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**TITLE 252. DEPARTMENT OF ENVIRONMENTAL QUALITY
CHAPTER 301. LABORATORY ACCREDITATION**

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SUBCHAPTER 1. GENERAL PROVISIONS

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252:301-1-1. Purpose, basis, authority, applicability

The rules in this Chapter provide standards for accreditation of privately and publicly owned laboratories for performance of analyses of water and wastewater. This Chapter was promulgated and adopted pursuant to the Oklahoma Environmental Quality Code (Code), 27A O.S. § 2-4-101 *et seq.*, and shall apply to all laboratories certified or applying to be accredited by the Department of Environmental Quality.

252:301-1-2. Accreditation exception

Operational testing analyses for municipal wastewater treatment systems and water supply systems may be submitted to the DEQ by an unaccredited laboratory if, at the time of the analyses, the laboratory was operated by an individual certified by the DEQ as a laboratory operator and the certified laboratory operator approves and signs the analyses report. For further explanation, refer to and comply with the following rules:

- (1) Oklahoma Pollutant Discharge Elimination System Standards (OPDES), OAC 252:606-11-2;
- (2) Public Water Supply Operations, OAC 252:631-3-2; and
- (3) Waterworks and Wastewater Works Operator Certification OAC 252:710-5-53.

252:301-1-3. Definitions

In addition to the definitions contained in the Environmental Quality Code (27A O.S. § 2-1-101 *et seq.*) and OAC 252:4 (Department of Environmental Quality Rules of Practice and Procedure), the following words or terms, when used in this Chapter, shall have the following meaning, unless the context clearly indicates otherwise.

"Acceptable results", as defined in 27A O.S. § 2-4-101, means *a result within limits determined on the basis of statistical procedures as prescribed by the Department.*

"Accreditation" means the process by which the DEQ recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

"Analyte" means the characteristics of a laboratory sample determined by an analytical laboratory testing procedure and is synonymous with "parameter". For purposes of this Chapter, "analyte" also means one of a set of inorganic or organic chemical, physical, radiochemical or microbiological properties whose value determines the characteristics of a water or wastewater sample.

"Applicant" means the owner of a laboratory, or a representative authorized by the owner to act on the owner's behalf, seeking accreditation from the DEQ.

"Applicant laboratory" means the laboratory and its owner or authorized representative for

which an application for accreditation has been filed with the DEQ.

"Approved method" means an analytical test method which has been required by law or is recognized by the DEQ as acceptable for a specific usage.

"Blind audit" means a process whereby the DEQ or any other designated agent submits proficiency testing samples to an accredited laboratory in a manner such that the laboratory is not aware of the process.

"Certificate" is defined in 27A O.S. § 2-4-101 and means the same as laboratory accreditation and includes primary accreditation and reciprocity accreditation.

"Corrective Action Plan" is a written plan of action, including a schedule for implementation, to correct deficiencies identified in the DEQ or DEQ-approved agent's inspection report, including a timeline for implementation; or to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent its recurrence.

"Critical Finding" means a conclusion of noncompliance that would require an immediate corrective action or an immediate stop to testing.

"DEQ" means the Oklahoma Department of Environmental Quality. For purposes of certifications issued and enforcement matters arising prior to July 1, 1993, "DEQ" also means predecessor agencies of the DEQ which had jurisdiction over environmental water quality laboratories on June 30, 1993.

"Evaluation", as defined in 27A O.S. § 2-4-101, *means a review of the quality control and quality assurance procedures, recordkeeping, reporting procedures, methodology, personal qualifications, equipment, facilities and analytical technique of a laboratory for measuring or establishing specific parameters.*

"Finding" means a conclusion of noncompliance of the evaluation process supported by objective evidence.

"Initial accreditation" means a first-time accreditation granted to a laboratory not previously accredited by the DEQ.

"Interim accreditation" means an out-of-time accreditation issued to a DEQ accredited laboratory in analytes for which the laboratory is not currently accredited by the DEQ.

"Laboratory", as defined in 27A O.S. § 2-4-101, *means a facility that performs analyses to determine the chemical, physical or biological properties of air, water, solid waste, hazardous waste, wastewater or soil or subsoil materials or performs any other analyses related to environmental quality evaluations.* "Laboratory" includes mobile laboratories.

"Laboratory waste" means by-products of the analytical process, residues of samples analyzed, discarded reagents or standards and any materials contaminated by any of these.

"Mobile laboratory" means a mobile facility that performs analyses in a self-contained environment with professional analytical instrumentation, excluding field testing of those analytes that require immediate measurement on site (conductivity, residual chlorine, pH, dissolved oxygen, temperature).

"Owner" means the sole proprietor of an individually owned laboratory, the controlling or managing partner of a laboratory held by a partnership, the major stockholders of a corporate owned laboratory, or a municipality or other local government entity which owns or operates a laboratory.

"Parameter" is defined in 27A O.S. § 2-4-101 and is synonymous with "analyte".

"Proficiency testing (PT) sample" means a sample submitted to a laboratory by the DEQ or other designated agent for the purpose of assessing the ability of the laboratory to correctly analyze samples using an approved method.

"Program" means the DEQ laboratory accreditation program.

"QA Plan" or "Quality Assurance Plan" means a written description of quality assurance

activities (quality control) that will ensure the generation of data that are scientifically valid, defensible and of known and acceptable limits of precision and accuracy.

"SOP manual" or "Standard Operating Procedure manual" means a document approved by a laboratory director that includes approved methods, equipment and instruments used by the laboratory for analyses.

252:301-1-4. Terms

Terms used in this Chapter shall have the meanings given to them in OAC 252:301-1-2 or the Oklahoma Environmental Quality Code. Any technical term not defined thereby shall be defined by its generally accepted scientific meaning or its standard dictionary meaning.

252:301-1-5. Accreditation matrices and types

(a) **Matrices.** Laboratories may be accredited in Drinking Water, General Water Quality, and/or Petroleum Hydrocarbons.

(b) **Types of accreditation.** An applicant laboratory may apply at any time for initial, interim or renewal accreditation. A laboratory applying for interim accreditation shall meet the same requirements as a laboratory applying for initial accreditation.

252:301-1-6. Drinking water laboratory

A drinking water laboratory may be accredited in the following category groups: metals, general chemistry, microbiology, asbestos, non-volatile synthetic organic chemicals (SOCs), volatile organic compounds (VOCs) and/or radionuclides.

252:301-1-7. General water quality laboratory

(a) **Category groups.** A general water quality laboratory may be accredited in the following category groups: metals, nutrients, demands, extractable organics, general chemistry I and/or II, microbiology, pesticides - herbicides - PCBs, purgeable organics, radiological, bioassay, hazardous waste characterization, petroleum hydrocarbons, perchlorate, and/or basic environmental laboratory.

(b) **Basic environmental laboratory analytes.** Basic environmental laboratory analytes include: temperature, five day biochemical oxygen demand, carbonaceous biochemical oxygen demand, chemical oxygen demand, total organic carbon (TOC), total Kjeldahl nitrogen (TKN), nitrate-nitrite nitrogen, organic nitrogen, ammonia nitrogen, total dissolved solids (filterable residue), total suspended solids (non-filterable residue), volatile residue, total phosphorous, orthophosphate phosphorus (reactive phosphorus), chloride, fluoride, oil and grease, sulfate, pH, specific conductance, dissolved oxygen, turbidity, total residual chlorine, hardness, alkalinity, color, fecal coliform, Escherichia coli, total coliform, cyanide, phenolics, copper, zinc, iron, sulfide, chromium, and hexavalent chromium.

252:301-1-8. Petroleum hydrocarbon laboratory

A petroleum hydrocarbon laboratory may be accredited in the following category groups: Total Petroleum Hydrocarbons (TPH), Benzene, Toluene, Ethylbenzene, and Xylene (BTEX), Flash Point, and MTBE

252:301-1-9. Fees

(a) **Applicable fees.** The following fees apply:

- (1) Initial accreditation \$1,140.00
- (2) Interim accreditation 671.00

- (3) Renewal fee 34.00
- (4) Renewal late fee 335.00
- (5) Accreditation amendment 67.00
- (6) Fee for 1 category 470.00
- (7) Fee for 2 categories 940.00
- (8) Fee for 3 categories 1,410.00
- (9) Fee for 4 categories 1,880.00
- (10) Fee for 5 or more categories 2,350.00
- (11) On-site evaluation Reimbursable Expense

(b) **Renewal.** Fees to renew accreditation consist of the renewal application fee and the applicable category fee.

(c) **Public water supply system fee exemption.** There is no laboratory accreditation fee for public water supply systems that pay the minimum annual public water supply regulatory service rate fee in accordance with 27A O.S. § 2-6-306.

(d) **Annual fee adjustment.** To assist in meeting rising costs to the DEQ of the environmental services and regulatory programs associated with the laboratory accreditation program, the fees set out in this Section shall be automatically adjusted on July 1st every year to correspond to the percentage, if any, by which the Consumer Price Index (CPI) for the most recent calendar year exceeds the CPI for the previous calendar year. The DEQ may round the adjusted fees up to the nearest dollar. The DEQ may waive collection of an automatic increase in a given year if it determines other revenues, including appropriated state general revenue funds, have increased sufficiently to make the funds generated by the automatic adjustment unnecessary in that year. A waiver does not affect future automatic adjustments.

(1) Any automatic fee adjustment under this subsection may be averted or eliminated, or the adjustment percentage may be modified, by rule promulgated pursuant to the Oklahoma Administrative Procedures Act. The rulemaking process may be initiated in any manner provided by law, including a petition for rulemaking pursuant to 75 O.S. § 305 and OAC 252:4-5-3 by any person affected by the automatic fee adjustment.

(2) If the United States Department of Labor ceases to publish the CPI or revises the methodology or base years, no further automatic fee adjustments shall occur until a new automatic fee adjustment rule is promulgated pursuant to the Oklahoma Administrative Procedures Act.

(3) For purposes of this subsection, "Consumer Price Index" or "CPI" means the Consumer Price Index - All Urban Consumers (U.S. All Items, Current Series, 1982-1984=100, CUUR0000SA0) published by the United States Department of Labor. The CPI for a calendar year is the figure denoted by the Department of Labor as the "Annual" index figure for that calendar year.

(e) An On-site evaluation fee shall be calculated at actual cost, not to exceed \$10,000 per individual laboratory, and includes but is not limited to the following: assessor(s) time and labor (preliminary document review, total travel, time-on-site, report preparation, and corrective action review), transportation, per diem (if required), as described in 252:301-5-4. The on-site evaluation will be invoiced at the closing of the evaluation.

SUBCHAPTER 3. LABORATORY ACCREDITATION PROCESS

PART 1. APPLICATION

Section

- 252:301-3-1. Application required
- 252:301-3-2. Contact information
- 252:301-3-3. Operational information
- 252:301-3-4. Renewals

PART 3. CONDITIONS OF ACCREDITATION

- 252:301-3-31. Conditions applicable to all accreditations
- 252:301-3-32. Amendments to accreditations
- 252:301-3-33. Self-reporting

PART 5. GROUNDS TO REVOKE

- 252:301-3-51. Grounds to take enforcement action
- 252:301-3-52. Notice
- 252:301-3-53. Individual proceedings

PART 1. APPLICATION

252:301-3-1. Application required

(a) **General.** A laboratory shall submit one copy of an application for accreditation to the DEQ along with relevant fees. The application shall be typed on forms provided by the DEQ and shall follow the general format designated by the DEQ.

(b) **Signature and verification.** An application shall be signed by the sole proprietor of an individually owned laboratory, the controlling or managing partner or partners of a laboratory held by a partnership, the authorized agent of a corporate owned laboratory, or the principal executive officer or ranking elected official of a municipality or other local government entity which owns or operates the applicant laboratory. The signer shall verify in the application that it was prepared under his direction or supervision and that the information it contains is, to the best of his knowledge, true, accurate and complete.

252:301-3-2. Contact information

In addition to other information required by this Chapter, an application shall contain the following information:

- (1) The name, mailing address, street address, telephone number, e-mail address and telefax number (if any) of the applicant.
- (2) The signature, typewritten name, address, telephone number and telefax number (if any) of the authorized representative of the owner.
- (3) The name, mailing address, street address, telephone and telefax number (if any) of the applicant laboratory's authorized technical representative.
- (4) The location(s) (address or legal description) of the laboratory, including county and driving directions and latitude/longitude.
- (5) Identification of the accreditation type and categories, analytes and/or methods sought.
- (6) The name and address of any owner, stockholder, or officer of the applicant laboratory or any person who receives compensation from the applicant laboratory, who has been or currently is an owner, stockholder, or officer of, or who has received compensation from, any

laboratory whose accreditation application has been previously denied or whose accreditation has been previously suspended or revoked in part or in whole by the DEQ.

252:301-3-3. Operational information

The application shall address the following operational issues:

- (1) A listing of equipment to be used for sample analysis, storage and reporting.
- (2) A description of the methods, equipment and instruments used by the applicant laboratory for specific analytes which may be in the form of an SOP manual when required.
- (3) A written laboratory QA plan which includes but is not limited to:
 - (A) A listing of laboratory personnel, including the laboratory director, which gives the academic training, experience and analytical and supervisory responsibilities of each; and
 - (B) A narrative description of the methods used for sample receipt, storage and disposal.
- (4) Results of laboratory's two most recent proficiency testing rounds, at least 15 calendar days apart.
- (5) A report of a laboratory evaluation conducted by the DEQ or a DEQ approved assessor within the twelve (12) months prior to the date of filing or, for in-state laboratories only, a letter requesting the DEQ to conduct an on-site evaluation. The evaluation report shall verify data submitted in an application, list any deficiencies and be signed by the DEQ or DEQ approved agent.
- (6) If deficiencies are listed in an evaluation report, the applicant shall submit a corrective action plan which specifies deadlines for implementation and completion of the plan. The DEQ may establish conditions, including compliance schedules, for the applicant's corrective action plan.
- (7) Hours of operation.

252:301-3-4. Renewals

- (a) **Annual renewal required.** A laboratory must apply to renew accreditation annually.
- (b) **Laboratory responsibility.** Each laboratory is responsible for renewing its accreditation by the annual renewal date. Failure to receive a renewal form and invoice does not exempt laboratories from meeting the renewal deadline.
- (c) **DEQ invoice date.** By April 15 of each year, the DEQ shall mail the renewal forms and invoices to each accredited laboratory.
- (d) **Renewal deadline.** The renewal application shall be accurately completed, signed and submitted to the DEQ with the renewal invoice and all applicable fees by 4:30 p.m. or postmarked on or before June 15. Any renewal application which is not accurately completed and is returned to the applicant or which is postmarked after June 15 but received on or before July 15 shall be considered only if accompanied by the renewal fee and a late fee. Any renewal application and fees received or postmarked after July 15 will be returned and accreditation shall not be renewed.
- (e) **Specified dates.** If any date specified in this section falls on a weekend or holiday, the date of the following working day shall be the effective date.
- (f) **Failure to renew.** To become accredited again, a laboratory that failed to renew its accreditation in a timely manner must apply for initial accreditation as a new laboratory.

PART 3. CONDITIONS OF ACCREDITATION

252:301-3-31. Conditions applicable to all accreditations

The following conditions shall apply to all existing accreditations and shall be incorporated expressly or by reference into all accreditations issued or renewed after the effective date of this Chapter.

(1) **Proper operation and maintenance.** The Laboratory shall at all times properly operate and maintain all facilities and equipment installed or used by the Laboratory to achieve compliance with the laboratory accreditation requirements of the Code, rules of the Board as they relate to laboratory accreditation, and the provisions and conditions of this Accreditation. Proper operation and maintenance includes effective performance of operations and adequate funding, operator staffing and training, and the provision of appropriate sample-handling equipment. All operational practices and procedures used at this site shall conform to the best possible public health and safety practices.

(2) **Duty to mitigate.** The Laboratory shall take all reasonable steps to minimize or correct any adverse impact on the environment and the public health resulting from noncompliance with this Accreditation and to minimize or correct any adverse impact on the environment arising from its analytical activities.

(3) **Duty to provide information.** The Laboratory shall furnish to the DEQ, within a time specified, any information which the DEQ may request to determine:

(A) whether cause exists for amending, suspending, or revoking this Accreditation;

(B) compliance with this Accreditation; or

(C) whether an accreditation should be issued or renewed.

(4) **Records.** The Laboratory shall keep its Accreditation, the application on which it is based, copies of all records required to be kept by OAC 252:320 and the provisions of its Accreditation on file at the accredited facility.

(5) **Reporting requirements.** The Laboratory shall give advance notice to the DEQ as soon as possible of any planned physical alterations, additions to the accredited facility or planned changes in the accredited facility which may result in noncompliance with accreditation requirements.

(6) **Signatory requirement.** All applications, reports, or information submitted to the DEQ shall be signed by the applicant.

(7) **Consent to conditions.** Commencing analytical activities as an accredited laboratory under DEQ Accreditation shall constitute consent to all conditions of Accreditation.

(8) **Transfer of accreditation.** Accreditation is not transferable. An accredited laboratory may apply to amend ownership or change names, provided that facilities, equipment, personnel and all other conditions of accreditation remain unchanged.

(9) **Duty to apply.** To maintain its accredited status, the Laboratory shall make timely application for annual renewal of Accreditation.

(10) **Severability.** The provisions of Accreditation are severable, and if any of its provisions or the application of its provisions are held invalid, the application of such provisions to other circumstances and the remaining provisions of the Accreditation shall not be affected thereby.

252:301-3-32. Amendments to accreditations

(a) **Changes to be reported.** Changes in laboratory name, ownership, form of ownership, location, and other changes, including personnel and/or equipment, which may significantly affect the performance of analyses for which the laboratory was originally accredited shall be reported in writing to the DEQ within 30 days of occurrence. If requested by owner, the DEQ may amend the accreditation to reflect reported changes.

(b) **Amendment fee.** An amendment fee shall be assessed in accordance with OAC 252:301-1-9.

(c) **Cause.** The DEQ may amend an accreditation for cause, with notice to the affected accredited laboratory and opportunity for hearing.

252:301-3-33. Self-reporting

An accredited laboratory shall promptly submit correct facts or information to the DEQ and/or to the client when:

- (1) it becomes aware that it failed to submit a material fact or submitted incorrect information in an application or a report to the DEQ or to a client for submission to the DEQ; or
- (2) the DEQ becomes aware of same and notifies the laboratory.

Failure to make a prompt submission may result in an enforcement action.

PART 5. GROUNDS TO REVOKE

252:301-3-51. Grounds to take enforcement action

In addition to the grounds listed in 27A O.S. §2-3-501 *et seq.*, § 2-4-305(A) and OAC 252:4-7-15, the DEQ may suspend, revoke or refuse to renew in part or in whole the accreditation of any laboratory for the following grounds:

- (1) consistent and significant errors in analyses, erroneous reporting or evidence of professional or technical incompetence;
- (2) misrepresentation to others regarding the type and conditions of DEQ accreditation and the reliance of others on such misrepresentation;
- (3) failure to perform any of the following:
 - (A) to correct deficiencies, comply with a corrective action plan, or take other action required by the DEQ pursuant to these rules;
 - (B) to participate or produce acceptable results in required proficiency testing;
 - (C) to cooperate with or allow on-site laboratory evaluations, inspections, or access to records; or
 - (D) failure to notify or submit reports to the DEQ as required by this Chapter
- (4) submission of a proficiency testing sample to another laboratory for analysis, and reporting data received as its own;
- (5) collaboration with other laboratories on results before proficiency testing sample results are submitted to the required agency;
- (6) allowing persons other than qualified laboratory employees to perform and report results of accredited analytes;
- (7) any other violation, action or inaction presenting good cause for such action, or
- (8) failure to make payment when due.

252:301-3-52. Notice

The DEQ may require an accredited laboratory to give written notice to its clients of the suspension or revocation of any part of its accreditation.

252:301-3-53. Individual proceedings

Proceedings for accreditation revocation, suspension or reinstatement shall be conducted in accordance with 27A O.S. § 2-3-501 *et seq.*, and OAC 252:4, DEQ Rules of Practice and Procedure.

SUBCHAPTER 5. GENERAL OPERATIONS

Section

- 252:301-5-1. Posting of accreditation
- 252:301-5-2. Personnel and subcontractors
- 252:301-5-3. Facilities, equipment and supplies
- 252:301-5-4. On-site evaluation
- 252:301-5-5. Recordkeeping and reporting

252:301-5-1. Posting of accreditation

Each accredited participant in the program shall maintain on file a list of the analytes for which it is accredited and shall provide a copy of the list upon request.

252:301-5-2. Personnel and subcontractors

(a) **All accredited laboratories.** All accredited laboratories shall have at least one full-time on-site employee who has laboratory experience in his area of analysis or works under the direct supervision of an individual who has laboratory experience.

(b) **Drinking water accredited laboratories.** In addition, personnel of drinking water accredited laboratories shall conform with the requirements established by the EPA for laboratories which analyze drinking water. Refer to the Manual for the Certification of Laboratories Analyzing Drinking Water, 5th Edition, January 2005.

(c) **Water quality accredited laboratories.** A general water quality accredited laboratory shall have an employee or supervisor qualified as follows:

(1) **Commercial laboratory.** At least one employee or supervisor in a commercial laboratory shall have an earned bachelor's degree in chemistry or a closely related field from an accredited institution of higher education, plus two years of analytical experience in an environmental laboratory.

(2) **Industrial laboratory, including a branch office or substation of a company's laboratory.** An employee or supervisor of an industrial laboratory, including a branch office or substation of a company's laboratory, must meet the educational requirements for a commercial laboratory unless the employee or supervisor is and has been performing environmental testing for not less than two (2) consecutive years and demonstrates satisfactory participation on performance evaluation samples.

(d) **Subcontractors.** A laboratory may subcontract with another laboratory (subcontractor) for analysis of analytes. Such subcontracting shall be only for those analytes for which the subcontractor is DEQ-accredited.

252:301-5-3. Facilities, equipment and supplies

(a) **All accredited laboratories.** All equipment, reagents, glassware and supplies necessary for the proper performance of laboratory analyses shall be on hand or readily available on the premises for analytes certified or analytes listed in an application for accreditation. Equipment shall be in good working order and properly maintained and shall consist of, at a minimum, the apparatus and supplies for which the laboratory is accredited. Facilities shall have a sink with hot and cold running water, electricity, a source of distilled and/or deionized water, proper laboratory waste disposal procedures, and other features/equipment necessary to properly perform approved EPA analytical methodologies. Facilities may be physically located apart in

separate buildings if the sites are within one (1) mile of each other and under the same direct management.

(b) **Drinking water accredited laboratories.** In addition to the general facilities, equipment and supply requirements, equipment required of a drinking water accredited laboratory shall include the apparatus and supplies listed by EPA or the DEQ or identified by the EPA for laboratories which analyze drinking water.

252:301-5-4. On-site evaluation

(a) On-site evaluations may be unannounced.

(b) During an on-site evaluation the DEQ may require on-site analyses of proficiency test samples by laboratory personnel.

(c) Following the on-site evaluation the DEQ will provide the laboratory with a copy of the evaluation report. The laboratory will be afforded a designated time period in which to correct any listed deficiencies. The DEQ will require a laboratory to develop and implement a Corrective Action Plan (CAP).

(d) Out-of-state laboratories already in the program may be required to have an on-site evaluation performed by a DEQ approved assessor. The laboratory shall be solely responsible for costs associated with the on-site evaluation, if any. The evaluation report shall be submitted to the DEQ along with any CAP if needed.

(e) The laboratory shall have an on-site evaluation prior to granting an initial accreditation.

(f) Prior to granting accreditation for an additional field of accreditation to a laboratory, DEQ may perform an on-site evaluation of the laboratory. All laboratories must pay an appropriate on-site evaluation fee for each evaluation requested by the laboratory for the additional fields of accreditation.

(g) DEQ may conduct on-site evaluation of a laboratory to ensure compliance with this Chapter approximately biennially, or upon receipt of complaint.

252:301-5-5. Recordkeeping and reporting

(a) The laboratory shall keep the following records on file in its accredited facility:

- (1) Accreditation and the application on which it is based;
- (2) copies of all records and documentation required to be kept by this Chapter;
- (3) repair and maintenance records;
- (4) reports filed with the DEQ or submitted to clients for filing with the DEQ;
- (5) equipment changes, additions or malfunctions; and
- (6) QA/QC plans and reports.

(b) Any data report given to a customer by an accredited laboratory shall identify:

- (1) the parameters for which the laboratory is DEQ-accredited;
- (2) the class of DEQ-issued accreditation of each analyte; and
- (3) which analytes were subcontracted out for analysis and the subcontracting laboratory's DEQ-issued accreditation number for each of the subcontracted analytes.

SUBCHAPTER 7. PROFICIENCY TESTING

Section

252:301-7-1. Applicability

252:301-7-2. Participation required

252:301-7-3. PT sample treatment

- 252:301-7-4. Initial accreditation
- 252:301-7-5. General Requirements
- 252:301-7-6. Cost responsibility
- 252:301-7-7. Alternate program
- 252:301-7-8. DEQ PT samples
- 252:301-7-9. Restrictions on exchanging information
- 252:301-7-10. Maintenance of PT records
- 252:301-7-11. Evaluation of PT results
- 252:301-7-12. PT report
- 252:301-7-13. PT report deadline
- 252:301-7-14. PT criteria for laboratory accreditation
- 252:301-7-15. Failure to perform
- 252:301-7-16. Analyte absence
- 252:301-7-17. Supplemental studies
- 252:301-7-18. Corrective action

252:301-7-1. Applicability

The requirements of this subchapter shall apply to proficiency testing for drinking water accredited laboratories and general water quality accredited laboratories.

252:301-7-2. Participation required

A laboratory must participate in two single-blind, single-concentration, regularly scheduled Proficiency Testing (PT) studies per calendar year for each analyte in each class of accreditation for which it seeks accreditation or renewal of accreditation. PT samples must be provided by a National Environmental Laboratory Accreditation Program (NELAP) Approved PT Provider.

252:301-7-3. PT sample treatment

Samples shall be analyzed and the results returned to the PT study provider no later than 45 calendar days from the scheduled study shipment date. The laboratory shall ensure that all PT samples are handled, *i.e.*, managed, analyzed and reported, in the same manner as actual environmental samples utilizing the same staff, methods as used for routine analysis of that analyte, procedures, equipment, facilities, and frequency of analysis.

252:301-7-4. Initial accreditation

To gain initial or interim accreditation, a laboratory shall successfully analyze two consecutive proficiency testing (PT) rounds. Proficiency testing (PT) rounds must have been performed within the last twelve (12) months and at least fifteen (15) calendar days apart.

252:301-7-5. General requirements

- (a) Laboratories seeking to renew accreditation must successfully analyze vendor supplied, regularly scheduled proficiency testing samples approximately six (6) months apart in each calendar year. Failure to meet the semiannual schedule shall be regarded as a failed study on the last day of the seventh (7th) month.
- (b) Laboratories shall successfully analyze at least two (2) PT studies within the most recent three rounds attempted (2 of 3) prior to renewal. Laboratories may analyze additional or supplemental studies; however, such studies must be reported to the DEQ.
- (c) General water quality proficiency testing samples must be Water Pollution (WP) type testing

samples.

(d) Drinking water proficiency testing samples must be Water Supply (WS) type testing samples.

(e) Petroleum hydrocarbon proficiency testing samples must include benzene, toluene, methylbenzene and xylene (BTEX) and Total Petroleum Hydrocarbons (TPH). Both soil and water matrices must be analyzed if both soil and water samples are to be accepted by the laboratory.

252:301-7-6. Cost responsibility

Laboratories shall bear the cost of any subscription to a proficiency testing program required by the DEQ. The DEQ shall not be charged a fee for the analysis of any proficiency testing samples.

252:301-7-7. Alternate program

The DEQ may designate an alternate proficiency testing program if it determines such designation is appropriate.

252:301-7-8. DEQ PT samples

As part of a laboratory's proficiency testing, the DEQ may also submit blind audit samples to an accredited laboratory.

252:301-7-9. Restrictions on exchanging information

A laboratory shall not attempt to obtain the prepared value of any PT sample from its PT Provider prior to the conclusion of the PT study.

252:301-7-10. Maintenance of PT records

The laboratory shall maintain copies of all written, printed and electronic records, including but not limited to bench sheets, raw data, instrument strip charts or printouts, data calculations, and data reports, resulting from the analysis of any PT sample for a minimum of five (5) years. The records shall include a copy of the PT study report forms used by the laboratory to record PT results. All of these laboratory records shall be made readily available during on-site inspections of the laboratory.

252:301-7-11. Evaluation of PT results

PT study providers shall evaluate results from all PT studies using NELAP-mandated acceptance criteria. Each result shall be scored on an acceptable/not acceptable basis.

252:301-7-12. PT report

The PT study provider shall provide the participant laboratories and the DEQ a report showing the laboratory's DEQ identification number and EPA identification number, prepared value, the acceptance range, and the acceptable/not acceptable status for each analyte reported by the laboratory and any other information the DEQ deems necessary for accreditation purposes. The report and all associated data shall also be made available in electronic format as specified by the DEQ. The report shall be submitted electronically or mailed no later than twenty-one (21) calendar days from the study closing date.

252:301-7-13. PT report deadline

Laboratories shall ensure that the PT provider has submitted all pertinent PT reports to the DEQ by 4:30 p.m. on or before July 15 of each year. Laboratories whose reports are postmarked or received after July 15 will not be considered for accreditation renewal on September 1.

252:301-7-14. PT criteria for laboratory accreditation

The following criteria apply individually to each analyte in each class of accreditation as defined by the laboratory seeking accreditation in its application:

- (1) Results of the PT study shall be considered successfully analyzed when the results are "acceptable" and are within the acceptable limits established and published by the PT Provider.
- (2) Successfully analyzed shall also mean an aggregate passing score of ninety percent (90%) for microbiological PT testing studies. No partial credit will be given;
- (3) The DEQ shall consider PT results along with the other elements of these rules when determining a laboratory's accreditation status;
- (4) For initial accreditation or supplemental testing, the studies must be at least fifteen (15) calendar days apart.

252:301-7-15. Failure to perform

The DEQ shall not renew accreditation for a failed or omitted analyte or category of analytes for a laboratory which does not meet the requirements of this subchapter. Once accreditation for an analyte or a category of analytes has been lost, the procedures for initial or interim accreditation shall apply.

252:301-7-16. Analyte absence

- (a) **Generally.** If a PT sample is not given for a particular analyte for which a laboratory is requesting accreditation, accreditation for the analyte may be obtained by qualifying for accreditation for the entire category in which the analyte is found. To be eligible for accreditation in the entire category, the laboratory shall pass seventy-five percent (75%) of all PT available analytes within the category. If a laboratory completely fails an individual analyte and still receives a 75% passing rate, the laboratory will not be granted accreditation for that particular analyte but will be accredited for the rest of the category.
- (b) **Exception.** Laboratories which have or are pursuing accreditation for the Basic Environmental Category are not subject to subsection (a) of this section.

252:301-7-17. Supplemental studies

A laboratory may elect to participate in PT studies more frequently than required by the semiannual schedule. Additional studies are not distinguished from the routinely scheduled studies. They are counted and scored the same way and must be at least fifteen (15) calendar days apart.

252:301-7-18. Corrective action

When a laboratory fails a study, in part or in whole, it shall determine the cause for the failure and take any necessary corrective action. The laboratory shall then document both the investigation and the action(s) in a corrective action report (CAP). The CAP shall be submitted to the DEQ within forty-five (45) days of PT study report issuance.

SUBCHAPTER 9. QUALITY ASSURANCE/QUALITY CONTROL

PART 1. QUALITY ASSURANCE/QUALITY CONTROL GENERAL CRITERIA

Section

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PART 1. QUALITY ASSURANCE/QUALITY CONTROL GENERAL CRITERIA

252:301-9-1. Quality Assurance/Quality Control (QA/QC) Plan required

Every accredited laboratory shall maintain a written QA plan and implement it into a QA/QC program to ensure that all routinely generated analytical data are scientifically valid and defensible and are of known and acceptable precision and accuracy.

252:301-9-2. Format

- (a) The QA plan shall list the following on the title page:
 - (1) a document title;
 - (2) the laboratory's full name and address;
 - (3) the names, addresses and telephone numbers of all individuals responsible for the laboratory;
 - (4) the name of the quality manager (however titled);

- (5) the identification of all major organizational units which are to be covered by this QA plan; and
 - (6) the effective date of the version.
- (b) The QA plan shall also contain a Table of Contents, applicable lists of references and glossaries, and appendices.

252:301-9-3. Management information required in QA plan

The QA plan and related quality documentation shall state the laboratory's policies established in order to meet the requirements of this rule. The QA plan and related quality documentation shall also contain:

- (1) a quality policy statement, including objectives and commitments, by top management;
- (2) the organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;
- (3) the relationship between management, technical operations, support services and the quality system;
- (4) job descriptions of key staff and reference to the job descriptions of other staff;
- (5) identification of the laboratory's approved signatories; at a minimum, the title page of the QA plan must have the signed and dated concurrence, (with appropriate titles) of all responsible parties including the quality manager(s), technical director(s), and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager; and
- (6) the laboratory management arrangements for exceptional departures from documented policies and procedures or from standard specifications.

252:301-9-4. Procedures required for QA Plan

The QA plan shall address supporting procedures including technical procedures and shall outline the structure of the documentation used in the quality system, including but not limited to the following:

- (1) ensuring that all records required are retained
- (2) control and maintenance of documentation through a document control system which ensures that all standard operating procedures (SOPs), manuals, or documents clearly indicate the time period during which the procedure or document was in force;
- (3) achieving traceability of measurements;
- (4) handling submitted samples;
- (5) feedback and corrective action whenever testing discrepancies are detected or departures from documented policies and procedures occur;
- (6) dealing with complaints;
- (7) protecting confidentiality (including national security concerns) and proprietary rights;
- (8) audits and data review; and
- (9) establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training.

252:301-9-5. References included in QA Plan

The QA plan shall make reference to the following:

- (1) the calibration and/or verification test procedures used;
- (2) the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;

- (3) procedures for calibration, verification and maintenance of equipment;
- (4) verification practices which may include inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes; and
- (5) procedures for reporting analytical results.

252:301-9-6. Additional items included in QA Plan

The QA plan shall also include the following:

- (1) a list of all test methods under which the laboratory performs its accredited testing;
- (2) mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work.

PART 3. STANDARD OPERATING PROCEDURES AND METHODS MANUAL

252:301-9-31. General

- (a) The laboratory shall use appropriate methods and procedures for all environmental tests and/or calibrations within its scope. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of environmental test and/or calibration data.
- (b) The laboratory shall have instructions on the use and operation of all relevant equipment, and on the handling and preparation of samples where the absence of such instructions could jeopardize the results of environmental tests and/or calibrations. All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be kept up to date and shall be made readily available to personnel. Deviation from environmental test and calibration methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the client.

252:301-9-32. Standard Operating Procedures (SOPs)

- (a) Laboratories shall maintain Standard Operating Procedures (SOPs) that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.
- (b) SOPs may be equipment manuals provided by the manufacturer or internally written documents. Test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the methods manual.
- (c) Copies of all SOPs shall be accessible to all personnel.
- (d) SOPs shall be well organized and shall clearly indicate the effective date of the document, the revision number and the signature(s) of the approving authority.

252:301-9-33. Laboratory method manual(s)

- (a) The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.
- (b) This manual may consist of copies of published or referenced test methods or SOPs that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described.

252:301-9-34. Test method(s)

Each test method shall include or reference the following, where applicable:

- (1) Identification of the test method;
- (2) Applicable matrix or matrices;
- (3) Detection limit;
- (4) Scope and application, including components to be analyzed;
- (5) Summary of the test method;
- (6) Definitions;
- (7) Interferences;
- (8) Safety;
- (9) Equipment and supplies;
- (10) Reagents and standards;
- (11) Sample collection, preservation, shipment and storage;
- (12) Quality control;
- (13) Calibration and standardization;
- (14) Procedure;
- (15) Calculations;
- (16) Method performance;
- (17) Pollution prevention;
- (18) Data assessment and acceptance criteria for quality control measures;
- (19) Corrective actions for out-of-control data;
- (20) Contingencies for handling out-of-control or unacceptable data;
- (21) Waste management;
- (22) References; and
- (23) Any tables, diagrams, flowcharts and validation data.

252:301-9-35. Selection of methods

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

252:301-9-36. Sources of methods

(a) Methods published in international, regional or national standards shall preferably be used. The laboratory shall ensure that it uses the latest valid edition of a standard unless it is not appropriate or possible to do so. When necessary, the standard shall be supplemented with additional details to ensure consistent application.

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

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

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

252:301-9-37. Methodology incorporated by reference

The following EPA methods are hereby incorporated by reference:

- (1) "National Primary Drinking Water Regulations", 40 CFR Part 141, published July 1, 2018.
- (2) "Test Methods for Evaluating Solid Waste, Laboratory Manual Physical/Chemical Methods," SW-846 Manual, Third Edition as amended by Final Update I, II, IIA, IIB, III, IIIA, IIIB, IV and VI . See further SW-846-ON-LINE.; and
- (3) "Guidelines Establishing Test Procedures for the Analysis of Pollutants 40 CFR Part 136, published July 1, 2018.

252:301-9-38. DEQ approved methodologies

The following methods are specifically approved by the DEQ:

- (1) TNRCC Method 1005 Total Petroleum Hydrocarbons (>nC6 to nC35)
- (2) Oklahoma GRO 8020/8015(Modified)
- (3) Oklahoma DRO 8000/8100(Modified)
- (4) ASTM mussels
- (5) On a case by case basis as approved by DEQ

252:301-9-39. New rules apply

As the Board promulgates new rules, accredited laboratories shall incorporate those procedures for all accredited analytes upon the effective date of the rule, July 1 of each year.

PART 5. QA/QC PROGRAM REQUIREMENTS

252:301-9-51. QA/QC program required

Each accredited laboratory shall maintain a QA/QC program to demonstrate the precision and accuracy of analyses. The program shall be in place before accreditation is granted. For a minimum of five (5) years, each laboratory shall maintain records of all analyte accredited analyses, including but not limited to those necessary for a QA/QC program. Laboratories shall perform individual quality control for every analyte for which the laboratory is accredited or is applying for accreditation.

252:301-9-52. Drinking water accredited laboratories

A laboratory accredited in drinking water classifications shall conform to the requirements established by the EPA for laboratories which analyze drinking water.

252:301-9-53. General water quality accredited laboratories

QC requirements specified by approved methodologies shall be practiced in the general water quality laboratory. The laboratory shall establish control limits for all types of QC samples.

252:301-9-54. Inorganic chemistry

If approved methodologies do not specify minimum laboratory QA/QC requirements for inorganic chemistry, the following frequencies shall apply:

- (1) A minimum of three concentrations of standards for daily calibration plus one continuing calibration check standard after each ten samples tested. The lab should repeat all samples tested after the last acceptable continuing calibration check if the continuing calibration

check standard is outside plus/minus 10%.

(2) One blank per twenty samples tested (or batch).

(3) If applicable, one spike per twenty samples tested or one per batch.

(4) One duplicate or spiked duplicate per twenty samples tested or one per batch.

252:301-9-55. Organics

If approved methodologies do not specify minimum laboratory QA/QC requirements for organic chemistry, the following frequencies shall apply, provided that where possible, surrogates shall be used for all samples:

(1) A minimum of three concentrations of standards for daily calibration plus one continuing calibration check standard after each ten samples tested inside established control limits; if any analytes are outside the limits, repeat all samples tested after the last acceptable continuing calibration check.

(2) One reagent blank (carried through preparation) per twenty samples tested (or per batch).

(3) One matrix spike per twenty samples tested (or per batch).

(4) One matrix spike duplicate per twenty samples tested (or per batch).

252:301-9-56. QA/QC documentation

(a) Documentation shall be kept to insure quality control has been maintained and that proper methodologies have been used for the preparation and analysis of samples. All documentation shall be maintained and be readily available for reference or inspection.

(b) The following QC documentation shall be maintained in each laboratory.

(1) **Bench records.** Data associated with analysis, date, time, analyst, method, amounts, calculations, sample matrix, sample identification.

(2) **Calibration data.** Calibration curve or coefficient of the linear equation which describes the calibration curve; concentration/response data (or relative response data) for standards; percent recovery of an initial calibration check standard and the date it was analytically determined; percent recovery of the continuing calibration check standard; and laboratory sample identification of the samples run with this curve.

(3) **Extraction/digestion records.** Date, analyst, type of extraction or digestion for each sample, and laboratory sample identification.

(4) **Surrogate records.** Amount of surrogate spiked, percent recovery of each surrogate, date, analyst, and laboratory sample identification.

(5) **Maintenance logs.** By instrument, dates and description of repairs, preventive maintenance, malfunctions, and other actions or events affecting instrument performance.

(6) **QC charts.** Quality control procedures for monitoring the validity of the environmental testing. The resulting data shall be recorded in such a way that trends are detectable, and statistical techniques shall be applied to the reviewing of the results. All laboratories shall have detailed written protocols in place for positive and negative controls, variability, repeatability, and accuracy of the methods.

(7) **Sample login.** Procedures plan for sample login, unique sample identification, date, time, source of sample (including name, location and sample matrix), preservative used, analysis required, name of collectors and any pertinent field data.

(8) **Spike/duplicate/spike-duplicate data.** Date, analyst, laboratory sample number, amount spiked, percent recovery, percent of difference, and makeup and concentration in the spiking solution.

(9) **Temperature logs.** By oven, incubator, freezer, and or refrigerator, daily (during periods of use) temperature readings and any temperature excursions with corrective action,

recorded in a bound logbook.

(10) **Weight logs.** By balance, checked with the appropriate range of class S weights weekly (during periods of use) before use and results recorded in a bound log book. Each balance serviced and calibrated at least once per year by an accredited technician.

252:301-9-57. Support equipment

All support equipment shall be calibrated or verified at least annually, using a recognized National Metrology Institute, such as NIST, traceable references when available, bracketing the range of use. The results of such calibration or verification shall be within the specifications required of the application for which this equipment is used or the equipment shall be removed from service and tagged as such until repaired.