively to the examination of environmental samples taken within such facility for the scope of that facility’s regulatory permit.


(a) A person who does not meet the education credential requirements for laboratory supervisor but possess the requisite years of experience required by §252.302 (relating to qualifications of the laboratory supervisor) shall qualify as laboratory supervisor subject to the following conditions:

(1) The person must be a laboratory supervisor of the laboratory on the date the laboratory becomes subject to accreditation.

(2) The person must have been a laboratory supervisor in that laboratory for at least the previous 12 months for the fields of accreditation for which the laboratory has applied.

(b) A person will be approved as a laboratory supervisor for only those fields of accreditation for which the person has been laboratory supervisor in that laboratory for the previous 12 months or more.

(c) A person who is admitted as a laboratory supervisor under these conditions and leaves the laboratory may be admitted as laboratory supervisor for the same fields of accreditation in another laboratory.

252.304. Personnel Requirements.

(a) General Requirements for Laboratory Staff

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

(2) Laboratory personnel shall be responsible for complying with 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.

(b) Laboratory Management Responsibilities. The laboratory management shall be responsible for:

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

(2) Ensuring that technical laboratory staff or work cells have demonstrated capability in the activities for which they are responsible. Such demonstration must be documented.

(3) Ensuring that the training of each member of the technical staff is kept up-to-date by the following:

   (i) Documenting that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation that relate to each employee's job responsibilities.

   (ii) Documenting training courses or workshops on specific equipment, analytical techniques or laboratory procedures that relate to each employee's job responsibilities.

   (iii) Documenting participation in training courses in ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions. Evidence must be on file that demonstrates that each employee has read, acknowledged and understood their personal ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions.

(4) Analyst training will be considered up to date if an employee training file contains the following:

   (i) 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).

   (ii) Documentation of continued proficiency by at least one of the following once per year:
(A) Acceptable performance of a blind sample (single blind to the analyst).

(B) Another demonstration of capability.

(C) 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 5035/8260) would only require documentation for one of the test methods.

(D) At least four consecutive laboratory control samples with acceptable levels of precision and accuracy.

(E) If clauses (A) through (D) cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

(5) Documenting analytical and operational activities of the laboratory.

(6) Supervising personnel employed by the laboratory.

(7) Ensuring that sample acceptance criteria are verified and that samples are logged into the sample tracking system and properly labeled and stored.

(8) Documenting the quality of data reported by the laboratory.

(9) Developing a proactive program for prevention and detection of improper, unethical or illegal actions. Components of this program can include the following:

   (i) Internal proficiency testing (single and double blind).

   (ii) Post-analysis, electronic data and magnetic tape audits.

   (iii) Effective reward program to improve employee vigilance and co-monitoring.

   (iv) Separate standard operating procedures identifying appropriate and inappropriate laboratory and instrument manipulation practices.

(c) The laboratory shall maintain records, including records on demonstrated proficiency for each laboratory test method, on the relevant qualifications, training, skills and experience of the technical personnel.
252.305. Physical Facilities.

(a) Environment.

(1) An environmental laboratory shall have accommodations, test areas, energy sources, lighting, heating and ventilation necessary to assure proper performance of tests.

(2) The environment in which testing or analysis of environmental samples is undertaken shall not adversely affect the results or the required accuracy of measurement.

(3) The laboratory shall provide for the effective monitoring, control and recording of environmental conditions where monitoring or control of environmental conditions is specified in a test method or by regulation.

(4) In instances where monitoring or control of environmental conditions is specified in a test method or by regulation, the laboratory shall meet and document adherence to the requirements.

(b) Work Areas.

(1) Work spaces must be available to ensure an unencumbered work area. Work areas include:

   (i) Access and entryways to the laboratory.

   (ii) Sample receipt area.

   (iii) Sample storage area.

   (iv) Chemical and waste storage area.

   (v) Data handling and storage area.

(2) There shall be effective separation between neighboring areas when the activities therein are incompatible, including culture handling or incubation areas and volatile organic chemicals handling areas.

(3) Access to and use of all areas affecting the quality of these activities shall be defined and controlled.

(4) Adequate measures shall be taken to ensure that any contamination does not adversely affect data quality.

(a) The laboratory shall be furnished with items of equipment (including reference materials) required for the correct performance of tests or analyses for which accreditation is sought. In those cases where the laboratory uses equipment outside its permanent control, it shall ensure that the relevant requirements of this section are met.

(b) Equipment shall be properly maintained, inspected and cleaned. Maintenance procedures shall be documented.

(c) Any item of the equipment that has been subjected to overloading, mishandling, gives suspect results, or has otherwise been shown to be defective, shall be taken out of service and clearly identified until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous testing or analysis.

(d) Each item of equipment, including reference materials, shall be labeled, marked or otherwise identified to indicate its calibration status.

(e) Records shall be maintained of each major item of equipment significant to the testing or analysis performed. These records shall include documentation on all maintenance activities and reference materials verifications. The records shall include:

1. The name of the item of equipment.
2. The manufacturer’s name, type identification, and serial number or other unique identification.
3. Date received and date placed in service (if available).
4. Current location, where appropriate.
5. If available, condition when received (e.g. new, used, reconditioned).
6. Copy of the manufacturer’s instructions, where available.
7. Dates and results of calibrations or verifications and date of the next calibration or verification.
8. Details of maintenance carried out to date and planned for the future.
(9) History of damage, malfunction, modification or repair.

(f) Records shall be maintained for all reference materials, reagents, and support services utilized by the laboratory in the generation of analytical data.

(g) Supplies used for environmental testing shall meet the following minimum requirements:

(1) Analytical reagents.

   (i) Analytical reagent grade chemicals or equivalent are acceptable, unless individual procedures specify other reagent requirements.

   (ii) Stock and working standard solutions shall be checked regularly for signs of decomposition and expiration.

   (iii) Solutions shall be labeled with identification of the compound, concentration, date prepared, analyst who prepared solution and expiration date.

   (iv) Purchased chemicals, solutions, and standards shall be labeled with date of receipt, the date of expiration on the container, and the date when the container is opened.

   (v) When reagents are removed from a container, they shall be used entirely or the unused portion discarded. Unused portions of a reagent may not be returned to the original container.

   (vi) Compressed gases shall be of commercial grade, unless individual procedures specify other requirements.

(2) Glassware shall be cleaned and maintained properly as required by the test methodology.

(3) Thermometers.

   (i) A laboratory shall have access to a NIST – traceable thermometer where applicable.

   (ii) The calibration of working thermometers, with the exception of dial thermometers, shall be checked at least annually against a NIST – traceable certified thermometer and results recorded and documented per thermometer.
(iii) The calibration of dial-type thermometers shall be checked at least quarterly against a NIST–traceable thermometer and results recorded per thermometer.

(iv) Thermometers shall be labeled when calibrated and the correction factor recorded.

(h) Equipment used for testing or analysis of environmental samples shall meet the following minimum requirements:

(1) Analytical balances/pan balances.

(i) Records of balance calibration shall be kept for at least two ranges with a minimum class S or S-1 reference weights or equivalent (weights should be recertified every two years). Records showing daily (or before each use) functional/calibration checks for analytical balances and monthly functional/calibration checks for pan balances shall be maintained.

(ii) Balances shall be calibrated and serviced at a minimum of once per year and the service date recorded on the balance.

(iii) Balances may only be used with suitable support.

(2) pH Meters.

(i) A laboratory shall use a pH meter with appropriate electrode with scale graduations at least 0.1 pH units (calibrated + 0.1 pH units for each use period) with temperature correction.

(ii) Either a thermometer or a temperature sensor for automatic compensation shall be used.

(iii) Records shall be maintained including calibration daily or before each use, whichever is less frequent.

(iv) Aliquots of standard buffers shall be used only once.

(3) Conductivity Meter.

(i) A conductivity meter and probe of sufficient sensitivity shall be used.

(ii) Records shall be kept to show a daily or before each use calibration check, whichever is less frequent. Calibration shall be within the range of interest using standard solutions.
(iii) Records shall be kept showing that the cell constant is determined annually.

(4) Refrigeration Equipment.

(i) Thermometer(s) in each refrigerator shall be immersed in liquid to the appropriate immersion line.

(ii) Thermometers shall be graduated in increments no larger than 1°C.

(iii) Temperatures for each refrigerator shall be recorded for each day in use for laboratory activities.

(iv) Samples shall be stored in separate refrigerators from all standards where a potential for cross-contamination exists.

(v) Refrigerator temperature must be maintained between 1°C and 6°C (inclusive), and freezer temperature shall be less than 0°C.

(5) Visual Comparison Devices.

(i) Visual devices shall be calibrated according to manufacturer's specifications and/or test methodologies.

(ii) Results shall be recorded and maintained.

(6) Ovens/Incubators/Baths.

(i) Temperature shall be adequately controlled.

(ii) Records shall be kept to show that temperature is maintained (e.g., beginning and end of each use cycle or daily for extended drying periods).

252.307. Methodology.

(a) A laboratory shall follow the requirements for testing or analysis, sample collection, sample preservation and holding times specified in this section.

(b) A laboratory shall make copies of the analytical methods, Department regulations and Department guidance pertaining to testing or analysis of environmental samples available to the analysts.

(c) A laboratory shall select the analytical method for a specific test or analysis that meets the following criteria:

(1) The method is appropriate for the analyte and sample matrix.
(2) The method is the analytical method required by applicable state or federal regulations or permit or is an approved alternate method under subsection (i) (relating to alternate test approval).

(3) The method enables the laboratory to quantitate at levels required by the Department.

(d) When promulgated or approved alternative methods are not available that meet the requirements of subsection (c)(2), the Department may allow use of alternate or experimental procedures.

(e) Sample collection methods required by applicable state and federal laws or regulations or permit conditions shall be followed.

(f) Laboratories shall follow the sample preservation procedures and holding times required by state and federal regulations. If the sample preservation procedures and holding times are not required by state or federal regulations, laboratories shall follow the sample preservation procedures and holding time established in the analytical method.

(g) The limit of quantitation and limit of detection shall be determined for each analyte reported by a laboratory in accordance with a method specified by the Department. The Department may also require that the limit of detection be determined for a specific matrix.

(h) When a method of analysis specifies a validation procedure, the validation procedure shall be completed before samples can be analyzed and reported to the Department. The results of this validation procedure shall be documented and kept on file for the duration of use of the method and for a period of not less than 3 years after the method is no longer in use.

(i) Alternate test approval.

(1) EPA approval. The Department may permit the use of alternate methodologies if EPA has granted an approval for their use. The laboratory shall submit to the Department a copy of EPA’s written approval for the use of the alternate method.

(2) Experimental Procedures. The Department may allow alternate methods that use new or innovative technologies on a case-by-case basis.

(i) Initial requests for using emerging technology methods shall be made in writing to the Department. The request shall include the reasons for proposing the method and the potential scope of use for the method.
(ii) The Department will approve or deny the request within 90 days based on a demonstrated need for the emerging technology method.

(iii) If the request is granted, the Department will establish criteria for validating the method on a case-by-case basis.

(iv) If the method validation meets the established criteria, the Department shall permit the use of the method.

(j) Demonstration of Capability.

(1) Prior to acceptance and institution of any test method, satisfactory demonstration of method capability is required.

(2) Thereafter, continuing demonstration of method performance is required. (Subsection ??)

(3) In cases where a laboratory analyzes samples using a test method that has been in use by the laboratory before January 1, 2004, and there have been no significant changes in instrument type, personnel or test 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.

(4) In all cases, forms provided by or approved by the Department must be completed and retained by the laboratory and made available upon request. The laboratory must retain all associated supporting data necessary to reproduce the analytical results.

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

(6) In laboratories with a specialized work cell, the group as a unit must meet the above criteria and demonstration of capability must be fully documented;

(7) When a work cell is employed, and the members of the cell change, the new employees must work with an experienced analyst in that area of the work cell where they are employed. This new work cell must demonstrate acceptable performance through acceptable continuing performance checks. 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.
(8) If the entire work cell is changed the work cell must perform the
demonstration of capability.

(9) When a work cell is employed, the performance of the group must
be linked to the training record of the individual members of the
work cell.
252.401. Establishment Of Quality System.

An environmental laboratory shall establish and maintain a quality system appropriate to the type, range and volume of environmental testing activities it undertakes.

(1) The elements of this quality system shall be documented in the organization’s quality manual.

(2) The quality manual shall be available for use by the laboratory personnel.

(3) The laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

(4) The laboratory shall ensure that these policies and objectives are documented in a quality manual and communicated to, understood and implemented by, all laboratory personnel concerned.

(5) The quality assurance officer shall maintain and update the quality manual.


(a) 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 chapter.

(b) 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 assurance officer; the identification of all major organizational units which are to be covered by this quality manual and the effective date of the version;

(c) The quality manual and related quality documentation shall also contain:

(1) A quality policy statement, including objectives and commitments, by top management;
(2) The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;

(3) The relationship between management, technical operations, support services and the quality system;

(4) Procedures to ensure that all records required under this Chapter are retained;

(5) A document control system that provides procedures for control and maintenance of documentation and ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;

(6) Job descriptions of key staff and reference to the job description of other staff;

(7) 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 assurance officer(s), laboratory supervisor(s), and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager;

(8) Procedures for achieving traceability of measurements;

(9) A list of all test methods under which the laboratory performs its accredited testing;

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

(11) Reference to the calibration and/or verification test procedures used;

(12) Procedures for handling submitted samples;

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

(14) Reference to procedures for calibration, verification and maintenance of equipment;
(15) Reference to verification practices which may include interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

(16) Procedures for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

(17) The laboratory (management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

(18) Procedures for dealing with complaints;

(19) Procedures for protecting confidentiality (including national security concerns), and proprietary rights;

(20) Procedures for audits and data review;

(21) Procedures for establishing that personnel are adequately trained;

(22) Ethics policy statement developed by the laboratory and procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions;

(23) Reference to procedures for reporting analytical results; and

(24) A table of contents, and applicable lists of references and glossaries, and appendices.

252.403. Audits, Reviews and Corrective Actions.

(a) Internal Audits. The laboratory shall conduct annual internal audits to verify that it complies with the requirements of the laboratory’s quality system. The quality assurance officer shall plan and organize audits as required by a predetermined 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. Where the audit findings cast doubt on the correctness or validity of the laboratory’s calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose work was involved.
Managerial Review. The laboratory management shall conduct an annual review of its quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review shall take account of reports from managerial and supervisory personnel, the outcome of recent internal audits or assessments by external bodies, the results of interlaboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors. The laboratory shall have a procedure for review by management and maintain records of review findings and actions.

Audit Review. All audit and review findings and any corrective actions taken as a result of the findings shall be documented. The laboratory shall ensure that these actions are discharged within the agreed time frame as indicated in the quality manual and/or SOPs.

Performance Audits. In addition to periodic audits, the laboratory shall ensure the quality of test results by implementing checks to monitor the quality of the laboratory’s analytical activities. Examples of such checks are:

1. Internal quality control procedures using statistical techniques;
2. Participation in proficiency testing or other interlaboratory comparisons;
3. Use of certified reference materials and/or in-house quality control using secondary reference materials;
4. Replicate testing using the same or different test methods;
5. Re-testing of retained samples; and
6. Correlation of results for different but related analysis of a sample (for example, total phosphorus should be greater than or equal to orthophosphate).

Corrective Actions.

1. In addition to providing acceptance criteria and specific protocols for corrective actions in the method standard operating procedures, 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:

(i) Identify the individual(s) responsible for assessing each QC data type;

(ii) Identify the individual(s) responsible for initiating and/or recommending corrective actions;

(iii) Define how the analyst shall treat a data set if the associated QC measurements are unacceptable;

(iv) Specify how out-of-control situations and subsequent corrective actions are to be documented; and

(v) Specify procedures for management (including the QA officer) to review corrective action reports.

(2) 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 within the appropriate data qualifier(s).


(a) These general quality control principles shall apply 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 §252.405 through §252.408. The standards for any given test type shall assure that the applicable principles are addressed:

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

(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 detection limits and quantitation limits 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.

(c) 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.

(d) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist.

(e) The quality control protocols specified by the laboratory’s method manual shall be followed. The laboratory shall ensure that the essential standards provided in §252.405 through §252.408, 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.
252.405. Essential Quality Control Requirements – Chemistry.

(a) In addition to the requirements of §252.404, laboratories performing testing or analysis in the area of chemistry, shall comply with this section.

(b) The quality control protocols specified by the laboratory’s method manual shall be followed.

(c) Negative Control - Method Performance: 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.

   (1) 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.

   (2) Composition: The method blank shall consist of a matrix that is similar to the associated samples and is known to be free of the analytes of interest.

   (3) Evaluation Criteria and Corrective Action: If a contaminant is detected, the 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 shall be reprocessed or data shall be appropriately qualified if:

   (i) 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.
(ii) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

(d) Positive Control – Method Performance

(1) Laboratory Control Sample (LCS): 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.

(2) Frequency: Except for analytes for which no spiking solutions are available, a laboratory shall analyze a LCS 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. Where 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.

(3) Composition: The LCS shall be a controlled matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. The matrix spike may be used in place of the LCS if 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:

(i) 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:

(ii) 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.
(iii) 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.

(iv) 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.

(v) For methods that include 1-10 targets, spike all components;

(A). For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;

(B). For methods with more than 20 targets, spike at least 16 components.

(4) Evaluation Criteria and Corrective Action:

(i) The results of the individual batch LCS are calculated in percent recovery.

(ii) The laboratory shall document the calculation for percent recovery.

(iii) The individual LCS is compared to the acceptance criteria as published in the mandated test method.

(iv) 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.

(v) 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.

(vi) Samples analyzed along with a LCS determined to be “out of control” should be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.
(e) Sample Specific Controls

(1) 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.)

(2) 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.

(3) Matrix Spike; Matrix Spike Duplicates:

(i) 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.

(ii) 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 required mandated test method.

(iii) 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 components to be spiked are not specified by mandated method or by permit condition, the laboratory shall spike per the following:

(A). 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.

(B). 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.

(C). For methods that include 1-10 targets, spike all components;

(D). For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;

(E). For methods with more than 20 targets, spike at least 16 components.

(iv) 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) and relative percent difference (RPD). The laboratory shall document the calculation for relative percent difference. 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. For matrix spike results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

(4) Matrix Duplicates:

(i) 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.

(ii) Frequency: If the frequency of the analysis of matrix duplicates is not specified by the approved method, it shall be specified in the laboratory’s systematic planning process (e.g. Data Quality Objectives).

(iii) Composition: Matrix duplicates are performed on replicate aliquots of actual samples. (The composition of which is usually not known.)

(iv) 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. 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.

(5) Surrogate Spikes:

(i) 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.

(ii) Frequency: Except where the matrix precludes its use or when not available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate test methods.

(iii) Composition: Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method. 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.

(iv) 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.

(f) Detection Limits

(1) The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the mandated test method or applicable regulation, e.g., Method Detection Limit (MDL). If the protocol for determining detection limits is not specified in the approved test method, the laboratory shall select the procedure that reflects instrument limitations and the intended application of the test method.

(i) A detection limit study is not required for any component for which spiking solutions or quality control samples are not available such as temperature.

(ii) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).

(iii) Detection limits 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.

(iv) All sample processing steps of the analytical method shall be included in the determination of the detection limit.

(v) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.

(2) The laboratory must have established procedures to relate detection limits with quantitation limits.

(3) The test method’s quantitation limits must be established and must be above the detection limits.

(g) Data Reduction

(1) The procedures for data reduction, such as use of linear regression, shall be documented.

(h) Quality of Standards and Reagents

(1) The source of standards shall comply with §252.304.

(2) Reagent Quality, Water Quality and Checks:

(i) 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.

(ii) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.

(iii) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.

(i) Selectivity

(1) The laboratory shall develop and document acceptance criteria for retention time windows.
(2) 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.

(3) The laboratory shall document acceptance criteria for mass spectral tuning.

(j) Constant and Consistent Test Conditions

(1) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

(2) 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.

(a) In addition to the requirements of §252.404, laboratories that measure the toxicity and/or bioaccumulation of contaminants, including testing of effluents (whole effluent toxicity or WET), receiving waters, sediments, elutriates, leachates and soils, shall comply with this section. 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 matrix evaluated. Additional information can be found in the following methods manuals (or most recent edition): EPA/600/4-91/002, EPA/600/4-91/003, EPA/600/4-90/027F (WET testing), EPA/600/4-90/031 (general aquatic toxicity testing), EPA/600/R-94/025, EPA/600/R-94/024, EPA/503/R-91/001, EPA/823/B-98/004 (sediments and elutriates), EPA/600/3-88/029, EPA/600/3-89/013, ASTM E1598-94 AND ASTM 1676-97 (soils).

(b) Positive and Negative Controls

(1) Positive Control - Reference Toxicants - Reference toxicant tests indicate the sensitivity of the test organisms being used and demonstrate a laboratory’s ability to obtain consistent results with the test method. The laboratory must demonstrate its ability to obtain consistent results with reference toxicants before it performs toxicity tests with effluents or other environmental samples for regulatory compliance purposes.

(i) To meet this requirement, the intra-laboratory precision must be determined by performing five or more acceptable reference toxicant tests for each test method and species with different batches of organisms and appropriate negative controls (water, sediment, or soil).

(ii) A laboratory shall maintain control charts for the control performance and reference toxicant statistical endpoint (such as NOEC or ECp) and shall evaluate the intra-laboratory variability with a specific reference toxicant for each test method.

(2) Ongoing laboratory performance shall be demonstrated by performing regular reference toxicant tests for each test method and species in accordance with the minimum frequency requirements specified in Section ??.
(i) Intra-laboratory precision must be determined on an ongoing basis through the use of reference toxicant tests and plotted in quality control charts. The control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC50 for acute tests, or as appropriate hypothesis test values, such as the NOEC or NOAEC, over time within a laboratory.

(ii) 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 std. dev.); these values are re-calculated with each successive test result. 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 central tendency (i.e. the mode).

(iii) 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.

(iv) 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.

(v) Laboratories shall develop an acceptance/rejection policy for reference toxicant data which considers test dilution factor, test sensitivity (for hypothesis test
values), testing frequency, out-of-control test frequency, relative width of acceptance limits and degree of difference between test results and acceptance limits.

(vi) In the case of reference toxicant data which fails to meet acceptance criteria, the results of environmental toxicity tests conducted during the affected period may be suspect and regarded as provisional. In this case the test procedure is examined for defects and the test repeated if necessary, using a different batch of organisms, as soon as possible or the data is qualified.

(3) The frequency of reference toxicant testing shall comply with the EPA or state permitting authority requirements. The following minimum frequency shall be met:

(i) Each batch of test organisms obtained from an outside source, field collection or from laboratory spawning of field-collected species not amenable to routine laboratory culture (for example, sea urchins and bivalve mollusks) must be evaluated with a reference toxicant test of the same type as the environmental toxicity test within the seven days preceding the test or concurrently with the test.

(ii) Test organisms obtained from in-house laboratory cultures must be tested with reference toxicant tests at least once each month for each test method. If a species produced by in-house cultures is used only monthly, or less frequently, a reference toxicant test of the same type must be performed with each environmental toxicity test.

(iii) For test methods and species commonly used in the laboratory, but which are tested on a seasonal basis (e.g. sea urchin fertilization tests), reference toxicant tests must be conducted for each month the method is in use.

(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, dilution or control water, feeding, organism age, 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.

(c) Negative Control - Control, Brine Control, Control Sediment, Control Soil or Dilution Water

(1) In addition to negative controls specified by test method, permit or regulation additional negative controls shall be included when sample adjustments (for example addition of sodium hydroxide for pH adjustment or thiosulfate for dechlorination) or solvent carriers are used in the test.

(2) Test Acceptability Criteria (TAC) - The test acceptability criteria (for example, the whole-effluent chronic Ceriodaphnia test, requires 80% or greater survival and an average 15 young per female in the controls) as 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) 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 above.

(e) Sensitivity

(1) If the Dunnett’s procedure is used, the statistical minimum significant difference (SMSD) shall be calculated according to the formula specified by the test method and reported with the test results.

(2) Estimate the SMSD for non-normal distribution and or heterogenous variances.
(3) Point estimates: \((LC_p, IC_p, \text{ or } EC_p)\) - Confidence intervals shall be reported as a measure of the precision around the point estimate value.

(4) The SMSD shall be calculated and reported only for hypothesis test values, such as the NOEC or NOAEC.

(f) Selection of Appropriate Statistical Analysis Methods

(1) If required, methods of data analysis and endpoints are specified by language in the regulation, permit or the test method.

(2) Dose Response Curves - When required, 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.

(g) Selection and Use of Reagents and Standards

(1) 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.

(2) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in Section ?? above.

(3) Only reagent-grade water collected from distillation or deionization units (> 17 megohm resistivity) is used to prepare reagents.

(h) Constant and Consistent Test Conditions

(1) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid loss of cultures due to cross-contamination.

(2) 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.
(3) Air used for aeration of test solutions, dilution waters and cultures must be free of oil and fumes.

(4) 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 name(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.

(5) Instruments used for routine measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, and weight shall be calibrated, and/or standardized per manufacturer's instructions and section ?? . Temperature shall be calibrated per section ?? . All measurements and calibrations shall be documented.

(6) 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 1 °C of the selected test temperature, for the duration of the test. 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.

(7) Reagent grade water, prepared by any combination of distillation, reverse osmosis, ion exchange, activated carbon and particle filtration, shall meet the following requirements as verified by monthly measurement: conductivity less than or equal to 0.1 umhos or resistivity greater than or equal to 17 megohm, pH 5.5 to 7.5 S.U. and total residual chlorine non-detectable.

(8) Standard dilution water used for testing or culturing must be of sufficient quality to achieve 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 detection limit is greater than the method-specified limit, the laboratory must demonstrate that the analyte at the measured concentration or reported detection limit 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 data base provide guidance and data on acceptability and toxicity of individual metals and organic compounds.

(9) For each new batch of laboratory-prepared food or lot of commercial food used by the laboratory, the performance of organisms fed with the new food shall be compared with the performance of organisms fed with a food of known quality. If the food is used for culturing, its suitability is determined using a measure that evaluates the effect of food quality on survival and growth or reproduction of each of the relevant test species. Where applicable, foods used only in chronic toxicity tests are evaluated using the reference toxicant regularly employed in the laboratory QA program and compared with results of previous test(s) using a food of known quality. In the case of algae, rotifers or other cultured foods, which are collected as a continuous batch, the quality is assessed as described above, each time new nutrient stocks are prepared, a new starter culture is employed or when a significant change in culture conditions occurs. The laboratory shall have written procedures for the statistical evaluation of food acceptance.

(10) Food used to culture organisms used in bioaccumulation tests must be analyzed for the compounds to be measured in the bioaccumulation tests.

(11) 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.

(12) 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.

(13) All organisms in a test must be from the same source. Where available certified seeds are used for soil tests.
(14) 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.

(15) 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.

(16) 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.

(17) During aquatic chronic testing dissolved oxygen and pH shall be measured daily in at least one replicate of each concentration. In static-renewal tests, dissolved oxygen must be measured at both the beginning and end of each 24-hour exposure period and may be measured in old and new solutions prior to organism transfer, or after organism transfer; pH must be measured at the end of each exposure period (i.e. in old solutions).

(18) 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 ?? above. The laboratory shall also record each of these observations and water quality parameters upon the arrival of the organisms at the testing laboratory.

(19) 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.

(20) The maximum holding time of effluents (elapsed time from sample collection to first use in a test) shall not exceed 36 hours and the last use of the sample in test renewals shall not exceed 72 hours without the permission of the permitting authority.

(21) All samples shall be chilled to 4°C during or immediately after collection.

(22) Organisms obtained from an outside source must be from the same batch. Chronic tests shall have a minimum of four replicates per treatment.

(23) The control population of Ceriodaphnia in chronic effluent or receiving water tests shall contain no more than 20% males.

(24) 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.

(25) The test soils or sediments must be within the geochemical tolerance range of the test organism.

(26) 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 employee and the permitting authority.

(a) In addition to the requirements of §252.404, laboratories testing microbiological samples shall comply with this section.

(b) 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 each filtration series in the filtration technique, the laboratory shall prepare at least one beginning and one ending sterility check. When an interruption of more than 30 minutes occurs, the filtration funnels shall be re-sterilized.

(3) For pour plate technique, sterility blanks of the medium shall be made by pouring at least 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.

(c) 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). 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.

(d) 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, appropriate to the method. This shall be done prior to first use of the medium.

(e) 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 laboratory 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.

(f) Method Evaluation

(1) Laboratories are required to demonstrate proficiency with the test method prior to first use. Proficiency may be achieved by;

(i) Comparison to a method already approved for use in the laboratory,

(ii) Analyzing a minimum of ten spiked samples whose matrix is representative of a matrix normally submitted to the laboratory; or

(iii) Analyzing and passing one proficiency test series provided by an approved proficiency sample provider. The laboratory shall document the method used to demonstrate proficiency and shall maintain this documentation as long as the method is in use and for at least 5 years past the date of last use.

(2) Laboratories shall participate in the proficiency test programs identified by NELAP (5.4.2.j or 5.5.3.4). The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data.
(g) Test Performance

(1) 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).

(2) 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.

(h) Data Reduction. The calculations, data reduction and statistical interpretations specified by each test method shall be followed.

(i) 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.

(1) Culture media may be prepared from commercial dehydrated powders or may be purchased ready-to-use. Preparation from different chemical ingredients shall not be done unless the media is not available commercially or unless specified by the method.

(2) Reagents, commercial dehydrated powders and media shall be used within the shelf-life of the product and shall be documented according to .

(3) 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 on a monthly basis for chlorine residual, specific conductance, and heterotrophic bacteria plate count 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.)
(4) 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 and pH of the media.

(j) Selectivity

(1) 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.

(2) 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. Reference stocks that have been thawed shall not be re-frozen and re-used.

(3) Working stocks shall not be sequentially cultured more than five times and shall not be subcultured to replace reference stocks.

(k) Constant and Consistent Test Conditions

(1) 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.

(2) Laboratory Equipment

(i) 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. Calibration shall be done at least annually.

(ii) Autoclaves: 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.

(A). 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.

(B). 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.

(C). 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.

(D). Autoclave maintenance 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.

(E). The autoclave mechanical timing device shall be checked quarterly against a stopwatch and the actual time elapsed documented.

(iii) Volumetric Equipment: Volumetric equipment shall be calibrated as follows:

(A). equipment with movable parts such as automatic dispensers, dispensers/diluters, and mechanical hand pipettes shall be calibrated quarterly.

(B). equipment such as filter funnels, bottles, non-class A glassware, and other marked containers shall be calibrated once per lot prior to first use.

(C). the volume of the disposable volumetric equipment such as sample bottles, disposable pipettes, and micropippette tips shall be checked once per lot.

(iv) 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.

(v) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements.

(vi) Incubators, Water Baths, Ovens

(A). 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.

(B). 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.

(vii) Labware (Glassware and Plasticware)

(A). The laboratory shall have a documented procedure for washing labware, if applicable. Detergents designed for laboratory use must be used.

(B). Glassware shall be made of borosilicate or other non-corrosive material, free of chips and cracks, and shall have readable measurement marks.

(C). 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.

(D). Washed labware shall be tested at least once daily, each day of washing, for possible acid or alkaline residue by testing at least one piece of labware with a suitable pH indicator such as bromothymol blue. Records of tests shall be maintained.
252.408. Radiochemical Testing.

(a) In addition to the requirements of §252.404, laboratories that examine environmental samples by radiochemical analysis shall comply with this section. 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.

(b) Negative and Positive Controls

(1) Negative Controls

(i) 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 specified in the laboratory method manual. If the specified method blank acceptance criteria is not met the specified corrective action and contingencies 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.

(ii) In the case of gamma spectrometry where the sample matrix is simply aliquoted into a calibrated counting geometry, the method blank shall be of similar counting geometry that is empty or filled to similar volume with ASTM Type II water to partially simulate gamma attenuation due to a sample matrix.

(iii) There shall be no subtraction of the required method blank 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, calibration blank, 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.

(iv) The method blank sample shall be prepared with similar aliquot size to that of the routine samples for analysis. The method blank result and acceptance criteria shall be calculated in a manner that compensates for sample results based upon differing aliquot size.

(2) Positive Controls

(i) 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 used to assess the batch. The laboratory control sample result shall be assessed against the specific acceptance criteria specified in the laboratory method manual. If the specified laboratory control sample acceptance criteria is not met, the specified corrective action and contingencies shall be followed. The occurrence of a failed laboratory control sample acceptance criteria and the actions taken shall be noted in the laboratory report.

(ii) A matrix spike shall be performed at a frequency of one per preparation batch for those methods which do not utilize an internal standard or carrier, for which there is a chemical separation process, and where there is sufficient sample to do so. The exceptions are gross alpha, gross beta and tritium which shall require matrix spikes for aqueous samples. The results of this analysis shall be one of the quality control measures used to assess the batch. The matrix spike result shall be assessed against the specific acceptance criteria specified in the laboratory method manual. When the specified matrix spike acceptance criteria is not met, the specified corrective action and contingencies shall be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report. The lack of sufficient sample aliquot size to perform a matrix spike shall be noted in the laboratory report.

(iii) The activity of the laboratory control sample shall: (1) be two to ten times the detection limit or (2) at a level comparable to that of routine samples if the sample
activities are expected to exceed 10 times the detection limit.

(iv) The activity of the matrix spike analytes(s) shall be greater than ten times the detection limit.

(v) 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.

(vi) The matrix spike shall be prepared by adding a known activity of target analyte. 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. Where more than one analyte isotope is present above the specified detection limit, each isotope shall be assessed against the specified acceptance criteria.

(vii) 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. The isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated. Guidance language

(viii) The laboratory control sample shall be prepared with similar aliquot size to that of the routine samples for analyses.

(3) Other Controls

(i) 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 recovery for each sample result shall be one of the quality control measures used to assess the associated sample result acceptance. The tracer recovery shall be assessed against the specific
acceptance criteria specified in the laboratory method manual. When the specified tracer recovery acceptance criteria is not met, the specified corrective action and contingencies shall be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report.

(ii) Carrier - For those methods that utilize a carrier, each sample shall have an associated carrier recovery calculated and reported. 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 specified in the laboratory method manual. When the specified carrier recovery acceptance criteria is not met, the specified corrective action and contingencies shall be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report.

(c) Analytical Variability/Reproducibility

(1) 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 used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria specified in the laboratory method manual. When the specified replicate acceptance criteria are not met, the specified corrective action and contingencies shall be followed. The corrective action shall consider the fact that sample inhomogeneity may be a cause of the failed replicate acceptance criteria. The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report.

(2) For low level samples (less than approximately three times the detection limit) 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) Method Evaluation: In order to ensure the accuracy of the reported result, the following procedures shall be in place:
(1) An initial demonstration of capability shall be performed prior to the analysis of any samples and with a significant change in instrument type, personnel or method.

(2) Proficiency Test Samples - The results of such analysis shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

(e) Radiation Measurement System Calibration: 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. This section addresses those practices that are necessary for proper calibration and those requirements of section 5.9.4.2 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

(1) Initial Instrument Calibration:

(i) 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 5.9.4.2.1 are not applicable to radiochemical method calibrations except mass attenuation in gas-proportional counting and sample quench in liquid scintillation counting. Radiochemistry analytical instruments are subject to calibration when purchased, when the instrument is serviced, when the instrument is moved and when the instrument setting(s) have been changed.

(ii) Instrument calibration shall be performed with reference standards that have the same general characteristics (i.e., geometry, homogeneity, density, etc.) as the associated samples.

(iii) The frequency of calibration shall be specified in the laboratory method manual if not addressed 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.

(2) Continuing Instrument Calibration Verification: Calibration verification checks shall be performed using appropriate check sources and monitored with control charts or tolerance charts. 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. For alpha and gamma spectroscopy systems, the instrument calibration verification shall include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.

(i) For gamma spectroscopy systems, the calibration verification checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.

(ii) For alpha spectroscopy systems, the calibration verification 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.

(iii) For gas-proportional and liquid scintillation counters, the calibration verification check for counting efficiency shall be performed on a day of use basis. Verification of instrument calibration does not directly verify secondary calibrations, e.g., the mass efficiency curve or the quench curve.

(iv) For scintillation counters the calibration verification for counting efficiency shall be performed on a day of use basis.

(3) Background Measurement: Background measurements shall be made on a regular basis and monitored using control charts or tolerance charts. These values are subtracted from the total measured activity in the determination of the sample activity.

(i) For gamma spectroscopy systems, background measurements shall be performed on at least a monthly basis.

(ii) For alpha spectroscopy systems, background measurements shall be performed on at least a monthly basis.

(iii) For gas-proportional counters background measurements shall be performed on a weekly basis.
(iv) For scintillation counters, background measurements shall be performed each day of use.

(f) Detection Limits 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. The procedures used to determine detection limits must be documented and consistent with mandated method or regulation.

(g) Data Reduction

(1) 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.

(h) Quality of Standards and Reagents

(1) The quality control program shall establish and maintain provisions for radionuclide standards.

(i) Reference standards that are used in a radiochemical 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 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.

(ii) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.

(iii) 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.

(2) All reagents used shall be analytical reagent grade or better.

(i) Constant and Consistent Test Conditions
(1) To prevent incorrect analysis results caused by the spread of contamination among samples, the laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

(2) For gamma spectrometry systems, background check measurements shall be performed each day of use.

(3) For alpha spectrometry systems, background check measurements shall be performed except when using the electro-plating method of sample preparation.

(4) For gas-proportional counter systems, background check measurements shall be performed each day of use.
Subchapter E. PROFICIENCY TEST SAMPLE REQUIREMENTS

252.501. Proficiency Test Sample Requirements.

(a). An environmental laboratory shall participate in proficiency testing studies, where available. Available fields of proficiency testing studies shall be those listed by NELAP.

(b). A laboratory applying for accreditation pursuant to this chapter shall successfully analyze at least one single blind, single concentration proficiency test study for each field of accreditation.

(c). A laboratory accredited pursuant to this chapter shall successfully analyze at least one single blind, single concentration proficiency test study for each field of accreditation the laboratory is accredited at least once every 12 months.

(d). Proficiency test studies must be purchased at the laboratory’s expense directly from suppliers approved by NELAP as a Proficiency Test Provider.

(e). To retain accreditation, a laboratory must maintain a history of at least 2 acceptable proficiency test studies out of the 3 most recent proficiency test studies.

(f). If a proficiency test study for a field of accreditation is not successfully analyzed at least once every 12 months or incorrectly analyzed, the laboratory’s accreditation will be suspended, revoked, or denied as provided in Sections ??

(g). A laboratory shall ensure that all proficiency test study samples are handled (i.e. managed, analyzed, and reported) in the same manner as real environmental samples and utilize the same staff, procedures, equipment, facilities, number of replicates, and methods for the routine analysis of the analyte.

(h). A laboratory shall not send any proficiency test study, or a portion of a proficiency test study, to another laboratory for analysis for which it seeks accreditation or is accredited.

(i). A laboratory shall not analyze a proficiency test study, or a portion of a proficiency test study, for another laboratory for which the sending laboratory seeks accreditation or is accredited.

(j). A laboratory shall not communicate with another laboratory, including other laboratories under common ownership, concerning the proficiency test study.
(k). A laboratory shall not attempt to obtain the prepared value of a proficiency test study from the proficiency test study provider.

(l). If a laboratory fails a proficiency test study, it shall determine the cause for the failure and take any necessary corrective action. The laboratory shall document the investigation and corrective action.

(m). The laboratory shall direct the proficiency test study Provider to report laboratory proficiency test study performance results directly to the Department at the same time that it reports the results to the laboratory.

(n). Laboratories shall maintain copies of all raw data associated with proficiency test studies for at least 5 years.
252.601. On-site Assessments.

(a) Prior to accrediting a laboratory, the Department will perform an on-site evaluation of the laboratory. The Department will arrange to conduct the initial on-site evaluation at a time convenient to both parties.

(b) The Department will conduct an on-site assessment of an accredited laboratory at least once every two years.

(c) The Department may conduct additional on-site assessments, announced or unannounced, of a laboratory whenever such an assessment is necessary to determine the extent of the laboratory’s compliance with the conditions of accreditation and the requirements of this chapter.

(d) The Department shall provide the laboratory with an on-site assessment report documenting any deficiencies found by the Department.

(e) A laboratory shall submit a corrective action report to the Department within 90 days of receipt of an on-site assessment report where the Department has found deficiencies. The corrective action report shall include the action the laboratory will implement to correct each deficiency and the time period required to accomplish the corrective action.

(f) Unless otherwise approved by the Department, all deficiencies shall be corrected within 90 days of receipt of the on-site assessment report.

(g) The Department may extend the period of implementing corrective actions for a maximum of 30 days upon receipt of the laboratory’s written petition and corrective action report, where the laboratory must take one or more of the following actions:

(1). Purchase new equipment;

(2). Revision of a quality assurance plan or standard operating procedure;

(3). Replacement of significant laboratory personnel; or
(4). Repeating a demonstration of capability study;

(h) If the laboratory fails to submit a corrective action report that is acceptable to the Department, the Department may suspend or revoke the laboratory’s accreditation for any or all fields of accreditation.

(i) If the laboratory fails to implement the corrective actions within the timeframes stated in the corrective action report, accreditation for fields of accreditation, specific methods, or analytes within those fields of accreditation shall be revoked.
252.701. Expiration of Application.

If a laboratory has not submitted all necessary application materials within one year from the date the application is received by the Department, the application shall expire. If an accreditation application expires pursuant to this section, a laboratory must submit a new application and pay the appropriate fee to become accredited pursuant to this chapter.

252.702. Denial of Application.

(a). The Department shall deny an application for accreditation or application for renewal of accreditation under the following circumstances:

(1). The laboratory is in continuing violation of, or demonstrates an inability or lack of intention to comply with, the provisions of this chapter or other laws administered by the Department. The Department may grant accreditation to a laboratory that has corrected or is in the process of correcting a violation to the satisfaction of the Department.

(2). A laboratory voluntarily relinquishes its certificate of accreditation, fails to correct outstanding violations, and six months have not elapsed since the laboratory voluntarily relinquished its certificate of accreditation.

(3). An application for accreditation or application for renewal of accreditation may be denied for due cause, including, but not limited to the following:

(4). Falsifying analyses, selectively reporting data, making misrepresentations to the Department, misrepresenting any fact pertinent to receiving or maintaining accreditation, or engaging in other unethical or fraudulent practices.

(5). Analysis of proficiency test samples by personnel other than the analysts associated with the routine analysis of compliance samples in the laboratory.

(6). Failure to submit a complete application.

(7). Failure to pay required fees.
(8). Failure of laboratory staff to meet the personnel qualifications of education, training, and experience.

(9). Failure to successfully analyze and report proficiency test samples as required by this chapter.

(10). Failure to respond to an assessment report from the on-site assessment with a corrective action report within 30 calendar days after receipt of the assessment report.

(11). Failure to implement the corrective actions detailed in the corrective action report within a time frame approved by the Department.

(12). Failure to implement a quality system.

(13). Failure to pass an on-site assessment.

(14). Denial of entry during normal business hours for an on-site assessment.

(b). If a laboratory does not correct deficiencies identified in an on-site assessment report, the laboratory must wait six months before reapplying for accreditation.

252.703. Revocation.

(a). The Department may revoke a laboratory’s accreditation, in part or in total, for one or more of the following reasons:

(1). Failure to correct deficiencies identified during an on-site assessment of the laboratory.

(2). Failure of a laboratory that has been suspended to correct all outstanding violations within six months of the effective date of the suspension.

(3). Failure to submit an acceptable corrective action report in response to an assessment report and failure to implement corrective action related to deficiencies found during an on-site assessment.

(4). After being suspended due to failure of proficiency testing samples, if the laboratory’s analysis of the next proficiency testing study results in three consecutively failed proficiency testing studies, accreditation will be revoked for each affected accredited field of accreditation.
(b). A laboratory may be subject to total revocation for one or more of the following reasons:

(1) Violation of a condition of accreditation.

(2) Violation of a statute, this title or an order of the Department.

(3) Falsifying analyses, selectively reporting data, making misrepresentations to the Department, misrepresenting a fact pertinent to receiving or maintaining accreditation, or engaging in other unethical or fraudulent practices.

(4) Analysis of proficiency test samples by personnel other than the analysts associated with the routine analysis of compliance samples in the laboratory.

(5) Failure to respond with a corrective action report within the required timeframes.

(6) Failure to participate in the proficiency testing program as required by this chapter.

(7) Misrepresentation of material fact pertinent to receiving and maintaining accreditation.

(8) Denial of entry during normal business hours for an on-site assessment.

(9) Failure to remit the accreditation fees.

(c). The laboratory may continue to test or analyze environmental samples for those fields of accreditation, methods and analytes not revoked.

(d). Upon revocation of accreditation, an environmental laboratory shall return the certificate of accreditation to the Department within 48 hours.

(e). Within 72 hours of receiving notice of the revocation of accreditation from the Department, the laboratory shall notify each of its customers in writing of the revocation on a form approved by the Department.
252.704. Suspension.

(a). The Department may suspend a laboratory’s accreditation in total or in part for one or more of the following reasons:

(1). The Department finds during an on-site assessment or inspection that protection of the public interest, safety or welfare requires emergency action.

(2). Failure to complete proficiency testing studies and maintain a history of at least 2 successful proficiency testing studies for each affected field of accreditation out of the 3 most recent proficiency testing studies.

(3). Failure to notify the Department of changes to accreditation criteria pursuant to § _____ (relating to __________).

(4). Failure to maintain a quality system.

(5). To employ staff that meets the personnel qualifications for education, training and experience. (Richard – disregard deletion of failure!)

(b). A laboratory may continue to test or analyze environmental samples for those fields of accreditation not affected by the suspension.

(c). A laboratory shall correct all outstanding violations within six months of the effective date of the suspension in order to regain accreditation.

(d). Denial of access to the Department during normal business hours shall result in immediate suspension of accreditation of the laboratory. Upon notice from the Department, the laboratory shall immediately cease testing or analysis of environmental samples.

(e). Within 72 hours of receiving notice of the suspension of accreditation from the Department, the laboratory shall notify each of its customers in writing of the suspension on a form approved by the Department.

252.705. Use of Accreditation.

(a). Laboratories accredited by the Department shall:
(1). Post or display their most recent certificate of accreditation, including their fields of accreditation, in a prominent place in the laboratory.

(2). Make accurate statements concerning their fields of accreditation and accreditation status.

(3). Not use their certificate of accreditation, accreditation status or the Department’s logo to imply endorsement by the Department.

(b). Laboratories using the Department’s name, making reference to its accreditation status or using the Department’s logo in catalogs, advertising, business solicitations, proposals, quotations, laboratory analytical reports or other materials, shall:

(1). Distinguish between proposed testing for which the laboratory is accredited and the proposed testing for which the laboratory is not accredited.

(2). Include the laboratory’s accreditation number or other identifier.

(c). Upon suspension, revocation or withdrawal of accreditation, a laboratory shall:

(1). Discontinue use of all catalogs, advertising, business solicitations, proposals, quotations, laboratory analytical results or other materials that contain reference to the laboratory’s past accreditation status and/or display the Department’s logo.

(2). Return certificates of accreditation to the Department within 48 hours.

(d). NELAP accredited laboratories shall accompany the Department’s name or the NELAC/NELAP logo with at least the phrase “NELAP accredited,” the laboratory’s accreditation number or other identifier when using the Department’s name or the NELAC/NELAP logo on general literature such as catalogs, advertising, business solicitations, proposals, quotations, laboratory analytical reports or other materials.

(e). NELAP accredited laboratories shall not use their NELAP certificate, NELAP accreditation status or NELAC/NELAP logo to imply endorsement by the Department.
252.706. Accreditation Renewal

(a). Applications for accreditation renewal shall be submitted annually to the Department at least 60 calendar days prior to the expiration date of the current certificate of accreditation. Applications shall be made under Subchapter B (relating to application, fees and supporting documents) and shall be accompanied by the appropriate fee.

(b). Failure to submit an application for renewal in accordance with this section will result in a lapse in accreditation if the Department has not approved the renewal application prior to the expiration of the certificate of accreditation. If a lapse in accreditation occurs, the laboratory shall cease all testing or analysis of environmental samples for the affected fields of accreditation.

252.707. Record keeping.

(a). An environmental laboratory shall maintain records in a manner accessible by the Department.

(b). Records required under this Chapter shall be maintained for a minimum of 5 years unless otherwise specified.

(c). An environmental laboratory shall have a plan to ensure that records are maintained or transferred in the event that the laboratory transfers ownership or terminates operations.

252.708. Subcontracting.

(a). An environmental laboratory shall not subcontract testing or analysis covered under this Chapter to a laboratory that is not accredited and in compliance with this Chapter.

(b). The subcontracted environmental laboratory shall be indicated on the final report.

252.709. Reporting and Notification Requirements.

For testing or analysis conducted pursuant to Chapter 109 (relating to safe drinking water):
(1). A laboratory accredited under this Chapter shall submit to the Department, on forms provided by the Department, the results of test measurements or analyses performed by the laboratory under this Chapter. These results shall be reported within either the first 10 days following the month in which the result is determined or the first 10 days following the end of the required monitoring period as stipulated by the Department, whichever is shorter.

(2). A laboratory accredited under this Chapter shall, whenever an MCL, MRDL or a treatment technique performance requirement under § 109.202 (relating to state MCLs, MRDLs and treatment technique requirements) is violated, or a sample result requires the collection of check samples under § 109.301 (relating to general monitoring requirements):

   (i.) Notify the public water supplier by telephone within 1 hour of the laboratory’s determination. If the supplier cannot be reached within that time, notify the Department by telephone within 2 hours of the determination. If the Department cannot be reached due to an occurrence during weekend, holiday or evening hours, notify the Department by phone within 2 hours of the beginning of the next business day.

   (ii.) Notify the Department in writing within 24 hours of the determination. For the purpose of determining compliance with this requirement, the postmark, if the notice is mailed, or the date the notice is received by the Department, whichever is earlier, will be used.