Minnesota Pollution Control Agency (MPCA)
Laboratory Certification Program Manual
Revision 0

This manual was written with plain language in mind. When 'we' or 'us' is used, this is referring to the Certification Program Administrator, his/her designee, or MPCA agency staff that implement the certification program requirements. When 'you' is used, it refers to the laboratory and applicable laboratory staff. Contact information can be found on the MPCA Quality Systems webpage: www.pca.state.mn.us/ktqh3d9.

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I. PURPOSE

The purpose of this manual is to communicate the certification program requirements for meeting MPCA data quality needs to the certified laboratories. The program applies to the certification of Minnesota municipal, government, or industrial laboratories that perform analyses as needed for NPDES/SDS permit compliance or water quality work for the MPCA. Applicable samples for compliance include wastewater discharge, local pretreatment, biosolids, and water quality samples as specified in permits and for covered programs. The certification program manual does not apply to commercial laboratories.

II. ADMINISTRATION

We are authorized to issue and renew certification, to reject applications for certification, and to issue suspensions and revocations. If we deny, suspend, or revoke certification, you may appeal to the MPCA Supervisor of the Environmental Data Quality Unit or the Director of the Environmental Outcomes Division.

We maintain the information supplied by the laboratory that is applicable to your quality system and compliance with the manual. We keep a current list of certified laboratories, copies of certificates including the list of parameters, methods and matrices, copies of the MPCA Laboratory Certification Program manual and its revisions and attachments.

We will meet with the MPCA Laboratory Certification Steering Committee for advice on training and outreach activities, manual updates, laboratory evaluation summaries, and annual fees.

III. DEFINITIONS

See Attachment 1.

IV. PARAMETERS AVAILABLE FOR CERTIFICATION

The parameters available for certification are included in Attachment 2.

Analysis of field parameters: dissolved oxygen, pH, temperature, conductivity and total residual oxidants when performed as field tests, do not require analysis by a certified laboratory. However, these tests are available for certification. Facilities that perform analyses and report results for field parameters are required to meet the Clean Water Act, permit, and approved method requirements as written in the most current update of 40 CFR Part 136.

Analysis of Whole Effluent Toxicity does not require certification. This testing is reviewed by specialists at the MPCA due to specific technical requirements.

If you need additional parameters or methods other than those found in Attachment 2 and 40 CFR Part 136, contact us and we will provide you with the procedure to obtain certification, if eligible.

V. CERTIFICATION

The following is a list of requirements for Certification which must be met prior to certification. Once certified, it is a violation to not meet each of the following requirements.

1. Laboratory Procedures.

   Analytical methods, sample preservation, sample containers and sample holding times are required to conform to the requirements in: 40 CFR part 136 and 40 CFR Part 503, as amended; and Test Methods for Evaluating Solid Waste, SW-846, as amended and published as final.

   When the test methods and regulation requirements are more stringent than the manual, you must meet the more stringent requirement unless the agency grants specific, alternative allowances. When it is not clear which of the requirements is more stringent, you must follow the test methods or regulations. We have the authority to make decisions concerning stringency when the applicability of a requirement is questioned.

   We may approve other analytical procedures, parameters, or parameter method technologies that have been demonstrated to produce verifiable and repeatable results and that have a widespread acceptance in the scientific community using the Alternate Testing Procedure (ATP) approval process in 40 CFR Part 136. If an ATP is needed, you may request this by writing to us. The request should include the supporting documentation listed in 40 CFR Part 136.5. The MPCA also has guidance at www.pca.state.mn.us/ktqh3d9 .

2. Proficiency Testing (PTs).

   The requirements for Proficiency testing are established in part 7001.4390 and must be followed.

   In the case of unacceptable results for multiple analytes, a laboratory must not fail the same analyte within a test method on two consecutive PT studies even if the method as a whole is considered acceptable by the PT vendor. This type of failure requires immediate corrective action including the generation of a corrective action report and the ordering and analysis of another PT sample containing the failed analyte. If the

laboratory fails the same analyte in two consecutive PTs their results for that analyte will be considered unacceptable to the MPCA until they can demonstrate they have taken corrective action and can produce an acceptable PT result.

3. General Requirements.
   a. Personnel: You are required to have personnel on staff that have the education, training or experience that allows them to meet the requirements of this manual. At least one individual within the organization is to be identified to us as responsible for the laboratory quality system. You are responsible for updating us when there are changes in contact information within 30 days of the change.
   b. Facilities and Equipment: You are required to have the appropriate, properly maintained equipment. The facility must be appropriate for analyzing samples. You are responsible for notifying us if there are changes in location or address within 30 days of the change.
   c. Quality System: You are required to have a Quality Assurance and Quality Control Program that meets the manual requirements, including procedures for proper documentation.
   d. External Evaluations: We will perform on-site evaluations. You must make the time and all relevant records available for the laboratory evaluation.
   e. Records: You are required to have procedures for proper documentation. If you accept samples from another laboratory or facility, you are required to maintain any analytical records needed to determine compliance with this manual. All records shall be made available to the permittee providing the samples, as well as to the MPCA, upon request.
   f. Subcontracting: If you need to have samples for permit required testing analyzed by another laboratory, you must use laboratories that have valid certifications under this manual or by another approved accrediting authority.

      Refer to the Quality Systems and Laboratory Evaluation sections for additional information and requirements.

4. Laboratory Evaluations.

   a. We will perform on-site evaluations to determine your ability to meet the requirements in this manual along with the requirements for the test methods.
   b. We will conduct on-site evaluations if we determine it is necessary to verify corrective action implementation of previously cited deficiencies or if there is reason to believe a laboratory is not in compliance with this manual.
   c. We will use procedures for the evaluations that are documented and promote consistency. You will be provided with feedback forms that may be used to communicate to the Supervisor of the Environmental Data Quality Unit.
   d. We will document the deficiencies discovered during an on-site evaluation in a report that will be provided to you.
   e. Evaluation corrective action and response.
      1. You are required to respond in writing to the evaluation report and to document corrective actions to address the deficiencies. The responses are due 30 days from the date of evaluation report and you need to include supporting documentation showing that the issues are resolved. If you need more time, contact us to discuss the delay and negotiate an expected delivery date.

2. We will review the response submitted and inform you whether the response and supporting documentation address all of the deficiencies satisfactorily or if additional action or documentation is needed.
3. If we need additional action or documentation, we will discuss this with you and will agree on a date for the second response.
4. If the second response is insufficient, another on-site evaluation or enforcement may be needed. If another on-site evaluation is needed to determine compliance with this manual, we will let you know the deadlines for the resolutions of remaining deficiencies.
5. When we determine that the submitted corrective action plan(s) address all of the deficiencies, the laboratory will be informed in writing.

VI. QUALITY SYSTEMS

1. General.

   The laboratory Quality System is an overall program that includes the Quality Assurance (QA) policies that specify the measures required to produce defensible data with known precision and accuracy along with the Quality Control (QC) processes needed to demonstrate the laboratory's competence and to ensure and document the quality of the analytical data.

   The manual will clarify the requirements related to the use of approved methods, quality assurance, equipment, personnel, sample handling, documentation requirements, and QC requirements. You are required to have at least one individual within the organization identified as responsible for the laboratory's quality system and the requirements in this manual.

2. Methods.

   You are required to use methods for environmental testing approved by covered programs under this manual, suitable for the matrix, analyte, and expected level of analyte, regulatory limit and potential interferences in the samples to be tested. When methods are not prescribed by covered programs under this manual or permits issued by the MPCA, you must contact us to provide assistance in selecting a method that is suitable.

   Approved analytical methods can found in the most recent update of 40 CFR part 136 and 40 CFR Part 503, as amended. Refer to the Certification Section, Laboratory Procedures, for additional information.

3. Quality Documents.

   The document information below, where applicable, is required to be included or referenced in your Quality Manual and/or Standard Operating Procedures (SOPs). You may organize these documents in a manner that works for you. If an item is needed as part of the Quality Manual, but is found in the SOP, the SOP can be referenced.

   The documents must be revised to address any changes. The SOPs need to be reviewed each year and the Quality Manual every two years. The laboratory is responsible for submitting the current documents to us.

   a. Quality Manual.

You are required to have a document defining the analytical quality control practices applicable to the certified parameters. The quality manual must include the name of the laboratory/facility, and effective date. All personnel must follow the policies and procedures in the quality manual. Below are the items to include or reference:

1. The name of the person responsible for the Quality System.
2. The organizational structure.
3. All policies and procedures that define the quality system.
4. A list of the major analytical instrumentation and support equipment.
5. The procedures for sample handling and preservation, including containers.
6. The traceability of standards, reference materials, and reagents used to obtain all results and measurements. You must be able to link the standards and reagents to the sample results.
7. The calibration, verification, and maintenance of major analytical instruments and support equipment.
8. The procedures and frequency for method detection limit studies or verifications and how reporting limits are determined.
9. The procedures for evaluating quality control samples, including (but not limited to) method blanks, laboratory control samples, laboratory control sample duplicates, matrix spike samples, matrix spike duplicates or sample duplicates.
10. If applicable, for bacterial methods, include the required QC checks for the positive and negative controls, sterile blanks, duplicate samples, and known quantitative cultures (when required).
11. Procedures for initiating, investigating, resolving and documenting corrective action addressing QA/QC failures or other discrepancies.
12. Procedures for reviewing data and reporting results.
13. Recordkeeping - the maintenance of records and documents associated with analyses.

b. Standard Operating Procedures (SOPs).

Standard Operating Procedures describe the analytical methods to be used in the laboratory in sufficient detail that a competent analyst unfamiliar with the method can conduct a reliable review and/or obtain acceptable results. SOPs are unique to the laboratory, describing tasks performed on a day-to-day basis, tailored to the laboratory's own equipment, instrumentation and sample types.

The SOPs must include the name of the laboratory/facility, the effective date(s), and may be included as part of the Quality Manual. Personnel are required to understand and follow all the procedures. Below are the items to include or reference:

1. Identification of the Method Reference(s).
2. Applicable analytes.
3. Applicable matrices.
4. Method sensitivity - Method Detection Limit.
5. Interferences.
6. Equipment and Supplies.
7. Reagents and Standards.

8. Sample preservation, storage and hold time.
9. Quality Control: including the demonstration of capability procedure and acceptance criteria; the QC samples analyzed, their frequency and acceptance criteria.
10. Calibration and Standardization, including zeroing of the instrument and acceptability of the initial calibration.
11. Procedures for preparation and analysis.
12. Corrective actions for out of control data.

4. Facilities and Equipment.

You must maintain the laboratory facilities and the equipment. The equipment must be able to meet the minimum criteria of the approved methods, the requirements in this manual, and meet the sensitivity applicable to meet permit limits. This also includes the source of water used, along with the glassware, chemicals, supplies and other equipment required to perform all the analytical procedures applicable to the certified parameters.

a. Analytical Instrumentation. The instruments must be operated by trained personnel who follow the instructions for their proper use and maintenance. The analytical instrumentation is required to be included or referenced in your Quality Manual and SOPs. The instructions for use and maintenance must be available to the analysts.

   If the instruments indicate that contamination or other issues are affecting results, the instrument may require maintenance, inspection, or cleaning. If it is necessary, take the instrument out of service. Once returned to service, you must assess and ensure the equipment calibration and function will meet requirements before analyzing samples and reporting results. You must document the maintenance done.

b. Support Equipment.

   You must maintain support equipment to ensure proper working order. If you need to remove support equipment from service due to performance, you must assess and ensure the equipment meets the requirements before it is used in support of the generation of sample results. You must document the measurements and any maintenance completed.

   1. Support equipment needs to be calibrated or verified at least annually and in accordance with the manufacturer's requirements. These checks need to include the range of use (when needed) and be done using reference materials traceable to NIST (where available). The acceptable criteria for the checks are in accordance with approved test methods, MPCA guidance, or, in their absence, established by the manufacturer.
   2. Types of support equipment and the calibration/verification requirements:
      (a) Thermometer/thermistor devices used to measure the temperature: if the certificate supplied by the manufacturer includes a calibration expiration date, you are required to calibrate/verify them prior to this date. Then, you are required to calibrate/verify them within one year. If a calibration expiration date is not indicated, the checks must be done each year. The calibration/verification is completed with thermometers traceable to NIST. Alternatively, they can be replaced with currently certified thermometers.
      (b) Equipment requiring temperature measurement:

      Temperature measurements apply to: laboratory refrigerators, freezers, ovens, incubators, water baths, autoclaves, hot blocks or hot plates, and when required, room temperature.

Record the temperatures on days of use, and per the method, and meet the temperature requirements. You must record the value with the appropriate correction factor, the date, time and initials. Also, document time periods when specific timing is required by the method. For example, in the BOD analysis, document the time and date the samples are placed in the incubator and the time and date the samples are taken out of the incubator.

(c) Balances. You must check analytical balances on days of use with at least 2 certified weights. The weights used should bracket the range of use or per method requirement. The balance checks must meet the tolerances applicable to their use. You are required to also check non-analytical balances on days of use. Balances should be evaluated by a qualified vendor each year as an additional check on the balance and the weights.

(d) Weights used for analytical balances. These must be traceable to NIST, be ASTM Class 1 or 2 or equivalent quality, and be used within the calibration certification expiration date. Recertification by appropriate vendors of the weights is required prior to the expiration date, and within five years of the last calibration. You must handle the weights correctly to protect their integrity.

(e) Mechanical and automatic volumetric dispensing devices: you must check these for accuracy at least quarterly when they are in use. Types of this equipment include those that have an accuracy requirement, such as: mechanical pipettes, micro-pipettes, burettes and automatic dilutors and dispensers. This requirement does not apply to gas-tight glass microliter syringes or class A glassware.

(f) Glassware: if you are using glassware that is Class A, you may use the manufacturer's certificate. Verify Class B glassware before using for the first time or once per lot.

5. Sample Handling.
   a. You are required to follow the sample preservation procedures and holding times required by state and federal regulations. Refer to Table II, in the most recent update of 40 CFR Part 136, for the container, preservation, and holding times for aqueous samples to be analyzed for compliance with the Clean Water Act. Refer to the Sampling and Analysis section in 40 CFR Part 503, as amended, for requirements related to biosolids, for container, preservation and hold times or alternatively refer to updated versions of SW–846 "Test Methods for Evaluating Solid Waste". Reference methods may include additional information.
   b. Document sample collection information such as: location, date, time, and collector initials. Collect enough sample so all the testing can be done to the sensitivity needed. Protect samples from breakage or contamination. Contamination can be from the environment, containers, reagents, and materials used.
   c. Samples need to be received and maintained in the condition needed for the analytical method intended. If you complete analysis for other facilities, see below.
   d. You must preserve samples correctly and check and document the preservation. If you complete analysis **only for samples generated by your facility**, once you have shown the chemical preservation is sufficient over time, you may reduce the pH checks to each season or whenever there is a suspected change in the matrix.
   e. Sample storage. You must have procedures and facilities for the proper storage of samples that protects the samples from contamination, loss or damage or deterioration. Store samples separately from food, standards, reagents and other sources that could potentially cause contamination. Sample extracts and digestates need to be stored according to the method requirements and this manual.

f. When you complete analyses for samples accepted from other facilities, you are required to make sure additional documentation is done:

1. Document the receipt and condition of all samples in hard copy or electronic records. Document any departures from the requirements.
2. The name of the facility submitting samples.
3. The dates of sample collection (and time of collection if holding times are 48 hrs. or less).
4. The facility relinquishing the samples - include the personnel signature, date and time.
5. The laboratory receiving the samples - include the receiver's initials, date and time.
6. The unique sample identification code assigned by the laboratory.
7. Documentation of sample preservation status (chemical and temperature) and other sample conditions on receipt. If the samples do not meet the required preservation, you need to discuss this with the facility. If the samples were just collected and hand delivered, they must show evidence that the cooling process has begun, such as arriving on ice.
8. You are required to have a written policy for sample acceptance and rejection.
9. There must be a link between the sample identification code assigned by the laboratory accepting the samples and the field collection identification assigned by the collector.
10. The requested analyses, and if required, the requested test methods.

6. Traceability, Documentation, Records and Reporting.

You must consistently document all records required to ensure reference method and MPCA Laboratory Certification Program manual traceability requirements are met. You are required to maintain all records necessary to allow historical reconstruction of all laboratory activities that contributed to generating reported results. You must handle and store the records and documents in a manner that ensures they are secure and that their retrieval is possible for the required time period.

a. Recordkeeping.

1. You are required to have a written procedure for the management of all records and documents. You must keep all records and documents that are part of your laboratory quality system according to state and federal requirements. Keep documents pertaining to the analysis of discharge samples applicable to NPDES/SDS permits for a minimum of 3 years and, for biosolids, a minimum of 5 years. Keep records longer than the minimum time if they are needed to reconstruct analytical results during the 3 or 5 year period.
2. If we require a longer time requirement we will send a request to you.
3. If the laboratory changes ownership, or ceases to be certified, you must let us know in writing who is responsible for the retention of the documents and records.
4. You are required to make sure records and documents are legible and their entries are safeguarded against obliteration, erasures, overwriting and corruption. Handwritten records must be in permanent ink. Correct errors by using a single line cross out, and writing the correct value nearby. Corrections should be initialed and dated.
5. You must maintain records and documents that are stored only on electronic media by keeping the hardware and software necessary for their retrieval and reproduction into hard copy for a minimum of 3 years for NPDES/SDS permit related results and 5 years for biosolids.

b. Administrative records required.

1. Retain laboratory certificates.
2. Maintain records of initial demonstration of capability (IDC) for each analyst required to perform the testing.
3. Maintain copies or access to the currently approved method references, quality documents, the MPCA Laboratory Certification Program manual, along with other regulations, as needed.

c. Analytical records. You need to maintain all analytical and technical records containing raw and derived data. See below for examples:
   1. Collection and arrival dates and times of samples received for analysis, where applicable.
   2. Analysis and preparation dates (and times for tests with holding time of 48 hours or less).
   3. Preservation status of samples on arrival at the laboratory, where applicable.
   4. Measurements of laboratory support equipment associated with sample analysis and storage.
   5. Identity of laboratory personnel preparing and testing samples.
   6. Identification of the analytes or analyte groups analyzed in samples.
   7. Preparatory methods that are used for sample digestions and extractions.
   8. Methods of analysis used for samples.
   9. Results of sample analysis, including the units.
   10. Traceability of standards and reagents used to perform preparation and analysis.
   11. Initial and continuing calibration data associated with samples analyzed.
   12. Calibration verification information.
   13. Raw data for analytical instrument calibrations and samples.
   14. Results of quality control samples associated with samples analyzed.
   15. Corrective actions associated with samples analysis.
   16. Maintenance performed on laboratory support equipment and analytical instruments.
   17. Environmental conditions, when crucial to the test method, at the time samples are prepared or analyzed.
   18. Reports of final results submitted to clients or the MPCA.

d. Standards, Reagents and Reference Materials.
   1. You are required to use standards and reagents that meet the purity requirements in the approved methods.
   2. Use reference materials, when available, that are traceable to (NIST) standard reference materials (SRMs) or commercially available reference materials traceable to international or NIST SRMs.
   3. You are required to retain records for all standards, reagents, and bacteriological media.

      Stock standards and reagents: Record: Vendor/Manufacturer name, Certificate of Analysis, Lot number, Date of Receipt, and expiration date. Include on the reagent or standard container, the date received and opened. In case there is no expiration date supplied by the manufacturer, contact them for the expiration date; for some solid reagents or for acids, the vendor typically may not supply an expiration date, in these cases, they are assigned by the laboratory.

      When the laboratory is preparing a reagent or standards: Record: the identification of the stock, preparation date, the method of preparation (initial and final amounts of each), the preparer's initials, and the expiration date.

If these are prepared each day of use, and not retained beyond the analysis day, the method of preparation must be in the SOP. This is needed unless the information is recorded each time so that there is clear traceability to the standards and reagents that were used.

4. The storage specifications of the reagents, standards, and consumables must be in accordance with the manufacturer instructions and done in a manner that prevents contamination.
5. You may not use standards and reagents beyond their expiration dates for quantitation. Prepared solutions should not exceed the expiration date of the stocks used.

e. Laboratory Reports.

1. If you are performing testing for samples generated by your facility for permit compliance, you are responsible for preparing regulatory reports in a specified format to the MPCA (e.g. the eDMR). You need to report data qualifiers where applicable. Qualifiers apply when the quality control limits were not met and it was not possible to reanalyze the samples; or if there were problems associated with the sample collection or condition; or in situations where the data are estimated.
2. If you complete analysis for other facilities or industries for permit compliance, you must supply the following:
   (a) Name, address, contact name and telephone # of the lab where samples were analyzed.
   (b) Lab EPA ID, or other certification ID of the lab where samples were analyzed.
   (c) Name and address of the client/facility whose samples were analyzed.
   (d) Sample description/ID provided by the client/facility.
   (e) The unique sample description/ID code assigned by the laboratory.
   (f) Test report date.
   (g) Collect date *(and collection time if holding time is 48 hrs. or less).*
   (h) Receipt date and time.
   (i) Parameter name, final result, reporting limit and unit of measure.
   (j) Analysis method used.
   (k) Analysis date *(and analysis time if holding time is 48 hrs. or less).*
   (l) Name and signature of the person authorizing reporting of the results.
   (m) The laboratory COC (include the temperature upon receipt if cooling is required).
   (n) If the parameter requires that an MDL be reported, then report MDL (method detection limit) along with the RL (reporting limit).
   (o) The sample batch QC results.
   (p) Dilution factors: The MDL and/or reporting limit must be adjusted by the dilution factor.
   (q) Applicable data qualifiers associated to the affected results.
   (r) If you have to supply a revised report, clearly explain the reasons for the revision, the date of revision, and reference the original report.

3. Pre-Treatment: If you have a signed written agreement from an industry that the reports may be issued without all the contents above, this agreement must be kept on file. You are required to be able to supply all the contents above, including the laboratory quality control results, if request by us or by the industry.

4. Subcontract results. You should review reports supplied by subcontract laboratories to make sure the reports include the laboratory certification ID; also review qualifiers applicable to results. Any analysis data, including reporting limits and data qualifiers that are transferred into another format (e.g. the eDMR), must be transcribed correctly.

7. Quality Control Requirements.

   The purpose of the content in the Quality Control Requirement section is to provide specific requirements, and clarification, when they are **not** sufficiently addressed in the analytical method or their associated quality control references. These QA/QC elements are required by 40 CFR 136.7, when applicable. Additional information on specific subjects can be found on the MPCA Quality System webpage: www.pca.state.mn.us/ktqh3d9.

   a. Instrument Calibration.

      Initial calibrations are performed when the instrument is set up and whenever calibration-verification criteria are not met. Verify the calibration before generating sample results. When there are method requirements for the frequency of the initial calibration and calibration verification, the method criteria must be met when it is more stringent. Calibrations establish a relationship between the instrument response and the analyte concentration and must include a sufficient number of non-zero standards that is appropriate for the calibration model, the instrument and its intended use.

      Sample results must not be reported if the sample response is above the response of the highest calibration standard. For some technologies (such as ICP, ICPMS), results may be quantified above the highest calibration standard but within the linear dynamic range, as allowed in the method.

      If there is an agreement or requirement to report the parameter to the method detection limit, the result between the MDL and RL requires qualification as estimated.

      Initial Calibration and Verification requirements:

      1. You must use the calibration model appropriate for the instrument.
      2. You must reference or include calculations, integrations, and equations used to generate the calibration curve. Note: certain instruments are tuned to conform to a universally accepted scientific law or scale, such as DO meters, ion selective electrodes, and pH meters, which are adjusted or tuned according to their specific principals and therefore do not require a calibration equation in the SOP.
      3. You must use at least the minimum number of standard concentrations for calibrations, which is 3 non-zero standards. Some exceptions are: Dissolved Oxygen (DO) meters; and ICP / ICPMS calibrations which may be calibrated with fewer standards in accordance with the approved methods.
      4. Non-linear calibrations will require additional calibration standards.
      5. Six calibration standards are required if the calibration model is quadratic.
      6. The concentration of the standards chosen to establish a calibration function should be at approximately equally spaced intervals and cover the expected concentration of the samples.
      7. The laboratory must specify how the instrument is zeroed and the treatment of calibration blanks.
      8. The low standard should be at or below the reporting level.

9. You must compare the calibration curve generated to the acceptance criteria. The type of criteria chosen and the acceptance range shall be appropriate for the type of analytes quantitated and the calibration model selected.

   Inorganic methods and organic methods using a linear fit typically require a correlation coefficient (R) > / = 0.995.

   For ISE and pH electrodes, compare the slope to the manufacturer's criteria.

10. If the method does not specify the acceptance criteria for the initial calibration, the criteria in an equivalent method may be used.
11. If the initial calibration criteria cannot be met, you need to evaluate the cause. If it is due to a single standard, a troubleshooting measure you may take is to re-prepare and reanalyze the standard.

    As long as the minimum number of calibration standards required are present, there are acceptable policies for removing either the highest or lowest calibration point. Middle point calibration standards must not be removed.

12. You need to verify all initial instrument calibrations after they are generated with a second source standard (with the exception of DO meters). When a factory calibration is used, additional verification is necessary. Verification must be done with at least three standards that cover the operational range and the accuracy of each standard must meet the manufacturer's criteria.
13. All sample results must be generated after the calibration curve meets the acceptance criteria.
14. You are required to complete calibration verification each day the instrument is used or with the frequency specified in the method. The standard recoveries must meet the acceptance limits of the second source standard (unless otherwise specified).
15. You must dilute samples if they exceed the highest calibration standard (unless otherwise specified). It is best to use the lowest dilution necessary to obtain a response within the calibration range.
16. You need to perform a new calibration after instruments undergo maintenance that could affect instrument response, and when their continuing calibration cannot be verified. When a factory calibration is used, additional verification is necessary. Verification must be done with at least three standards that cover the operational range and the accuracy of each standard must meet the manufacturer's criteria.
17. Since calibrations apply to the generation of sample data, make sure to retain these records for the additional time needed to reconstruct the permit and biosolids sample results for the required time period for the covered program.
18. Other requirements:

    You need to select the simplest linear calibration function unless a non-linear function provides a documented improvement. You may not use non-linear functions to compensate for instrument problems, such as: saturation, insensitivity, or malfunction.

    Once you have selected a calibration model, established the calibration function, and finalized the initial calibration, you may not change the model or function after samples have been analyzed without performing another calibration.

    You may use weighted algorithms or reduction techniques, unless they are used to compensate for instrument saturation, insensitivity or malfunction.

    You are not allowed to use calibrations that are forced through zero, unless explicitly allowed in the approved method. However, average response factors and automatic zeroing as part of an initial calibration is allowed when written in applicable methods.

b. Initial Demonstrations of Capability (IDC).
    1. You are required to have personnel with the education, training, or experience that allows them to meet the requirements in the test method and in this manual. You must have procedures for training and maintain records that show analysts are capable of completing the required tasks.
    2. Analysts are required to be able to demonstrate proficiency in performing the method and obtaining accurate results for each certified parameter for which they generate results.
    3. If the approved methods include specific demonstration of capability procedures and criteria, then use the method instructions. If the method or quality control sections do not include this information, you must determine and document your own procedures and criteria.
    4. Typically, an initial demonstration of capability (IDC) includes analysis of a reagent or method blank that is less than the lowest reporting limit, along with at least four laboratory control samples (LCSs) which meet the method or laboratory acceptance criteria for accuracy and precision.
    5. Microbiology methods require correct identification and quantification, along with method precision.
    6. IDC records must be provided when requested.
c. Method Detection Limit.
    1. Unless the method detection limit (MDL) is not required for a specific test, you must establish the MDL for each test and instrument. Use an acceptable procedure, such as the procedure in 40 CFR Part 136, Appendix B, unless otherwise specified.
    2. MDL studies should be done each year, and are also required when there is a change in the method or instrumentation that has an effect on the sensitivity. Alternatively, the MDL can be verified each year using a defensible procedure.
    3. You must include the sample processing steps in the MDL study. MDL verifications, if done, must take into account the processing steps.
    4. It is recommended the MDL study be completed over at least a 3 day period to obtain a more representative result. The MDL results should be evaluated using accuracy, precision, and response. It is recommended that the standards recover ~ 50-150% of the true value, the RSDs are </= 20%, and the response of the standards have ~3 x the response of the method blank.
    5. Reporting limits (RL) must be above the MDL, and are recommended to be set between 2 and less than 10 times the MDL result.
    6. When MDL studies are required and there are multiple instruments in the laboratory for the same test, you need to complete studies for each instrument. Establish a policy of which MDL and RL will be used. One recommendation is to use the highest MDL and RL for the specific analytical method for all like instruments.
    7. Tests where MDLs are not required: DO, BOD, CBOD, titration tests, tests where analyzing a fortified sample is impossible, and gravimetric tests. For these tests, the laboratory must establish the test's sensitivity and reporting limit.
d. Batch: Refer to the definitions in Attachment 1.
e. Blanks.
    1. A reagent blank consists of reagent water and all reagents (including preservatives) that normally are in contact with a sample during the entire analytical procedure.

2. Method blanks are reagent blanks that are prepared with the samples. These blanks are used to determine whether the reagents and the preparative analytical steps contribute to measurement uncertainty or may be a source of potential contamination.
3. As a minimum, you must include one method blank with each sample set (batch) on a 5% basis (1 per 20 samples) or each day samples are analyzed, unless otherwise specified.
4. If unacceptable contamination is present in the method blank, identify and eliminate the source.

   Typically, sample results are suspect if the method or reagent blank result is equal to or greater than the reporting level (unless the sample concentration is at least 10 times the method blank result).

   Samples analyzed with an associated blank that has unacceptable contamination must be re-prepared and reanalyzed when possible; if reanalysis is not possible, the affected sample results must be qualified.
5. The method blank, along with other QC and the samples, are analyzed only after the calibration has been verified.

f. Laboratory Control Sample.

1. A laboratory control sample (LCS) is a reagent water sample to which a known concentration of the analyte(s) of interest have been added.
2. An LCS is used to evaluate laboratory performance and analyte recovery in a clean matrix.
3. At a minimum, you are required to include one LCS with each sample set (batch) on a 5% basis (1 per 20 samples) or each day samples are analyzed. Process the LCS through all sample preparation and analysis steps.
4. Evaluate the LCS for percent recovery of the added analytes by comparing results to method specified limits, control limits, or other approved criteria. If the LCS acceptance criterion is not included in the method, use control limits to generate your laboratory's acceptance limits.
5. If LCS results are out of control, take corrective action, including re-preparation and reanalysis of associated samples if required. When the LCS recovery is higher than the upper control limit and the samples are below the RL, no reanalysis or data qualification is required.
6. The LCS concentration should be high enough to be measured precisely, but not so high as to be irrelevant to measured environmental concentrations. For methods with a calibration curve, the LCS should be near the middle of the calibration curve and must never exceed the highest calibration standard.
7. If you don't have enough sample, for methods that require an MS/MSD, you may substitute an LCS/LCSD for precision.

g. Matrix Spike.

1. A Matrix Spike (MS) is an additional portion of a sample to which a known amount of the analyte(s) of interest is added before sample preparation. For some parameters, an MS is not appropriate.
2. The MS is used to evaluate analyte recovery in a sample matrix.
3. If an MS is feasible and the method does not specify MS frequency requirements, then include at least one MS with each sample set (batch) on a 5% basis (1 per 20 samples) or each day samples are analyzed.
4. It is recommended that the MS concentration be at the same concentration as the LCS so that the analyst can compare the recoveries to evaluate the matrix effect. If possible, estimate the amount of the analyte in the sample, so that a high background level does not have an adverse effect on the MS

recovery. When the analyte background is more than 4-5x the matrix spike concentration, the accuracy of the recovery may be affected.

5. Evaluate the MS for percent recovery of the added analytes by comparing results to method specified limits, control limits, or other approved criteria. If the MS criterion is not included in the method, use control limits to generate your acceptance limits.
6. If MS results are out of control, then take corrective action to try to rectify the matrix effect. If this is not possible, qualify the results.

h. Matrix Spike Duplicate (MSD) or Sample Duplicate.

1. Duplicate samples and/or MSD samples are analyzed to assess precision on an ongoing basis.
2. The method may indicate when a sample duplicate or an MSD is more appropriate. If samples are typically reported as <RL, an MSD is a more appropriate evaluation of precision.
3. An MSD is a second portion of the sample described above to which a known amount of the analyte(s) of interest is added before sample preparation. The added concentration should be the same as the MS.
4. If the method does not specify a frequency, then at a minimum, include one duplicate sample or one MSD with each sample set (batch) on a 5% basis (1 per 20 samples) or each day samples are analyzed. MSD or duplicate samples are processed as independent samples through the entire sample preparation and analysis.
5. Evaluate MSD results for precision and accuracy (precision alone for duplicate samples). Refer to the approved method specific acceptance criteria for MSDs or duplicate samples. If the precision criterion is not included in the method, use control limits to generate your acceptance limits.
6. If MSD results are out of control, then investigate the cause and take appropriate corrective action. If duplicate results are out of control, then re-prepare and reanalyze the samples; if it is not possible to reanalyze the duplicate or MSD, qualify the results.

i. Internal Standard.

1. Internal standards are used for organic analyses by gas chromatography/mass spectrometry (GC/MS), some GC analyses, some ion chromatography (IC) analyses, and some metals analyses by inductively coupled plasma/atomic emission spectrometry (ICP/AES) and inductively coupled plasma /mass spectrometry (ICP/MS).
2. An internal standard is a unique analyte included in each standard and added to each sample or sample extract/digestate just before sample analysis. Internal standards should mimic the analytes of interest but not interfere with the analysis.
3. Choose an internal standard whose retention time or mass spectrum is separate from the analytes of interest and that elutes in a representative area of the chromatogram. Internal standards are used to monitor retention time, calculate relative response, or quantify the analytes of interest in each sample or sample extract/digestate.
4. When quantifying by the internal standard method, measure all analyte responses relative to this internal standard, unless interference is suspected. Refer to the approved method for specific internal standards and their acceptance criteria.
5. If internal standard results are out of control, take corrective action, including re-analysis if required.

j. Surrogates.

1. Surrogates are used for organic analyses to evaluate method performance in each sample.

2. A surrogate standard is a known amount of a unique compound added to each sample before extraction. Surrogates mimic the analytes of interest and are compounds unlikely to be found in environmental samples.
3. Surrogates are introduced to samples before extraction to monitor extraction efficiency and percent recovery in each sample. Surrogate limits are typically established using laboratory control limits.
4. If surrogate results are out of control, take corrective action (including re-preparation and reanalysis, if possible). Because surrogates are supposed to mimic the behavior of the target analytes, surrogate failures should be qualified and an assessment included in the final report of the possible impact of the failure on the sample data reported.

k. Control Limits.
1. Control limits are generated for both accuracy and precision when needed. There are acceptable procedures that can be referenced to evaluate laboratory performance.
2. The laboratory may use the default (fixed) limits if they are specified in the approved method; if they are not specified then the laboratory must generate their own control limits.
3. Laboratory generated limits should be evaluated at least each year and assessed to make sure they reflect expected performance of the technology.

l. Corrective Actions.
1. QC data that are outside the acceptance limits are evidence of unacceptable error in the analytical process. Corrective action begins with analysts who are responsible for knowing when the analytical process is out of control. Analysts must initiate corrective action when a QC check exceeds acceptance limits or when sample data are out of control.
2. Take corrective action promptly to determine the source of error. Do not report the data until the cause of the problem is identified and corrected, whenever possible. In cases where the sample results must be reported with QC data that are outside the limits, the sample results must be qualified.
3. Qualifying data does not eliminate the need to take corrective actions, but allows analysts to report data of known quality when it is either impossible or impractical to re-analyze the sample(s).
4. Maintain records of all out-of-control events, determined causes, and corrective action taken. The goal of corrective action is not only to eliminate such events, but also to reduce repetition of the causes.
5. Analysts need to communicate the out-of-control event (e.g., QC outliers, hold time failures, loss of sample, equipment malfunctions, and evidence of sample contamination) to the person responsible for the laboratory's quality system.
6. Corrective actions should be considered for the following situations (unless otherwise directed in the method):
    a. calculation or transcription errors;
    b. sample was not prepared and/or not analyzed according to the SOP and the approved method;
    c. QC failures.
        i. For QC failures, reanalyze it, if the second analysis fails, locate the source of the problem and re-prepare and reanalyze the affected samples when possible.

ii. When the calibration verification standard fails, and another standard source also fails, this indicates a problem with the calibration, therefore recalibration may be required.

iii. If method blanks or laboratory control standards fail after a second analysis, the samples may need to be re-prepared. If matrix spikes fail, and the laboratory control standard passes, the analyst should evaluate whether the failure is due to matrix interference. If the matrix interference cannot be eliminated, then qualify the sample results.

7. The person responsible for the laboratory's quality system needs to monitor whether the corrective actions implemented are working. If there are repeated failures, the corrective actions taken may not be addressing the actual problem, therefore re-evaluate the corrective action.

m. Quality Control for Microbiological Tests.

If you analyze samples for bacterial organisms, you are required to follow the approved method and the associated quality control requirements. Because bacteria testing is done on living organisms, there is inherent variability.

While some of the requirements above do not apply, the majority of the Quality Systems requirements are applicable. These include using approved methods for the correct program; using written SOPs; proper sample handling; meeting hold times; assuring the facility does not cause sample contamination; training personnel, using appropriate equipment and supplies; completing documentation, meeting precision criteria; and implementing corrective actions when sample or quality control data are shown to be out of control. Depending on the test method, positive/negative controls and sterility checks may apply.

VII. REFERENCES See Attachment 3**.**