CHAPTER 4740

DEPARTMENT OF HEALTH

LABORATORIES; ACCREDITATION REQUIREMENTS

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4740.2010 DEFINITIONS.

Subpart 1. **Scope.** The terms used in parts 4740.2050 to 4740.2120 have the meanings given them in this part and in the National Environmental Laboratory Accreditation Conference (NELAC) Standards, chapters 1 to 6, effective July 1, 2005, or a more current revision, provided the revision is in effect, upon the date it becomes effective. The standards are incorporated by reference, are not subject to frequent change, and are available on the Internet at http://www.epa.gov/nelac or by contacting the National Technical Information Service in the United States Department of Commerce.

Subp. 2. **Acceptable performance or acceptable results.** "Acceptable performance" or "acceptable results" means analytical test results generated by a laboratory using methods as specified in part 4740.2060 that fall within the acceptance range allowed by the approved provider.

Subp. 3. **Approved provider or approved PT provider.** "Approved provider" or "approved PT provider" means a provider of proficiency testing samples that the commissioner has determined meets the requirements of part 4740.2075.

Subp. 4. **Base certification.** "Base certification" means acknowledgment by the commissioner that a laboratory has the policies, procedures, equipment, and practices to produce reliable data in the analysis of environmental analytes.

Subp. 5. **Batch.** "Batch" means one to 20 environmental samples of the same matrix that are prepared together with the same process and personnel, using the same lot of reagents, with the maximum time between the start of processing of the first sample and the start of processing of the last sample being 24 hours, unless the method requirements are more stringent.

Subp. 6. **Bias.** "Bias" means the systematic or persistent distortion of a measurement system that causes errors in one direction, so that the expected sample measurement is different from the true value.

Subp. 7. **Calibration.** "Calibration" means testing an instrument's response by analyzing a series of analyte standards of differing concentrations, which are plotted on a graph that defines the instrument's linearity and dynamic range.

Subp. 8. **Calibration range.** "Calibration range" means the concentrations between and including the concentration of the lowest calibration standard at or above the detection limit and the highest concentration at which linearity has been established.

Subp. 9. **Certified test category or test category.** "Certified test category" or "test category" means a group of analytes available for certification. The analysis of the analytes is intended to test for compliance with specific environmental programs.

Subp. 10. **Certification.** "Certification" means the written acknowledgment of a laboratory's demonstrated capability to perform tests for a specific purpose.

Subp. 11. **Chain of custody.** "Chain of custody" means the procedures and records that document the possession and handling of samples from collection through disposal.

Subp. 12. **Chemical materials.** "Chemical materials" means a product or by-product of an industrial process or collection mechanism that results in a matrix not otherwise defined in subpart 30.

Subp. 13. **Commissioner.** "Commissioner" means the commissioner of health or the commissioner's designee.

Subp. 14. **Corrective action.** "Corrective action" means an action taken by the laboratory to eliminate or correct the causes of an existing nonconformance to prevent the recurrence of the nonconformance.

Subp. 15. **Corrective action plan.** "Corrective action plan" means a report, including specific items addressed and a specific date of completion, generated by a laboratory in response to deficiencies.

Subp. 16. **Deficiency or deviation.** "Deficiency" or "deviation" means a failure of the laboratory to meet any of the requirements in parts 4740.2010 to 4740.2120.

Subp. 17. **Denial.** "Denial" means the commissioner's refusal to certify a laboratory after submission of an application.

Subp. 18. **Document.** "Document" means any written or pictorial information describing, defining, specifying, reporting, or certifying any activities, requirements, procedures, or results.

Subp. 19. **Drinking water.** "Drinking water" means water used or intended for use as potable water.

Subp. 20. **Duplicate.** "Duplicate" means replicate.

Subp. 21. **EPA.** "EPA" means the United States Environmental Protection Agency.

Subp. 22. **Fees.** "Fees" means the fees described in Minnesota Statutes, section 144.98, subdivision 3.

Subp. 23. **Field of testing.** "Field of testing" means the combination of analyte, method, matrix, and test category for which a laboratory has applied or received certification by the commissioner.

Subp. 24. **Inspection.** "Inspection" means an on-site evaluation of laboratory facilities, records, personnel, equipment, methodology, and quality assurance practices by the commissioner for compliance with the applicable provisions of this chapter.

Subp. 25. **Internal standard.** "Internal standard" means a pure analyte or analytes added to a test sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes and surrogates that are components of the sample or solution. The analyte or analytes used for the internal standard is not present in the test sample.

Subp. 26. **Laboratory.** "Laboratory" means the state, a person, corporation, or other entity, including a governmental entity, that examines, analyzes, or tests samples.

Subp. 27. **Laboratory control sample or LCS.** "Laboratory control sample" or "LCS" means a sample of a controlled matrix known to be free of the analyte of interest, to which the laboratory has added a known and verified concentration of analyte and that the laboratory has taken through all preparation and analytical steps in the method.

Subp. 28. **Laboratory director.** "Laboratory director" means an agent or affiliate of the laboratory responsible for ensuring compliance with parts 4740.2010 to 4740.2120.

Subp. 29. **Managing agent.** "Managing agent" means a person, as defined in Minnesota Statutes, section 326.71, subdivision 8, who is legally authorized to direct the activities of a laboratory and commit the appropriate resources to comply with parts 4740.2010 to 4740.2120.

Subp. 30. **Matrix or matrices.** "Matrix" or "Matrices" means the predominant material of which the sample to be analyzed is composed. Matrices include but are not limited to air, drinking water, nonpotable water, sewage sludge, and solid and chemical materials.

Subp. 31. **Matrix spike.** "Matrix spike" means a sample prepared by adding a known quantity of analyte and subjecting the sample to the entire analytical procedure to determine the ability to recover the known analyte or compound.

Subp. 32. **Matrix spike duplicate.** "Matrix spike duplicate" means a replicate matrix spike that is prepared and analyzed to determine the precision of the approved test method.

Subp. 33. **Measurement system.** "Measurement system" means any instruments, gauges, tools, devices, equipment, procedures, methods, or aggregates thereof, used to acquire or control sample data generated according to parts 4740.2010 to 4740.2120.

Subp. 34. **Method.** "Method" means the published scientific technique recognized by the commissioner for performing a specific measurement. Methods include instructions for sample preparation and sample analysis.

Subp. 35. **Method blank or blank.** "Method blank" or "blank" means a sample free of the analyte of interest and processed according to the laboratory's standard operating procedures manual according to part 4740.2065.

Subp. 36. **Method detection limit or MDL.** "Method detection limit" or "MDL" means the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix type containing the analyte. Unless specified in the approved test method, the method detection limit is determined using the procedures specified in the applicable permit, program, or rule.

Subp. 37. **NELAC.** "NELAC" means the National Environmental Laboratory Accreditation Conference, which is a voluntary association of state and federal agencies whose purpose is to establish and promote mutually acceptable performance standards for the operation of environmental laboratories.

Subp. 38. **Nonconformance or noncompliance.** "Nonconformance" or "noncompliance" means deficiency of a laboratory to meet any requirement in parts 4740.2010 to 4740.2120.

Subp. 39. [Repealed, 44 SR 371]

Subp. 40. **Owner.** "Owner" means a person who:

A. is a sole proprietor of a laboratory;

B. holds a partnership interest in a laboratory; or

C. owns five percent or more of the shares in a corporation that owns a laboratory.

Subp. 41. **Parameter.** "Parameter" means an analyte.

Subp. 42. **Precision.** "Precision" means the measure of mutual agreement among individual measurements of a sample, usually under prescribed similar conditions, usually expressed as the standards deviation, variance, or range, in either absolute or relative terms.

Subp. 43. **Proficiency testing sample or PT sample.** "Proficiency testing sample" or "PT sample" means a sample obtained from an approved provider to evaluate the ability of a laboratory to produce an analytical test result meeting the definition of acceptable performance. The concentration of the analyte in the sample is unknown to the laboratory at the time of analysis.

Subp. 44. **Quality control.** "Quality control" means the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service so that it meets the needs of users.

Subp. 45. **Quality control data.** "Quality control data" means data generated to assess the accuracy and precision of test data. Quality control data includes data on calibration standards, proficiency testing samples, known standards, duplicate samples, blanks, spiked samples, and limits for quality control spiked samples, reference standards, duplicates, and detection levels.

Subp. 46. **Quality system or quality assurance.** "Quality system" or "quality assurance" means the actions planned and taken that involve activities including control, assessment, reporting, and improvement in a laboratory's processes to ensure that a product or service meets the requirements of parts 4740.2010 to 4740.2120.

Subp. 47. **Quantitate.** "Quantitate" means the arithmetic process of determining the amount of analyte in a sample.

Subp. 48. **Replicate.** "Replicate" means two or more substantially equal aliquots analyzed independently for the same parameter.

Subp. 49. **Reporting limit.** "Reporting limit" means the lowest level of an analyte that can be accurately recovered from the matrix of interest, for example, the level of quantitation.

Subp. 50. **Revocation.** "Revocation" means a determination by the commissioner to invalidate in part or in total a laboratory's certification.

Subp. 51. **Sample or environmental sample.** "Sample" or "environmental sample" means a substance derived from a nonhuman source and collected for the purpose of analysis.

Subp. 52. **Scope of certification.** "Scope of certification" means the sum of all fields of testing for which a laboratory has been granted certification by the commissioner.

Subp. 53. **Second source.** "Second source" means a different vendor or manufacturer, or different lots from the same vendor or manufacturer, usually in reference to standards.

Subp. 54. **Solid.** "Solid" means:

A. soils as defined in Minnesota Statutes, section 103F.401, subdivision 10;

B. sediments as defined in Minnesota Statutes, section 103F.401, subdivision 9;

C. solid waste as defined in Minnesota Statutes, section 115A.03, subdivision 31; and

D. biosolids as defined in Minnesota Statutes, section 115A.03, subdivision 29.

Subp. 55. **Standard.** "Standard" means:

A. the certified reference materials produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method; or

B. the dilutions made from these certified reference materials for the purposes of calibration or determining accuracy of a test method.

Subp. 56. **Successor in interest.** "Successor in interest" means a laboratory that is owned or controlled by a majority of persons owning or controlling a laboratory certified under a previously issued certificate.

Subp. 57. **Surrogate.** "Surrogate" means a compound that is similar to the analytes of interest in chemical composition and behavior in the analytical process, but that is not normally found in environmental samples.

Subp. 58. **Suspension.** "Suspension" means the temporary invalidation in part or in total of a laboratory's certification for a defined period of time according to part 4740.2050, subpart 9, to allow a laboratory time to correct deficiencies or areas of noncompliance to comply with parts 4740.2010 to 4740.2120.

Subp. 59. **Target or target analyte.** "Target" or "target analyte" means an analyte or list of analytes within a test method that may be analyzed and for which the laboratory has obtained certification from the commissioner to test as part of a field of testing.

Subp. 60. **Verification.** "Verification" means confirmation by examination of and provision of objective evidence that specified requirements have been fulfilled. Verification is the process of examining a result of a given activity to determine conformance with parts 4740.2010 to 4740.2120.

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4740.2050 APPLICATION FOR CERTIFICATION.

Subpart 1. **Base certification requirements.**

A. A laboratory may request to be certified by the commissioner for the use of methods to test the analytes eligible for certification.

B. A laboratory must specify the fields of testing for which it seeks certification. No certification shall be awarded for any field of testing without the laboratory meeting base certification requirements. No laboratory may receive base certification without approval of at least one field of testing.

C. A laboratory must apply on a form that is provided by the commissioner.

D. Applications for renewal of certification must be received no later than 90 days before the expiration of certification. The application must meet the criteria of this subpart. If a laboratory fails to submit a renewal application within 90 days before the expiration of certification, the commissioner must notify the regulatory authorities that receive data that the laboratory did not apply to renew its certification. The laboratory must not report results as certified after its certification expires.

Subp. 2. **Requirements for mobile laboratories.**

A. A mobile laboratory is considered a separate laboratory and is subject to all requirements, including application requirements, of parts 4740.2010 to 4740.2120.

B. In addition to the requirements under subpart 1, a mobile laboratory must submit a vehicle identification number, license plate number, or other uniquely identifying information.

C. A mobile laboratory must designate which fields of testing, equipment, and personnel are associated with the mobile laboratory. Changes to the numbers and types of equipment within the mobile laboratory may require reapplication according to subpart 1.

Subp. 3. [Repealed, 44 SR 371]

Subp. 4. **Changes in scope of certification.**

A. The commissioner shall approve a laboratory's application to add a field of testing at any time other than the time of renewal if the laboratory meets the criteria in parts 4740.2010 to 4740.2120 and submits the applicable fees.

B. Requests to add fields of testing for new analytes in response to a notice of availability do not require payment of additional fees if the laboratory holds a certification for that test category and applies for additional analytes within the same test category. Applications for fields of testing for new analytes in response to a notice of availability must meet the requirements of subpart 1 and must be received by the commissioner no later than 180 days after the notice of availability is posted.

C. Requests for the addition of fields of testing received more than 180 days after the notice of availability is posted are subject to fees according to subpart 16.

Subp. 5. **Review of application.** After receiving the application and information required in subpart 1, the commissioner shall:

A. notify the laboratory in writing of any omission or error in the application;

B. deny certification for an initial application or revoke certification for a renewal application if the laboratory does not submit to the commissioner the required information within 15 days after receiving an error notice under item A;

C. award certification according to subpart 7 if the laboratory's application meets the applicable standards of parts 4740.2010 to 4740.2120; or

D. notify the laboratory that its current certification for fields of testing shall be continued until the commissioner fully reviews all documentation for compliance with parts 4740.2010 to 4740.2120.

Subp. 6. **Laboratory inspection.**

A. The commissioner may conduct inspections of certified laboratories or laboratories applying for certification.

B. The commissioner may notify the laboratory prior to arrival at the facility or may conduct an inspection without prior notice at any time during normal business hours to verify compliance with parts 4740.2010 to 4740.2120. When the commissioner provides notification, the notification may be written or oral.

C. When the commissioner determines after inspection that a certified laboratory does not comply with applicable provisions of parts 4740.2010 to 4740.2120, the commissioner shall notify the laboratory of the deficiencies in writing.

D. A laboratory must remedy any deficiencies and provide documentation of the correction to the commissioner. Within 30 days of receiving the report of deficiencies, the laboratory must submit documentation of corrective actions planned and taken. If the laboratory does not provide acceptable documentation of corrective actions or corrective action plans within 30 days, the commissioner shall notify the laboratory that its certification may be suspended in total or in part according to subpart 9. If the laboratory does not provide any documentation of deficiency corrections within 30 days, the commissioner shall notify the laboratory that its certification is revoked in total according to subpart 10.

E. A laboratory may not reapply for certification after suspension or revocation until it has corrected all deficiencies. After all deficiencies are corrected, the laboratory may apply for certification according to subpart 1. With its new application, the laboratory must submit written documentation of the steps taken to correct the deficiencies.

Subp. 7. **Awarding certification.**

A. Documentation of a laboratory's certification must include:

(1) a certificate acknowledging the laboratory's compliance with base certification requirements; and

(2) the scope of certification for the laboratory.

B. If a laboratory's scope of certification changes, the commissioner shall issue a new certificate and scope of certification.

C. A laboratory must return its certificate to the commissioner upon suspension or revocation of certification.

D. A certified laboratory must not misrepresent its certification on any document, including laboratory reports, catalogs, advertising, business solicitations, proposals, quotations, or other materials.

E. A laboratory must make available its current certificate and corresponding scope of certification upon the request of a client, certification authority, or regulatory agency. The laboratory must not supply a copy of its current certificate without the accompanying copy of its scope of certification.

Subp. 8. **Denial.**

A. The commissioner shall deny certification if a laboratory's initial or renewal application does not meet the requirements of subpart 1 or if a laboratory's request for variance does not satisfactorily address all items in subpart 13.

B. A laboratory that has had its request for certification denied may reapply according to subpart 1. The application and all required documentation must be accompanied by repayment of applicable fees.

C. The commissioner shall not refund fees if an application is denied.

Subp. 9. **Suspension.**

A. When the commissioner determines that there are grounds for suspension, the commissioner must notify the laboratory in writing. A laboratory's certification may be suspended in total or in part for a period not to exceed 180 days and not to extend beyond the expiration date of the current certification. If a laboratory takes corrective action before the end of the suspension period, certification for the suspended fields of testing or for the base certification and fields of testing must be restored if the corrective actions satisfactorily address the deficiencies cited in the notice of suspension, except when contrary to an applicable reciprocity agreement. The laboratory shall retain certification for the fields of testing for which it continues to meet the requirements of parts 4740.2010 to 4740.2120.

B. Grounds for suspension of certification are:

(1) failure to produce acceptable results in two consecutive proficiency testing studies for the same field of testing;

(2) failure to use an approved method or to follow the method in sample analysis;

(3) failure to submit an acceptable corrective action report in response to an inspection or unacceptable proficiency testing results;

(4) failure to notify the commissioner of any changes according to subpart 15;

(5) failure of the laboratory to maintain records that demonstrate the capability of laboratory staff as required by part 4740.2099; or

(6) suspension of certification by a certifying authority with which the commissioner has a reciprocity agreement.

C. The effective date of suspension is the date that the laboratory receives the suspension notice from the commissioner. Upon receiving the notice, the laboratory must notify all clients whose samples have been received or analyzed within 30 days prior to the notification or back to the date at which the laboratory was in compliance, whichever is greater. Notification is required for all fields of testing for which the laboratory's certification has been suspended. The notification from the laboratory must be in writing. The laboratory must submit copies of each notification to the commissioner at the time that the notification is sent to the client.

D. A laboratory that has had its certification suspended may reapply according to subpart 1. Repayment of fees is not required for reinstatement if the laboratory corrects the deficiencies within the time frame required by the commissioner, not to exceed 180 days or the expiration date of the current certification, whichever is sooner. If the laboratory fails to correct the causes of suspension within the specified time frame, the commissioner shall revoke in total or in part the laboratory's certification according to subpart 10, item A.

E. A laboratory that has had its certification suspended due to unacceptable proficiency testing results must submit acceptable proficiency testing results for the fields of testing from two successive studies to restore certification.

Subp. 10. **Revocation.**

A. When the commissioner determines that there are grounds for partial or total revocation of a laboratory certification, the commissioner must notify the laboratory in writing. The laboratory shall retain certification for the fields of testing for which it continues to meet the requirements of parts 4740.2010 to 4740.2120.

B. Grounds for partial or total revocation of certification are:

(1) failure to respond to deficiencies according to subpart 6;

(2) failure to correct the deficiencies cited in a notice of suspension within the time frame specified by the commissioner;

(3) failure to implement corrective action related to any deficiencies found during a laboratory inspection;

(4) failure to implement corrective action in response to an unacceptable proficiency testing result;

(5) failure to complete proficiency testing studies and maintain a history of successful proficiency testing studies;

(6) revocation of certification by a certifying authority with which the commissioner has a reciprocity agreement; or

(7) failure to comply with applicable standards of parts 4740.2010 to 4740.2120.

C. Grounds for total revocation of a laboratory's certification are:

(1) failure to respond with a report of corrective actions or corrective action plans for deficiencies identified during an on-site inspection within 30 days of receiving the inspection notice of deficiencies;

(2) submittal of proficiency test sample results generated by another laboratory as its own;

(3) reporting sample results without qualification or notation for fields of testing for which the laboratory's certification has been suspended or for which the laboratory has not requested or received certification;

(4) misrepresentation of any material fact pertinent to receiving and maintaining certification;

(5) denial of entry during normal business hours for an inspection as required under subpart 6, unless circumstances endangering safety or welfare prohibit entry;

(6) failure to send written notification of revocation or suspension to clients within the time frame specified in this subpart;

(7) conviction of charges relating to the falsification of any report relating to a laboratory analysis; or

(8) for laboratories certified through reciprocal agreements, failure to notify the commissioner within 30 days after any enforcement action is taken by the reciprocal certifying authority.

D. The effective date of revocation is the date that the laboratory receives the revocation notice from the commissioner. Upon receiving the notice, the laboratory must notify all clients whose samples have been received or analyzed within 30 days prior to the notification or back to the date at which the laboratory was in compliance, whichever is greater. Notification is required for all fields of testing for which the laboratory's certification has been revoked. The notification from the laboratory must be in writing. The laboratory must submit a copy of each notification to the commissioner at the time that the notification is sent to the client.

E. A laboratory that has had its certification revoked must not advertise itself as certified and, when possible, must remove or replace any advertisements that indicate that the laboratory is certified.

F. A laboratory that has had its certification revoked may not reapply for certification until it has corrected all deficiencies. The laboratory may reapply according to subpart 1 and, with the application, must provide documentation of the steps taken to correct the deficiencies.

Subp. 11. **Successor in interest; recertification.** A successor in interest of a laboratory that has had its certification revoked or suspended may not apply for recertification until the end of the term for which the certification was suspended or until all conditions for reapplication after revocation are met.

Subp. 12. **Reciprocity and laboratories in other states.**

A. A laboratory in another state may request certification in Minnesota. In addition to following the application process under subpart 1, the laboratory must submit the appropriate fees with its application.

B. The commissioner may enter into agreements with certifying authorities of federal agencies and agencies of other states for reciprocal recognition of laboratory certification programs or portions of programs that are substantially equivalent.

C. When a reciprocal agreement exists, the commissioner shall certify an out-of-state laboratory that:

(1) submits an application meeting the requirements of subpart 1;

(2) submits the appropriate fees;

(3) provides a copy of current certification from the reciprocal state or private or federal agency; and

(4) provides a copy of the certifying authority's most recent inspection report.

D. A laboratory certified under this subpart must notify the commissioner within 30 days after any enforcement action is taken by the reciprocal certifying authority.

E. Laboratories certified under reciprocity agreements are subject to parts 4740.2010 to 4740.2120.

F. The commissioner shall provide a list of reciprocity agreements upon request.

Subp. 13. **Request for variance.**

A. The commissioner may grant a variance from parts 4740.2010 to 4740.2120. Variances from the use of an approved method may be granted according to part 4740.2060. To request a variance, a laboratory must pay the appropriate variance fee and must indicate in writing:

(1) the rule part and language for which the variance is sought;

(2) reasons for the request;

(3) alternate measures that will be taken if the request for a variance is granted;

(4) the length of time of the variance; and

(5) data to ensure analytical results of equal or better reliability.

B. The commissioner shall review information submitted with the variance request. If the laboratory proposes alternatives equivalent or superior to those requirements in the rule, shows that strict enforcement of the rule would cause undue hardship, and shows that the variance will not adversely affect the reliability of the data produced by the laboratory, the commissioner shall grant the variance provided the variance does not conflict with statutory provisions. The commissioner shall grant or deny the variance within 60 days after receipt of the request, giving the laboratory written justification for the decision. The commissioner must specify an expiration date for the variances the commissioner issues.

Subp. 14. **Voluntary withdrawal of certification.**

A. If a laboratory chooses to withdraw its application for certification or its current certification in total or in part, the laboratory must notify the commissioner in writing and specify the effective date of withdrawal.

B. The commissioner shall consider that a laboratory has chosen to voluntarily withdraw its certification if the laboratory has not submitted a complete renewal application within 90 days before the expiration date of its current certification. In this situation, the effective date is the expiration date of the laboratory's current certification.

C. By the effective date of the withdrawal of certification, in total or in part, the laboratory must notify current clients and regulatory agencies of its intent to withdraw its certification and must indicate the effective date of the withdrawal. Notification is required for all fields of testing for which the laboratory has chosen to voluntarily withdraw certification. The notification from the laboratory must be in writing. The laboratory must submit a copy of each notification to the commissioner at the time that the notification is sent to the client.

D. The commissioner shall not refund fees if a current certification is voluntarily withdrawn by the laboratory.

Subp. 15. **Duty to notify.**

A. A laboratory must notify the commissioner in writing within 30 days of a change in:

(1) the name of the laboratory;

(2) the physical location, postal mailing address, and electronic mailing address of the laboratory;

(3) the owner of the laboratory;

(4) the names and telephone numbers of a designated contact person and the laboratory director;

(5) the name of at least one managing agent with signature attested by a notarial officer;

(6) the names of supervisory professional staff responsible for the analyses;

(7) major analytical equipment; or

(8) test methods.

B. With the notification, a laboratory must provide results of proficiency testing samples, or a demonstration of capability, analyzed in the new laboratory location or analyzed under the change in laboratory owner, instrumentation, or methods.

Subp. 16. **Payment of fees.**

A. All applications or requests to change the scope of certification submitted to the commissioner for approval must be accompanied by the fee specified in Minnesota Statutes, section 144.98.

B. When a laboratory requests certification for additional fields of testing at any time other than the time of initial or renewal application, the laboratory must submit fees equal to the fees for the test category in which the method or analyte is requested. The fee also applies to the addition of methods or analytes for reinstatement after revocation or denial of certification. No fee shall be assessed for the addition of fields of testing in response to a notice of availability when an application is submitted under the conditions specified in subpart 4.

C. When a laboratory requests a variance according to subpart 13, the request must be accompanied by applicable fees according to Minnesota Statutes, section 144.98.

D. Payment of fees must be in the form of a check, money order, or electronic transfer of funds. When payment is in the form of an electronic transfer of funds, proof of deposit must be verifiable before the date the fees are due to the commissioner.

Subp. 17. **Appeal of administrative decision.**

A. The commissioner shall notify a laboratory in writing of the reasons for a decision to suspend or revoke a certification.

B. A laboratory has 30 days from the date of receiving the decision to appeal the decision. A request to appeal the decision must:

(1) be in writing;

(2) indicate the facts the laboratory disputes;

(3) be signed by the laboratory director; and

(4) be sent to the commissioner.

C. Upon receipt of an appeal request, the commissioner shall initiate the procedure for a contested case hearing according to Minnesota Statutes, chapter 14, and rules of the Office of Administrative Hearings.

Statutory Authority: *MS s 14.05; 14.3895; 144.97; 144.98*

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4740.2060 METHODS REQUIRED FOR CERTIFICATION.

Subpart 1. **Scope.** Laboratories must observe appropriate methodologies for conducting analyses. Methods contain specific instructions on sample collection and preservation procedures. The federal and state methods under subparts 2 to 5 are incorporated by reference, are not subject to frequent change, and are available on the Internet at http://www.gpo.gov or through the Minitex interlibrary loan system.

Subp. 2. **Clean water program.**

A. Methods for the clean water program test category are as provided under Code of Federal Regulations, title 40, part 136.

B. In the absence of an applicable federal regulation, alternative methods may be used for state-specific testing if the state agency administering the permit, program, or rule grants written approval citing the laboratory's name and the title, revision date, and revision number of the procedure receiving approval.

C. The laboratory must submit a copy of the approval for alternate methods to the commissioner along with an application, as required under part 4740.2050.

D. If certification for an alternative method is requested, the laboratory must apply for a variance from this subpart according to part 4740.2050, subpart 13.

Subp. 3. **Safe drinking water program.**

A. Methods for the safe drinking water program test category are as provided under chapter 4720 and Code of Federal Regulations, title 40, parts 141 and 143.

B. In the absence of an applicable federal regulation alternative methods may be used for state-specific testing if the state agency administering the permit, program, or rule grants written approval citing the laboratory's name and the title, revision date, and revision number of the procedure receiving approval.

C. The laboratory must submit a copy of the approval for alternate methods to the commissioner along with an application, as required under part 4740.2050.

D. If certification for an alternative method is requested, the laboratory must apply for a variance from this subpart according to part 4740.2050, subpart 13.

Subp. 4. **Resource conservation recovery program.**

A. Methods for the resource conservation recovery program test category are as provided under Code of Federal Regulations, title 40, part 261, and "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," Publication SW-846, third edition, as updated and published as final, United States Environmental Protection Agency. The test methods are available on the Internet at http://www.epa.gov/epaoswer/hazwaste/test/main.htm.

B. In the absence of an applicable federal regulation, alternative methods may be used for state-specific testing if the state agency administering the permit, program, or rule grants written

approval citing the laboratory's name and the title, revision date, and revision number of the procedure receiving approval.

C. The laboratory must submit a copy of the approval of alternate methods to the commissioner along with an application, as required under part 4740.2050.

D. If certification for an alternative method is requested, the laboratory must apply for a variance from this subpart according to part 4740.2050, subpart 13.

Subp. 5. **Underground storage tank program.**

A. Methods for the underground storage tank program test category are "Modified DRO Method for Determining Diesel Range Organics," Wisconsin Department of Natural Resources, Publication PUBL-SW-141 (September 1995), available on the Internet at http://www.health.state.mn.us/divs/phl/accreditation/statereq.html; "Modified GRO Method for Determining Gasoline Range Organics," Wisconsin Department of Natural Resources, Publication PUBL-SW-140 (September 1995), available on the Internet at http://www.health.state.mn.us/divs/phl/accreditation/statereq.html; and "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," Publication SW-846, United States Environmental Protection Agency, third edition, as updated, available on the Internet at http://www.epa.gov/epaoswer/hazwaste/test/main.htm.

B. In the absence of an applicable federal regulation, alternative methods may be used for state-specific testing if the state agency administering the permit, program, or rule grants written approval citing the laboratory's name and the title, revision date, and revision number of the procedure receiving approval.

C. The laboratory must submit a copy of the approval for alternate methods to the commissioner along with an application, as required under part 4740.2050.

D. If certification for an alternative method is requested, the laboratory must apply for a variance from this subpart according to part 4740.2050, subpart 13.

Subp. 6. **Other required methods.** The analytical methods, sample collection, and preservation procedures used for samples required to be analyzed under a permit, program, or rule administered by a state agency must meet the requirements specified by the permit, program, or rule. The analytical methods, sample collection, and preservation procedures used to analyze samples for programs required by a federal agency must meet the requirements specified in the relevant parts of the Code of Federal Regulations.

Statutory Authority: *MS s 14.05; 14.3895; 144.97; 144.98*

History: *31 SR 446; 44 SR 371*

Published Electronically: *September 16, 2019*

4740.2065 STANDARD OPERATING PROCEDURES.

Subpart 1. **Written procedures required.** A laboratory must possess a written manual of standard operating procedures used by laboratory personnel for the analysis of samples. A laboratory

must prepare written procedures for all laboratory activities including, but not limited to, sample analysis, operation of instrumentation, generation of data, and performance of corrective action.

Subp. 2. **Quality control.** Actual practice must conform to the written procedures. A laboratory must ensure that the applicable requirements in parts 4740.2080 to 4740.2120 are incorporated into each procedure. All quality control measures must be assessed and evaluated on an ongoing basis. Quality control acceptance criteria in the laboratory's quality assurance manual must be used to determine the validity of the data.

Subp. 3. **Manual requirements.** A standard operating procedures manual must contain:

A. a table of contents;

B. a unique identification of the manual, such as a serial number, an identification on each page to ensure that the page is recognized as a part of the manual, and a clear identification of the end of the manual;

C. the laboratory's name. When several separate procedures are included in the manual, the name must appear on each procedure;

D. a revision number; and

E. a date indicating when the revision became effective.

Subp. 4. **Effective dates.** A laboratory must maintain a record of effective dates for all procedures. A copy of the procedure and the record of effective dates must be maintained for the same period of time that records of the data generated by those procedures are required to be maintained.

Subp. 5. **Availability.** A copy of a written procedure must be available to all personnel that engage in that particular activity.

Subp. 6. **Required use.** An analyst must use the laboratory's standard operating procedure beginning on the effective date for all laboratory activities for the analysis of samples for which certification is required.

Subp. 7. **Copy to commissioner.** A laboratory must submit a copy of its laboratory standard operating procedures manual to the commissioner at the time of application and within 30 days after the effective date of the revision. All changes to the standard operating procedures must be documented. The changes must be incorporated into the manual at least annually. All updated standard operating procedures must include the signature of the managing agent upon revision. The revised procedure manual must be forwarded to the commissioner in its entirety no later than 30 days after its effective date of revision.

Subp. 8. [Repealed, 44 SR 371]

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446; 44 SR 371*

Published Electronically: *September 16, 2019*

4740.2070 PROFICIENCY TESTING REQUIREMENTS.

Subpart 1. **Use of approved providers.** A laboratory must obtain proficiency testing samples from an approved provider meeting the requirements under part 4740.2075.

Subp. 2. [Repealed, 44 SR 371]

Subp. 3. [Repealed, 44 SR 371]

Subp. 4. **Laboratory testing of PT study samples.**

A. A laboratory's management and all analysts must ensure that all PT samples are managed, analyzed, reported, and otherwise handled in the same manner as routine samples, including utilizing the same staff, procedures, equipment, facilities, and frequency of analysis as used for routine analysis for that field of testing.

B. When analyzing a PT sample, a laboratory must employ the same calibration, quality control, acceptance criteria, sequence of analytical steps, number of replicates, and other standard operating procedures as used when analyzing routine samples. The laboratory must follow sample preparation steps for the PT sample as instructed by the approved PT provider for which the PT sample was obtained.

Subp. 5. [Repealed, 44 SR 371]

Subp. 6. [Repealed, 44 SR 371]

Subp. 7. **Evaluation of results.** A laboratory may not request from the PT provider a revised report when the revisions to the report are due to any error on the part of the laboratory.

Subp. 8. [Repealed, 44 SR 371]

Subp. 9. **Corrective actions for unacceptable results.** When an approved provider notifies a laboratory that a PT sample result for any reported field of testing is unacceptable, the laboratory must:

A. within 30 days after receiving the notification of unacceptable results from the approved provider, submit written documentation to the commissioner indicating corrective actions planned and taken;

B. within 30 days after receiving the notification of unacceptable results from the approved provider, submit written documentation to the commissioner indicating the laboratory's request to purchase a PT sample from an approved provider; and

C. within 30 days after receiving the results of the PT sample under item B, supply a copy of the results to the commissioner.

Subp. 10. [Repealed, 44 SR 371]

Subp. 11. **Additional samples for compliance.** The commissioner may require certified laboratories to test additional PT samples at any time to determine compliance with parts 4740.2010 to 4740.2120.

Statutory Authority: *MS s 14.05; 14.3895; 144.97; 144.98*

History: *31 SR 446; 44 SR 371*

Published Electronically: *September 16, 2019*

4740.2075 APPROVAL OF PROVIDERS OF PT SAMPLES.

Subpart 1. **Provider availability.** The commissioner shall make available a list of approved PT providers.

Subp. 2. **Criteria for approval.** The commissioner must approve a PT provider if the PT provider:

A. is compliant with the NELAC standards effective July 1, 2004, to June 30, 2005, or a more current revision, provided the revision is in effect, upon the date it becomes effective;

B. defines the scope of each PT study;

C. evaluates results from all proficiency testing studies using the acceptance criteria described in the NELAC standards or those specified by the commissioner;

D. scores each result as either "acceptable," "not acceptable," "no evaluation," or "not reported";

E. provides to participant laboratories reports that include:

(1) the provider name, in the header;

(2) the laboratory name, laboratory address (physical location), and EPA laboratory ID number, in the header, and the name, title, and telephone number of the laboratory point of contact in the header or cover letter;

(3) the study number and study type in the header;

(4) the shipment date and closing date of the study in the header;

(5) the date of any amended report, if applicable, in the header; and

(6) the following report information:

(a) analyte name for each analyte included in the sample;

(b) method description;

(c) laboratory value as reported;

(d) assigned values and acceptance values reported to three significant figures, with the exception of tests requiring reports of presence or absence of the analyte;

(e) the acceptable/not acceptable status;

(f) a "no evaluation" score for reported values containing alpha characters;

(g) an indication of the amended results, for amended reports, including a brief description of the reason for the amendment; and

(h) an indication of the length of the report presented by either "page X of Y" or the total number of pages with each page numbered consecutively;

F. sends reports of results no later than 21 calendar days after the study closing date. If the report and other proficiency testing sample information are available in electronic format, it must be available only to the participant laboratory and the state agencies selected by the laboratory;

G. maintains the overall effectiveness of the provider's quality system to indicate that samples provided for testing are verifiable, homogeneous, and stable;

H. makes available to the commissioner and any participating laboratory, upon request, a complete report of the provider's analytical data and documentation of the provider's quality system, which relates to the assigned values, homogeneity, and stability of a particular proficiency testing study;

I. makes available to the commissioner, upon request, a report listing the total number of participating laboratories and the number of laboratories scoring "not acceptable" for each analyte;

J. supplies reports to the commissioner in an electronic format acceptable to the commissioner; and

K. supplies the laboratory with a PT sample formulated from a lot that has not been previously sent to the laboratory. If the lot has previously been used in a proficiency testing sample or its assigned values sent to any laboratory, the original PT sample tracking ID must be obliterated and the new sample tracking ID must be unique.

Subp. 3. **Obtaining or maintaining approval status.** In order to obtain and maintain the commissioner's approval to supply PT samples for particular fields of testing, providers must establish and maintain a quality system meeting the requirements of this part.

Subp. 4. **Questionable PT samples.** Upon notice from a laboratory and verification by the approved provider that a PT sample did not meet the requirements in this part, the commissioner may:

A. determine that the affected laboratory must analyze another PT sample for that field of testing; or

B. review quality control data produced by the laboratory to determine compliance with parts 4740.2010 to 4740.2120.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446*

Published Electronically: *October 9, 2006*

4740.2080 QUALITY ASSURANCE PRACTICES; ALL TEST CATEGORIES.

Parts 4740.2087, 4740.2089, and 4740.2095 to 4740.2099 apply to all practices related to the analysis of samples for environmental testing from the time of collection to disposal for all fields of testing whenever a requirement is not listed in the approved method or by permit, program, or rule. The requirements of parts 4740.2087, 4740.2089, and 4740.2095 to 4740.2099 must be included in a laboratory's quality assurance manual. If a requirement is included in an approved method or by permit, program, or rule, a laboratory must demonstrate that the requirements therein are met. If it is not clear which requirements are more stringent, the requirements in parts 4740.2010 to 4740.2120 are to be followed.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446*

Published Electronically: *October 9, 2006*

4740.2085 QUALITY ASSURANCE MANUAL.

A. A laboratory must possess and follow a written manual of quality assurance.

B. The manual may include several separate procedures or incorporate documents by reference.

C. The manual or its separate procedures must contain:

(1) identification on each page to ensure that the page is recognized as part of the manual and clear identification of the end of the manual;

(2) the laboratory's name;

(3) a revision number; and

(4) a date indicating when the revision became effective.

D. The manual must be reviewed periodically and updated when necessary. Documentation of the review process must include the scope of the review, identification of the reviewer, and the date the review was completed.

E. At the time of application, a laboratory must submit a copy of the manual, including documents incorporated by reference if these documents are not generally available to the commissioner. Each subsequent revision of the manual or any of its separate procedures must be submitted to the commissioner in its entirety no later than 30 days after the effective date of the revision.

F. Unless a laboratory justifies why an item is not applicable, the manual must incorporate the quality assurance practices described in parts 4740.2087 and 4740.2089, including but not limited to policies and procedures used to:

(1) determine continual compliance with parts 4740.2010 to 4740.2120;

(2) collect and transport samples, including containers and preservatives according to part 4740.2087, subpart 1;

(3) track samples from the time the laboratory receives them through the time the samples are disposed, including chain-of-custody procedures for samples requested to be processed for possible legal action according to parts 4740.2087, subparts 2 and 3; and 4740.2097;

(4) track the purity and acceptability of laboratory standards and reagents, including the laboratory's source of reagent grade water according to part 4740.2089;

(5) maintain functional equipment, including routine maintenance performed and scheduled according to parts 4740.2091, subpart 2; and 4740.2093, subpart 2;

(6) determine data accuracy and precision for each certified method and analyte within each test category, for example, establishing control limits, preparing control charts, and performing calculations, according to the applicable provisions of parts 4740.2100 to 4740.2120;

(7) validate data conversion, transcription, and reporting according to part 4740.2095;

(8) accept or reject samples according to part 4740.2087, subpart 3;

(9) correct unacceptable proficiency testing results according to part 4740.2070, subpart 9, or perform quality control checks according to the applicable provisions of parts 4740.2087 to 4740.2120;

(10) record changes in training and education of laboratory personnel, including on-the-job training relevant to analysis and reporting of results according to part 4740.2099;

(11) subcontract testing; and

(12) address client complaints.

G. A laboratory must routinely evaluate and document the effectiveness of its quality system to ensure that requirements for certification in parts 4740.2010 to 4740.2120 are met.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446; 44 SR 371*

Published Electronically: *September 16, 2019*

4740.2087 SAMPLE HANDLING, RECEIPT, AND ACCEPTANCE.

Subpart 1. **Handling samples.**

A. A laboratory must have procedures for the transportation, receipt, handling, protection, storage, retention, and disposal of samples. The procedures must include provisions necessary to protect the integrity of the sample and to protect the interests of the laboratory and the client.

B. A laboratory must have a system for identifying samples. The sample's identification must be retained throughout the life of the sample in the laboratory. The identification system must be designed and operated so as to ensure that samples cannot be confused physically or when

referred to in laboratory documentation. The identification of samples must accommodate a subdivision of groups of samples and the transfer of samples between laboratories.

C. Upon receipt of samples, the condition, including any abnormalities or departures from specified conditions as described in the laboratory's quality assurance manual, must be recorded. When there is doubt as to the suitability of a sample for environmental testing, when a sample does not conform to the description provided, or when the environmental test required is not specified in sufficient detail, the laboratory must consult the client for further instructions before proceeding and must maintain a written record of the discussion.

D. When an insufficient amount of sample is received, a laboratory may choose to subsample if subsampling would not cause loss of sample integrity. Information concerning the insufficient amount of sample and any decision to subsample must be indicated with the test results.

E. A laboratory must have procedures and appropriate facilities for avoiding deterioration, contamination, loss, or damage to the sample during storage, handling, preparation, and testing.

F. When samples require storage under specified environmental conditions, the conditions must be maintained, monitored, and recorded. When a sample or a portion of a sample is to be held secure, a laboratory must have arrangements for storage and security that protect the condition and integrity of the secured samples or portions concerned.

G. Samples, sample fractions, extracts, leachates, and other products of sample preparation must be kept in storage units, such as cabinets, refrigerators, or freezers, that are separate from the storage units for all standards, reagents, food, and other potentially contaminating sources. Samples must be stored in such a manner to prevent contamination between samples.

Subp. 2. **Sample receipt protocols.** The following items must be verified and the results documented:

A. all samples that require chemical preservation are considered acceptable if the laboratory verifies that the preservation meets the requirements of the approved method. A laboratory must implement procedures for checking chemical preservation before sample preparation or analysis except for methods where postanalysis preservation checks are required to ensure that sample integrity is not compromised. When specified in permit, program, or rule, chemical preservation must be verified upon receipt; and

B. a laboratory must maintain chronological records, either paper-based or electronic, such as a log book or database, to document receipt of all samples, including the number and types of containers received for each field of testing. The records must include:

(1) the client and project name, if applicable;

(2) the date and time of laboratory receipt;

(3) a unique laboratory-assigned identification code;

(4) the signature, initials, or equivalent electronic identification of the person making the entries;

(5) the field identification code, which identifies each container, linked to the laboratory-assigned identification code in the sample receipt log;

(6) the date and time of sample collection, linked to the sample container and to the date and time of receipt in the laboratory;

(7) the requested field of testing, linked to the laboratory-assigned identification code; and

(8) any comments resulting from inspection for sample rejection, linked to the laboratory-assigned identification code.

Subp. 3. **Sample acceptance policy.**

A. A laboratory must have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted or rejected by the laboratory. Data from samples that do not meet the laboratory's criteria must be recorded in an unambiguous manner clearly defining the nature and substance of the deviation from acceptable procedures.

B. A laboratory's sample acceptance policy must be made available to sample collection personnel and must address, at a minimum:

(1) documentation, including sample identification; location, date, and time of collection; collector's name; preservation type; sample type; and any special remarks concerning the sample;

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

(3) use of appropriate sample containers;

(4) adherence to specified holding times;

(5) adequate sample volume to perform the requested tests and relevant quality control determinations; and

(6) procedures to be used when samples show signs of damage, contamination, inadequate preservation, or loss of integrity.

C. If the sample does not meet the sample receipt acceptance criteria listed in the laboratory's quality assurance manual, the laboratory must retain correspondence and records of conversations concerning the final disposition of rejected samples or fully document any decision to proceed with the analysis of samples not meeting acceptance criteria. The report of samples analyzed without meeting the sample acceptance criteria must indicate, at a minimum, the condition of the samples on the chain-of-custody, transmittal form, or the laboratory receipt documents in addition to appropriately qualifying the analysis data on the final report.

Statutory Authority: *MS s 14.05; 14.3895; 144.97; 144.98*

History: *31 SR 446; 44 SR 371*
Published Electronically: *September 16, 2019*

4740.2089 STANDARDS, REAGENTS, AND BACTERIOLOGICAL MEDIA.

A. Reference standards that are used in the laboratory must be obtained, when available, from the National Institute of Standards and Technology (NIST), manufacturers that supply NIST standards or NIST traceable standards, or an international standard-setting organization.

B. A laboratory must retain records for all standards, reagents, and bacteriological media. The records must include:

(1) identification of the manufacturer or vendor;

(2) certificate of analysis or purity, if supplied;

(3) lot number;

(4) date of receipt or preparation;

(5) preparer's initials, if applicable;

(6) method of preparation, when prepared in the laboratory;

(7) recommended storage conditions; and

(8) expiration date after which the material must not be used unless its reliability is verified by the laboratory.

Statutory Authority: *MS s 14.05; 14.3895; 144.97; 144.98*

History: *31 SR 446; 44 SR 371*
Published Electronically: *September 16, 2019*

4740.2091 REQUIREMENTS FOR CALIBRATION OF SUPPORT EQUIPMENT.

Subpart 1. **Scope.** This part applies to all devices that may not be the actual test instrument, but that are necessary to support laboratory operations, if quantitative results are dependent on their accuracy. Such devices include, but are not limited to, balances; ovens; refrigerators; freezers; incubators; water baths; temperature measuring devices, including thermometers and thermistors; thermal/pressure sample preparation devices; autoclaves; and volumetric dispensing devices, such as Eppendorf or automatic diluter/dispensing devices.

Subp. 2. **Requirements.**

A. Equipment must be operated by trained personnel. Up-to-date instructions on the use and maintenance of equipment, including any relevant manuals provided by the manufacturer of the equipment, must be readily available for use by the appropriate laboratory personnel.

B. All equipment must be properly maintained, including inspection, calibration, and cleaning. Maintenance procedures must be documented. Calibration of balances, weights, temperature

recording devices, light sources, and detectors must be appropriate to the required precision and accuracy of the method. Calibrations must be performed at least annually and must be traceable to appropriate standards.

C. Records must be maintained for each major item of equipment, including software. The records must include:

(1) the identity of the item of equipment, including software;

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

(3) documentation that equipment complies with the manufacturer's specification;

(4) the current location within the laboratory;

(5) the manufacturer's instructions, if available;

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

(7) the maintenance plan and maintenance carried out to date, documentation on all routine and nonroutine maintenance activities, and reference material verifications;

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

(9) date received and date placed in service or the date on which its first use or repair was recorded; and

(10) if available, condition when received, such as new, used, or reconditioned.

Subp. 3. **Frequency of calibration.**

A. All support equipment described in subpart 1 must be calibrated or verified at least annually, using National Institute of Standards and Technology (NIST) traceable references when available, over the entire range of use.

B. On each working day, balances, ovens, refrigerators, freezers, and water baths must be checked in the expected use range with NIST traceable references, when available.

C. Mechanical volumetric dispensing devices including burettes, except Class A glassware, must be checked for accuracy at least quarterly. All glassware, including glass microliter syringes used for calibration, must be checked for accuracy and documented before its first use in the laboratory if the glassware does not come with a certificate attesting to established accuracy.

D. For chemical and biological tests using an autoclave, the temperature, cycle time, and pressure of each run must be documented by the use of appropriate chemical indicators, temperature recorders, and pressure gauges.

E. Volumetric equipment must be calibrated as follows:

(1) equipment with movable parts, such as automatic dispensers, dispensers/diluters, and mechanical hand pipettes, must be calibrated quarterly;

(2) equipment such as filter funnels, bottles, non-Class A glassware, and other marked containers must be calibrated once per lot prior to first use; and

(3) the volume of the disposable volumetric equipment such as sample bottles, disposable pipettes, and micropipette tips must be checked once per lot.

F. Dial thermometers must be checked on a quarterly basis. All measurements must be recorded. When the thermometer is used for microbiological methods, all thermometers must be calibrated on an annual basis against a NIST thermometer. When the thermometer is used for nonmicrobiological methods, the thermometer is valid for the time period specified on the vendor's certificate. If a time period is not specified, the thermometer must be calibrated on an annual basis against an NIST thermometer.

Subp. 4. **Acceptance criteria.**

A. The results of calibrations must be within the specifications required of the application for which the equipment is used.

B. The acceptability for use or continued use must be according to the needs of the analysis or application for which the equipment is being used.

C. When the results of calibration of support equipment are not within the required specifications, the laboratory must remove the equipment from service until repaired.

D. Records must be retained to document equipment performance.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446*

Published Electronically: *October 9, 2006*

4740.2093 REQUIREMENTS FOR INSTRUMENT CALIBRATION.

Subpart 1. **Scope.** This part applies to all devices that are the actual test instrument used to quantify the test results.

Subp. 2. **Requirements.**

A. Equipment must be operated by trained personnel. Up-to-date instructions on the use and maintenance of equipment, including any relevant manuals provided by the manufacturer of the equipment, must be readily available for use by the appropriate laboratory personnel.

B. All equipment must be properly maintained, including inspection, calibration, and cleaning. Maintenance procedures must be documented. Calibration of balances, weights, temperature recording devices, light sources, and detectors must be appropriate to the required precision and accuracy of the method. Calibrations must be performed at least annually and must be traceable to appropriate standards.

C. Records must be maintained for each major item of equipment, including software. The records must include:

(1) the identity of the item of equipment, including software;

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

(3) documentation that equipment complies with the manufacturer's specification;

(4) the current location within the laboratory;

(5) the manufacturer's instructions, if available;

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

(7) the maintenance plan and maintenance carried out to date, documentation on all routine and nonroutine maintenance activities, and reference material verifications;

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

(9) date received and date placed in service or the date on which its first use or repair was recorded; and

(10) if available, condition when received, such as new, used, or reconditioned.

Subp. 3. **Initial calibration.**

A. Sufficient records must be retained to permit reconstruction of the instrument calibration, such as calibration date, approved method, instrument, analysis date, each analyte name, the manual or electronic identification of the analyst performing the test, concentration and response, calibration curve or response factor, or unique equation or coefficient used to reduce instrument responses to concentration.

B. Sample results must be quantitated from the most recent instrument calibration and may not be quantitated from any instrument calibration verification unless otherwise allowed by permit, program, or rule.

C. All instrument calibrations must be verified with a standard obtained from a second source. Traceability must be to a national standard, when available.

D. Criteria for the acceptance of an instrument calibration must be established, such as correlation coefficient or relative standard deviation. The criteria used must be appropriate to the calibration technique employed and must be documented in the laboratory's standard operating procedure.

E. If allowed in the permit, program, or rule, results of samples outside of the concentration range established by the calibration must be reported with defined qualifiers, flags, or explanations estimating the quantitative error.

F. The following must occur for methods employing standardization with a zero point and a single point calibration standard:

(1) before the analysis of samples, the linear range of the instrument must be established by analyzing a series of standards, one of which must encompass the single point quantitation level;

(2) a zero point and a single point calibration standard must be analyzed with each analytical batch;

(3) a standard corresponding to the reporting limit must be analyzed with each analytical batch and must meet established acceptance criteria as specified under part 4740.2100, subpart 8;

(4) the linearity must be verified at a frequency established by the method or the manufacturer; and

(5) if a sample within an analytical batch produces results above its associated single point standard, then:

(a) a standard with a concentration at or above the analyte concentration in a sample must be analyzed and must meet established acceptance criteria for validating the linearity;

(b) the sample must be diluted such that the result falls below the single point calibration concentration; or

(c) the data must be reported with an appropriate data qualifier or an explanation in the test report.

G. If the instrument calibration results are outside established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed. If reanalysis of the samples is not possible, data associated with an unacceptable instrument calibration must be appropriately qualified on the test report.

H. Calibration standards must include concentrations at or below the limit specified in the permit, program, or rule.

I. If an approved method does not specify the number of calibration standards, the minimum number is three, one of which must be at the reporting limit, not including blanks or a zero standard, with the exception of instrument technology for which it has been established by methodologies and procedures that a zero and a single point standard are appropriate for calibrations. The laboratory must document in its standard operating procedures how it determines the number of points required for the instrument calibration employed, and the acceptance criteria for calibration.

Subp. 4. **Calibration verification.**

A. When an instrument calibration is not performed on the day of analysis, the instrument calibration must be verified before analysis of samples by analyzing a calibration standard with each batch.

B. If calibration verification is not described in the approved method, a calibration verification must be repeated at the beginning and end of each batch.

C. Sufficient raw data records must be retained to permit reconstruction of the calibration verification, such as test method; instrument; analysis date; each analyte name, concentration, and response; calibration curve or response factor; or unique equations or coefficients used to convert instrument responses into concentrations. Calibration verification records must explicitly connect the verification data to the instrument calibration.

D. Criteria for the acceptance of a calibration verification must be established and evaluated using the same technique used to evaluate the instrument calibration.

E. If the calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then the laboratory must either demonstrate performance after corrective action with two consecutive successful calibration verifications or perform a new instrument calibration. If the laboratory has not demonstrated acceptable performance, sample analyses must not occur until a new instrument calibration is established and verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

(1) when the acceptance criteria for the calibration verification are exceeded high (high bias) and all associated samples contain analytes below the reporting limit, then those sample results may be reported; and

(2) when the acceptance criteria for the calibration verification are exceeded low (low bias), the sample results may be reported if the concentration exceeds a maximum regulatory limit as defined by the permit, program, or rule.

F. When allowed by permit, program, or rule, verification procedures may result in a set of correction factors. If correction factors are employed, the laboratory must have procedures to ensure that copies of all data records, such as in computer software, are correctly updated.

G. Test equipment, including both hardware and software, must be safeguarded from adjustments that would invalidate the test results.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446*

Published Electronically: *October 9, 2006*

4740.2095 REPORTING.

A. Analytical results must be reported accurately, legibly, unambiguously, objectively, and according to any specific instructions in the laboratory's standard operating procedure or quality assurance manual.

B. Laboratories that are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes must have all applicable information specified in item C readily available for review by the state agency administering the permit, program, or rule. Formal reports detailing the information are not required if:

(1) the laboratory is itself responsible for preparing the regulatory reports; or

(2) the laboratory provides information to another individual within the organization for preparation of regulatory reports.

C. The test report must include:

(1) a title, such as "Test Report" or "Laboratory Results";

(2) the name, address, and commissioner-designated identification number of the laboratory;

(3) the telephone number and name of a contact person;

(4) the information in subitem (2) for the subcontracted laboratory and the phrase "This report contains data that were produced by a subcontracted laboratory certified for the fields of testing performed," if data were produced by a laboratory other than the laboratory reporting the results;

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

(6) the name of the client and project name, if applicable;

(7) identification of the approved method used;

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

(9) date and time of sample collection;

(10) the date of receipt of the sample when critical to the validity and application of the results;

(11) time of sample preparation and time of sample analysis when critical to the validity of the sample result;

(12) date of analysis of the environmental test;

(13) the test results with, when appropriate, the units of measurement; whether data are calculated on a dry weight or an "as received" basis; the reporting or detection limit for each sample with appropriate units of measurement; and the counting error for each radiochemistry sample;

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

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

(16) a statement that the report must not be reproduced, except in full, without the written approval of the laboratory;

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

(18) test results that do not meet the requirement, or for which the laboratory is not certified, must be documented with the reason why the result does not meet the requirements and justification as to why the result was reported.

D. When the laboratory analyzes samples by a procedure other than as written, the laboratory record must include:

(1) the sample identification traceable to client;

(2) the modification to the procedure;

(3) the reason for the modification; and

(4) the client's authorization or acknowledgment of the modification.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446*

Published Electronically: *October 9, 2006*

4740.2097 RECORDS RETENTION AND RETRIEVAL.

A. The record-keeping system must allow historical reconstruction of all laboratory activities that produced the analytical data. This also applies to interlaboratory transfers of samples or extracts and the data resulting from the analysis of the samples or extracts.

B. Unless otherwise required by permit, program, or rule, all records must be retained for a minimum of five years after generation of the last entry in the record. All information required for the historical reconstruction of the data must be maintained by the laboratory. If records are retained only in electronic form, the hardware and software required for the retrieval of electronic records must be retained for the same time period as the records to be retrieved.

C. The records must include the identity of personnel designated by the laboratory as responsible for the task performed, as described in the person's job description. The laboratory must retain records of the signatures and initials of designated personnel.

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

E. The record-keeping system must allow the retrieval of all working files and archived records for inspection and verification purposes, including but not limited to systematic naming of electronic files.

F. All records must be signed or initialed by personnel designated by the laboratory as responsible for the task performed. All changes must be clearly indicated in the records. The laboratory must have procedures for recording changes and identifying the personnel making the change.

G. All observations used to calculate the final result must be recorded immediately. If the record is handwritten, the record must be legible and in permanent ink.

H. Entries in records must not be obliterated by methods such as erasures, overwritten files, or markings. All corrections to records on paper must be made by one line marked through the error. The individual making the correction must sign or initial and date the handwritten or electronic correction.

I. A laboratory must maintain a record-keeping system that includes procedures for protecting the integrity and security of the data.

J. A laboratory must supply any documentation or data listed in parts 4740.2010 to 4740.2120 within 30 days of the date that the commissioner requests the information.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446*
Published Electronically: *October 9, 2006*

4740.2099 DOCUMENTATION OF LABORATORY PERSONNEL TRAINING.

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

B. The laboratory must maintain a current table of organization showing relationships between all job classifications and responsible lines of authority associated with the procurement, analysis, reporting, and disposal of samples.

C. The laboratory's managing agents and owners must ensure that all laboratory staff have demonstrated capability in the activities for which they are responsible. Such demonstration must be documented. For new laboratory personnel, the demonstration of capability must be performed prior to their analysis of any sample for that field of testing. Failure to maintain records that demonstrate the capability of laboratory staff as required in this part is grounds for suspension of certification under part 4740.2050, subpart 9. In the absence of method requirements, an analyst must analyze four reagent blanks spiked at the concentration of the calibration check standard. The recoveries must meet the criteria in the laboratory's quality assurance manual.

D. Data produced by analysts while in the process of obtaining required training are acceptable only when reviewed and validated by an analyst or supervisor trained in such evaluations and assessments.

E. The laboratory's managing agents and owners must ensure that laboratory staff maintain capability to perform job functions by:

(1) providing evidence that demonstrates that each employee has read, understood, and is using the approved revision of the laboratory's quality assurance manual;

(2) ensuring that attendance at training courses or workshops for specific equipment, analytical techniques, or laboratory procedures is documented;

(3) maintaining documentation of continued proficiency per analyst by at least one of the following once per year:

(a) acceptable results for a proficiency testing sample or other sample prepared in-house for which the concentrations of analyte are unknown to the analyst at the time of testing;

(b) another demonstration of capability as described in item C;

(c) at least four consecutive laboratory control samples with acceptable levels of precision and accuracy; or

(d) for bacteriological tests, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst;

(4) ensuring that training files contain evidence that laboratory staff have read, understood, and agreed to perform the analysis using the approved revision of the laboratory's procedures; and

(5) providing adequate supervision for all laboratory activities associated with the procurement, analysis, reporting, and disposal of samples for environmental testing from the time of collection to disposal.

F. The laboratory must maintain initials and signatures of anyone analyzing or reviewing data so that the records can be traced back to an individual approving the data.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446*

Published Electronically: *October 9, 2006*

4740.2100 QUALITY CONTROL CRITERIA FOR CHEMISTRY EXCEPT RADIOCHEMISTRY.

Subpart 1. **Scope.** This part applies to laboratories performing testing under the inorganic chemistry, metals, volatile organic compounds, and other organic compounds test categories unless otherwise indicated. All requirements in this part must be incorporated into the laboratory's procedures unless otherwise directed by the approved method. The quality control requirements specified by the laboratory's standard operating procedures manual must be followed. All quality control measures must be assessed and evaluated on an ongoing basis and quality control acceptance criteria must be used to determine the validity of the data.

Subp. 2. **Method blanks.**

A. The method blank must be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure.

B. Each contaminated 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 must be investigated and measures taken to minimize or eliminate the problem. Affected samples must be reprocessed or data must be appropriately qualified if:

(1) the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the test method or by regulation and is greater than one-tenth of the amount measured in any sample; or

(2) the blank contamination otherwise affects the sample results according to test method requirements or the individual project data quality objectives.

C. Procedures must be in place to determine whether a method blank is contaminated. Any affected samples associated with a contaminated method blank must be reprocessed for analysis or the results reported with appropriate data qualifying codes.

D. The method blank must be analyzed at a minimum of one per batch.

Subp. 3. **Laboratory control sample.**

A. A laboratory control sample (LCS) must be used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS must be compared to established criteria and, if found to be outside of established criteria, must indicate that the analytical system is "out of control." Any affected samples associated with an out-of-control LCS must be reprocessed for reanalysis or the results reported with appropriate data qualifying codes.

B. A laboratory control sample must be analyzed at a minimum of one per preparation batch except:

(1) analytes for which no spiking solutions are available; or

(2) in instances for which no separate preparation method is used, such as volatiles in water, the batch must be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

C. All analyte concentrations must be within the calibration range of the instrument calibration. The components to be spiked must be as specified by the permit, program, or rule requirement. In the absence of permit, program, rule, or method requirements, the laboratory must spike as follows:

(1) for those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene, and PCBs, the spike must be chosen that represents the chemistries and elution patterns of the components to be reported; and

(2) the number of analytes selected is dependent on the number of analytes reported. The analytes selected for the spiking solution must be representative of all analytes reported. The following criteria must be used for determining the minimum number of analytes to be spiked:

(a) for methods that include one to ten analytes, spike all components;

(b) for methods that include 11 to 20 analytes, spike at least ten components or 80 percent of the analytes, whichever is greater; and

(c) for methods with more than 20 analytes, spike at least 16 components.

D. The results of the analytes included in the LCS are calculated in percent recovery or measure that allows comparison to established acceptance criteria. The laboratory must document the calculation. The individual LCS is compared to the acceptance criteria as published in the approved method. When there are no established criteria, the laboratory must determine its own criteria and document the method used to establish the limits or utilize client-specified assessment criteria within a permit, program, or rule requirement.

E. A laboratory control sample that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be "out of control" must be considered suspect. The samples must be reprocessed and reanalyzed or the data reported with appropriate data qualifying codes.

Subp. 4. **Matrix spike and matrix spike duplicates.**

A. The frequency of the analysis of matrix spikes and matrix spike duplicates must be determined as part of a systematic planning process or as specified by the required approved method. The matrix spikes must be prepared from samples contained in the batch.

B. For a matrix spike, the components to be spiked must be as specified by the approved method or permit, program, or rule requirement. In the absence of specified spiking components, the laboratory may follow client instructions and then must document its criteria for quality control. In the absence of client instruction, the laboratory must spike as follows:

(1) for those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene, and PCBs, the spike must be chosen that represents the chemistries and elution patterns of the components to be reported; and

(2) the number of analytes selected is dependent on the number of analytes reported. The analytes selected for the spiking solution must be representative of all analytes reported. The following criteria must be used for determining the minimum number of analytes to be spiked:

(a) for methods that include one to ten analytes, spike all components;

(b) for methods that include 11 to 20 analytes, spike at least ten or 80 percent of the analytes, whichever is greater; and

(c) for methods with more than 20 analytes, spike at least 16 components.

C. The results from matrix spikes and matrix spike duplicates must be expressed as percent recovery, relative percent difference, absolute difference, or other measure. Results of matrix spikes and matrix spike duplicates must be compared to the acceptance criteria as published in the approved method. When there are no established criteria, the laboratory must determine its own criteria and document the procedure used to establish the limits or utilize client-specified assessment criteria within a permit, program, or rule requirement.

Subp. 5. **Surrogate spikes.**

A. This subpart applies to the analysis of organic compounds.

B. Except when the matrix precludes their use, or when not available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate test methods.

C. Surrogate compounds must be chosen to represent the various chemistries of the analytes in the method. When specified, the surrogates mandated in the method must be used.

D. The results from surrogate spikes must be expressed as percent recovery. Results of surrogate spikes must be compared to the acceptance criteria as published in the approved method. When there are no established criteria, the laboratory must determine its own criteria and document the method used to establish the limits or utilize client-specified assessment criteria within a permit, program, or rule requirement.

Subp. 6. **Internal standards.**

A. When internal standards are recommended or required by the test method, such as mass spectrometry techniques, a laboratory must add the internal standards to all samples, standards, blanks, and quality control samples before analysis.

B. When specified in the test method, a laboratory must use the internal standards mandated in the test method. If internal standards are not recommended in the method, then the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest and not expected to be found in the samples otherwise.

C. A laboratory must monitor and document the results from analysis of internal standards.

D. Results of internal standards must be compared to the acceptance criteria as published in the approved method. When there are no established criteria, the laboratory must determine its own criteria and document the procedure used to establish the limits or utilize client-specified assessment criteria within a permit, program, or rule requirement.

Subp. 7. **Detection limits.**

A. A laboratory must utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. The detection limit, such as method detection limit (MDL), must be determined by the protocol in the approved method or applicable regulation. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

B. The commissioner shall not require a detection limit study for any component for which spiking solutions or quality control samples are not available.

C. A laboratory must initially determine the detection limit for the compounds of interest in each test method in a matrix in which there are not target analytes or interferences at a concentration that would impact the results or the laboratory must determine the detection limit in the matrix of interest.

D. A laboratory must determine the detection limits each time there is a change in the test method that may affect how the test is performed or when a change in instrumentation occurs that affects the sensitivity of the analysis.

E. A laboratory must include all sample processing steps of the analytical method in the determination of the detection limit.

F. A laboratory must document all procedures used to determine the detection limit, including the matrix type of the sample and all supporting data.

Subp. 8. **Reporting limits.**

A. A laboratory must document all procedures used to determine the reporting limit.

B. A laboratory must establish reporting limits for each field of testing. The reporting limits must be greater than detection limits.

C. A laboratory must verify the reporting limit each time the instrument is calibrated, or monthly at a minimum. The laboratory must analyze a verification standard with a concentration at or below the reporting limit. The percent recovery of the standard must fall within plus or minus 40 percent of the true value.

D. If the percent recovery of the reporting limit verification standard is outside the acceptance criteria, a laboratory must elevate the reporting limit for the associated samples to the concentration of the lowest point, above the zero blank, that meets the acceptance criteria defined in item C. The laboratory must report all samples analyzed after the failed reporting limit check using the elevated reporting limit until a new calibration curve and reporting limit verification standard meet the acceptance criteria.

Subp. 9. **Selectivity.**

A. Absolute retention time and relative retention time aid in identifying components in chromatographic analyses and evaluating the effectiveness of a chromatographic medium to separate constituents. A laboratory must develop and document acceptance criteria for retention time windows if the acceptance criteria are not specified in the approved method.

B. A confirmation must be performed to verify the compound identification when positive results are detected on drinking water. The confirmations must be performed on organic tests, such as pesticides, herbicides, or acid-extractable compounds, or when recommended by the analytical test method, except when the analysis involves the use of a mass spectrometer or Fourier transform infrared spectrometer (FTIR). All confirmations must be documented.

C. A confirmation must be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested. The confirmations must be performed on organic tests, such as pesticides, herbicides, or acid-extractable compounds, or when recommended by the analytical test method, except when the analysis involves the use of a mass spectrometer or Fourier transform infrared spectrometer. A confirmation is not required on positive results for samples analyzed for diesel range organics and gasoline range organics under the underground storage tank program. All confirmations must be documented.

D. A laboratory must document acceptance criteria for mass spectral tuning. The laboratory must ensure that the tuning criteria meets the specifications in the approved method or as established by the client, whichever is more stringent.

Subp. 10. **Manual integrations.** If the integrations are not calculated by the equipment's software, a laboratory must document acceptable use of manual integrations and must have in place a system for review of manual integrations performed to verify adherence to the policies and procedures of the laboratory.

Subp. 11. **Constant and consistent test conditions.**

A. A laboratory must ensure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

B. A laboratory must ensure that glass and plastic containers are cleaned so that they meet the sensitivity of the test method. Any cleaning and storage procedures that are not specified by the test method must be documented in laboratory records and the laboratory standard operating procedures manual.

Statutory Authority: *MS s 14.05; 14.3895; 144.97; 144.98*

History: *31 SR 446; 44 SR 371*

Published Electronically: *September 16, 2019*

4740.2110 QUALITY CONTROL CRITERIA FOR BACTERIOLOGY.

Subpart 1. **Scope.** This part applies to laboratories performing tests under the bacteriological test category unless otherwise indicated. All requirements in this part must be incorporated into the laboratory's procedures unless otherwise directed by the approved method. The quality control requirements specified by the laboratory's standard operating procedures manual must be followed. All quality control measures must be assessed and evaluated on an ongoing basis and quality control acceptance criteria must be used to determine the validity of the data.

Subp. 2. **Sterility checks and blanks.**

A. A blank must be analyzed for each lot of preprepared, ready-to-use media, including chromofluorogenic reagent, and for each lot of media prepared in the laboratory. The analysis must be done before first use of each lot of media.

B. For filtration technique, a laboratory must conduct one beginning and one ending sterility check for each laboratory-sterilized filtration unit used in a filtration series. The filtration series

may include single or multiple filtration units that have been sterilized before beginning the series. For presterilized single-use funnels purchased, a sterility check must be performed on one funnel per lot. The filtration series is considered ended when more than 30 minutes elapse between successive filtrations. During a filtration series, filter funnels must be rinsed with three 20 to 30 milliliter portions of sterile rinse water after each sample filtration. In addition, laboratories must insert a sterility blank after every ten samples per filtration unit or sanitize filtration units by ultraviolet light after each sample filtration.

C. For pour-plate technique, sterility blanks of the media must be made by pouring, at a minimum, one uninoculated plate for each lot of preprepared, ready-to-use media and one for each lot of media prepared in the laboratory.

D. Sterility checks on sample containers must be performed on at least one container for each lot of purchased, presterilized containers. For containers sterilized in the laboratory, a sterility check must be performed on one container per sterilized batch using nonselective growth media.

E. A sterility check must be performed on each batch of dilution water prepared in the laboratory and on each batch of preprepared, ready-to-use dilution water using nonselective growth media.

F. At least one filter from each new lot of membrane filters must be checked for sterility using nonselective growth media.

Subp. 3. **Positive controls.** Each preprepared, ready-to-use lot of media, including chromofluorogenic reagent, and each lot of media prepared in the laboratory must be tested with at least one pure culture of a microorganism known to elicit a positive reaction. This must be done before first use of each lot of media.

Subp. 4. **Negative controls.** Each preprepared, ready-to-use lot of selective media, including chromofluorogenic reagent, and each lot of selective media prepared in the laboratory must be analyzed with one or more known negative culture controls, that is, nontarget microorganisms that should not grow on the test media, as appropriate to the method. This must be done before first use of each lot of media.

Subp. 5. **Test variability.** For test methods that specify colony counts, such as methods using membrane filters or plated media, duplicate counts must be performed monthly on at least one positive sample for each month that the test is performed. With respect to this test for variability, if the laboratory has two or more analysts, each analyst must count typical colonies on the same plate and counts must be within ten percent difference between analysts to be acceptable. In a laboratory with only one microbiology analyst, the same plate must be counted twice by the analyst, with no more than five percent difference between the counts.

Subp. 6. **Method evaluation.** A laboratory must demonstrate proficiency with the test method before first use, by comparison to a method already approved for use in the laboratory, by analyzing a minimum of ten spiked samples whose matrix is representative of those normally submitted to the laboratory, or by analyzing and passing one proficiency test series provided by an approved

proficiency sample provider. The laboratory must maintain documentation of the proficiency demonstration as long as the method is in use and for at least five years after the date of last use.

Subp. 7. **Test performance.** To ensure that analytical results are accurate, a laboratory must confirm a target organism specified in the method.

Subp. 8. **Quality of standards, reagents, and media.**

A. Culture media may be prepared from commercial dehydrated powders or may be purchased ready to use, unless otherwise indicated in the approved method. Media may be prepared by the laboratory from basic ingredients when commercial media are not available or when it can be demonstrated that commercial media do not provide adequate results. Media prepared by the laboratory from basic ingredients must be tested for performance, such as for selectivity, sensitivity, sterility, growth promotion, and growth inhibition, before first use. Detailed testing criteria information must be defined in the laboratory's standard operating procedures manual or quality assurance manual.

B. Reagents, commercial dehydrated powders, and media must be used within the shelf life of the product. The specifications of the reagent, powder, or media must be documented according to the laboratory's quality assurance manual.

C. Distilled water, deionized water, or reverse-osmosis produced water that is free from bactericidal and inhibitory substances must be used in the preparation of media, solutions, and buffers. The quality of the water must be monitored for chlorine residual, specific conductance, and heterotrophic bacteria plate count monthly, when in use; when maintenance is performed on the water treatment system; or at startup after a period of disuse longer than one month. Analysis for metals and the bacteriological water quality test, to determine the presence of toxic agents or growth promoting substances, must be performed annually. Results of these analyses must meet the specifications of the required method and records of analyses must be maintained for five years. Laboratories that can supply documentation to show that their water source meets the criteria, as specified by the method, for ASTM or NCCL Type I or Type II reagent water and is free of bacteria that can grow under these test conditions are exempt from performing the bacteriological water quality test.

D. Media, solutions, and reagents must 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 must include the date of preparation, preparer's initials, type and amount of media prepared, manufacturer and lot number, final pH of the media, and expiration date.

E. Documentation for media purchased preprepared and ready-to-use must include the manufacturer, lot number, type and amount of media received, date of receipt, expiration date of the media, and the verification pH of the liquid.

Subp. 9. **Selectivity.**

A. To ensure identity and traceability, reference cultures used for positive and negative controls must be obtained from a recognized national collection or organization.

B. Microorganisms may be single-use preparations or cultures maintained by documented procedures that demonstrate the continued purity and viability of the organism.

C. Reference cultures may be revived, if freeze-dried, or transferred from slants and subcultured once to provide reference stocks. The reference stocks must be preserved by a technique that maintains the characteristics of the strains. Reference stocks must be used to prepare working stocks for routine work. If reference stocks have been thawed, they must not be refrozen and reused.

D. Working stocks must not be cultured sequentially more than five times and must not be subcultured to replace reference stocks.

Subp. 10. **Temperature measuring devices.** Temperature measuring devices such as liquid-in-glass thermometers, thermocouples, and platinum resistance thermometers used in incubators, autoclaves, and other equipment must be of the appropriate quality to meet specifications in the test method. The gradation of the temperature measuring devices must be appropriate for the required accuracy of measurement and the devices must be calibrated to national or international standards for temperature. All measurements must be recorded.

Subp. 11. **Autoclaves.**

A. The performance of each autoclave must be evaluated initially by establishing its functional properties and performance, for example heat distribution characteristics with respect to typical uses. Autoclaves must meet specified temperature tolerances. Pressure cookers must not be used for sterilization of growth media.

B. Demonstration of sterilization temperature must be provided by use of a continuous temperature recording device or by use of a maximum registering thermometer with every cycle. Appropriate biological indicators must be used once per month to determine effective sterilization. Temperature-sensitive tape must be used with the contents of each autoclave run to indicate that the autoclave contents have been processed.

C. Records of autoclave operations must be maintained for every cycle. Records must include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time, which may be recorded as time in and time out, and operator's initials.

D. Autoclave maintenance, either internally or by service contract, must be performed annually and must include a pressure check and calibration of the temperature device. Records of the maintenance must be maintained in equipment logs.

E. The autoclave's mechanical timing device must be checked quarterly against a stopwatch and the actual time elapsed must be documented.

Subp. 12. **Ultraviolet instruments.** Ultraviolet (UV) instruments used for sanitization must be tested quarterly for effectiveness with an appropriate UV light meter or by plate counts on agar spread plates. Bulbs must be replaced if output is less than 70 percent of original for light tests or if count reduction is less than 99 percent for a plate containing 200 to 300 organisms.

Subp. 13. **Incubators, water baths, ovens.**

A. The stability and uniformity of temperature distribution and the time required after test sample addition to reestablish equilibrium conditions in incubators and water baths must be documented. Temperature of incubators and water baths must be documented twice daily, at least four hours apart, on each day of use.

B. Ovens used for sterilization must be checked for sterilization effectiveness monthly with appropriate biological indicators. Records must be maintained for each cycle that include the date, cycle time, temperature, contents, and analyst's initials.

Subp. 14. **Procedure for washing labware.**

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

B. Glassware must be made of borosilicate or other noncorrosive material, free of chips and cracks, and have readable measurement marks.

C. Labware that is washed and reused must be tested for possible presence of residues that may inhibit or promote growth of microorganisms by performing the inhibitory residue test annually and each time the laboratory changes the lot of detergent or washing procedures.

D. Washed labware must 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 must be maintained.

Statutory Authority: *MS s 144.97; 144.98*

History: *31 SR 446*

Published Electronically: *September 26, 2013*

4740.2120 QUALITY CONTROL CRITERIA FOR RADIOCHEMISTRY.

Subpart 1. **Scope.** This part applies to laboratories performing radiochemistry testing on environmental samples. All requirements in this part must be incorporated into the laboratory's standard operating procedures unless otherwise directed by the approved method. The quality control requirements specified by the laboratory's standard operating procedures manual must be followed. All quality control measures must be assessed and evaluated on an ongoing basis and quality control acceptance criteria must be used to determine the validity of the data.

Subp. 2. **Method blanks.**

A. A laboratory must analyze at least one method blank per batch. The method blank result must be evaluated according to the acceptance criteria in the laboratory's standard operating procedures manual.

B. When the method blank acceptance criteria are not met, a laboratory must take corrective action. The occurrence of a failed method blank and the actions taken must be noted in the laboratory report.

C. In the case of gamma spectrometry where the sample matrix is simply aliquoted into a calibrated counting geometry, the method blank must 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 the sample matrix.

D. A laboratory must not subtract results of method blank analysis from the sample results in the associated batch unless permitted by the approved method. This does not preclude the application of any correction factor, such as instrument background, analyte presence in tracer, reagent impurities, peak overlap, or calibration blank, to all analyzed samples, both program- or project-submitted and internal quality control samples. However, the correction factors must not depend on the required method blank result in the associated analytical batch.

E. The method blank sample must be prepared with similar aliquot size to that of the routine samples for analysis whenever possible.

Subp. 3. **Laboratory control sample.**

A. Laboratory control samples must be performed at a frequency of one per batch. The results of the analysis must be one of the quality control measures to be used to assess the batch. The laboratory control sample result must be assessed against the specific acceptance criteria specified in the laboratory standard operating procedures manual. When the specified laboratory control sample acceptance criteria are not met, the specified corrective action and contingencies must be followed. The occurrence of a failed laboratory control sample acceptance criterion and the actions taken must be noted in the laboratory report.

B. The activity of the laboratory control sample must:

(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 ten times the detection limit.

C. The laboratory standards used to prepare the laboratory control sample must be from a source independent of the laboratory standards used for instrument calibration, if available.

D. The matrix spike must be prepared by adding a known activity of target analyte. When a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope, such as plutonium, Pu 238 and Pu 239, using alpha spectrometry, only one of the analyte isotopes need be included in the laboratory control sample. When more than one analyte isotope is added to the laboratory control sample, each isotope must be assessed against the specified acceptance criteria.

Subp. 4. **Matrix spikes.**

A. Matrix spikes must be performed at a frequency of one per batch for those methods that do not utilize an internal standard or carrier for which there is a chemical separation process and when there is sufficient sample to do so. The exceptions are gross alpha, gross beta, and tritium, which require matrix spikes for aqueous samples. The results of the analysis must be one of the

quality control measures to be used to assess the sample results acceptance. The matrix spike result must be assessed against the specific acceptance criteria specified in the laboratory standard operating procedures manual. When the specified matrix spike acceptance criterion is not met, the corrective actions specified in the laboratory's standard operating procedures must be followed. The occurrence of a failed matrix spike acceptance criterion and the actions taken must be noted in the laboratory report. The lack of sufficient sample aliquot size to perform a matrix spike must be noted in the laboratory report.

B. The activity of the analytes in the matrix spike must be greater than ten times the detection limit.

C. The laboratory standards used to prepare the matrix spike must be from a source independent of the laboratory standards used for instrument calibration, if available.

D. The matrix spike must be prepared by adding a known activity of target analyte. When a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope, such as plutonium, Pu 238 and Pu 239, using alpha spectrometry, only one of the analyte isotopes need be included in the matrix spike sample. When more than one analyte isotope is added to the matrix spike, each isotope must be assessed against the specified acceptance criteria.

E. When gamma spectrometry is used to identify and quantitate more than one analyte isotope, the laboratory control sample and matrix spike must contain isotopes that represent the low (americium-241), medium (cesium-137), and high (cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples, the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

F. The matrix spike sample must be prepared with similar aliquot size to that of the routine samples of analyses.

Subp. 5. **Tracer.** For those approved methods that allow or require the use of a tracer, that is, internal standard, each sample result must have an associated tracer recovery calculated and reported. The tracer recovery for each sample result must be one of the quality control measures used to assess the associated sample result acceptance. The tracer recovery must be assessed against the specific acceptance criteria specified in the laboratory standard operating procedures manual. When the specified tracer recovery acceptance criteria are not met, corrective actions specified in the laboratory's standard operating procedures must be followed. The occurrence of a failed tracer recovery and the corrective actions taken must be noted in the laboratory report.

Subp. 6. **Carrier.** For those approved methods that allow or require the use of a carrier, each sample must have an associated carrier recovery calculated and reported. The carrier recovery for each sample must be one of the quality control measures used to assess the associated sample result acceptance. The carrier recovery must be assessed against the specific acceptance criteria specified in the laboratory standard operating procedures manual. When the specified carrier recovery acceptance criteria are not met, the corrective actions specified in the laboratory's quality assurance manual must be followed. The occurrence of failed carrier recovery acceptance criteria and the actions taken must be noted in the laboratory report.

Subp. 7. **Analytical variability; reproducibility for radiochemistry testing.**

A. A laboratory must analyze replicate samples at least once per batch when there is sufficient sample to do so. The results of the analysis must be one of the quality control measures used to assess sample results acceptance. The replicate result must be assessed against the specific acceptance criteria specified in the laboratory's standard operating procedures manual.

B. When the specified replicate acceptance criteria are not met, the corrective actions specified in the laboratory's standard operating procedures manual must be followed. The occurrence of failed replicate acceptance criteria and the actions taken must be noted in the laboratory test results.

C. If sample concentrations are expected to contain analytes of interest below three times the detection limit, a laboratory may substitute replicate laboratory control samples or replicate matrix spiked samples for replicate samples in item A. The replicate result must be assessed against the specific acceptance criteria specified in the laboratory's standard operating procedures manual. When the specified replicate acceptance criteria are not met, the corrective actions specified in the laboratory's standard operating procedures manual must be followed. The occurrence of failed replicate acceptance criteria and the actions taken must be noted in the laboratory test results.

Subp. 8. **Instrument calibration.**

A. Radiochemistry analytical instruments must be calibrated prior to first use in sample analysis.

B. Calibration must be verified when:

(1) the instrument is serviced;

(2) the instrument is moved; and

(3) the instrument settings have been changed.

C. The standards used for calibration must have the same general characteristics, that is, geometry, homogeneity, and density, as the associated samples.

D. The calibration must be described in the laboratory's standard operating procedures manual.

Subp. 9. **Continuing calibration verification.**

A. Calibration verification checks must be performed using appropriate check standards and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the calibration has not changed.

B. The same check standards used in the preparation of the tolerance chart or control chart at the time of calibration must be used in the calibration verification of the instrument.

C. The check standards must provide adequate counting statistics for a relatively short count time. The sources must be sealed or encapsulated to prevent leakage and contamination of the instrument and laboratory personnel.

D. For alpha and gamma spectroscopy systems, the instrument calibration verification must include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.

E. For gamma spectroscopy systems, the calibration verification checks for efficiency and energy must be performed at least weekly along with performance checks on peak resolution.

F. For alpha spectroscopy systems, the calibration verification check for energy must be performed at least weekly and the performance check for counting efficiency must be performed at least monthly for each day the instrument is used for sample analysis.

G. For gas-proportional and scintillation counters, the calibration verification check for counting efficiency must be performed each day of use.

Subp. 10. **Background radiation measurement.**

A. Background radiation measurements must be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality objectives.

B. Background radiation measurement values must be subtracted from the total measured activity in the determination of the sample activity.

C. For gamma spectroscopy systems, background radiation measurements must be performed at least monthly.

D. For alpha spectroscopy systems, background radiation measurements must be performed at least monthly.

E. For gas-proportional counters, background radiation measurements must be performed at least weekly.

F. For scintillation counters, background radiation measurements must be performed each day of use.

Subp. 11. **Instrument contamination monitoring.** A laboratory must have a written procedure for monitoring radiation measurement instrumentation for radioactive contamination. The procedure must indicate the frequency of the monitoring and must indicate criteria that initiate corrective action.

Subp. 12. **Detection limits.**

A. Detection limits must be determined before sample analysis and must be redetermined each time there is a significant change in the test method or instrument type.

B. The procedures employed must be documented and consistent with published references.

Subp. 13. **Quality of standards and reagents.**

A. The quality assurance manual must describe the procurement, use, and storage of radioisotope standards.

B. Reference standards that are used in a radiochemical laboratory must be obtained from the National Institute of Standards and Technology (NIST), EPA, suppliers of NIST standards or NIST traceable radioisotopes, or suppliers located outside of the United States. Reference standards must be traceable back to the appropriate country's national standards laboratory.

C. Reference standards must be accompanied with a certificate of calibration that describes traceability to NIST or another country's national standards laboratory, when appropriate.

D. Laboratories must consult with the supplier if the laboratory's assessment of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory must not use a value other than the decay-corrected certified value.

E. All reagents used must be analytical reagent grade or better.

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