Laboratory Certification

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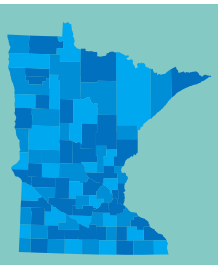
Revision 1

Laboratory Certification Program Manual

A manual for requirements of laboratories certified in the Minnesota Pollution Control Agency Laboratory Certification Program.

MINNESOTA POLLUTION CONTROL AGENCY



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Contents

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 National Pollution Discharge Elimination System (NPDES)/ State Disposal System (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.

This manual was written with plain language in mind. When 'we' or 'us' is used, this is referring to the Certification Program Administrator, their 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 is located on the MPCA Laboratory Certification webpage.

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.

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.

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 Appendix A.

IV. Parameters available for certification

The parameters available for certification are included in Appendix B. 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 and/or 40 CFR Part 503.

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 Appendix B and 40 CFR Part 136 or 40 CFR Part 503, contact us to discuss the process 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. 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.

2. Proficiency Testing (PTs)

Requirements for proficiency testing are established in Minn. Rule 7001.4390 and must be followed. A passing PT performed in the previous 12 months is required for each parameter and method when applying for initial or renewal certification. The laboratory may use discharge monitoring report-quality assurance (DMR-QA) studies to satisfy PT requirements.

a. Testing and reporting of PT samples
    1. Obtain PTs from a nationally recognized accreditation program vendor.
    2. Manage PTs in the same manner as a routine sample.
    3. Submit a copy of the original result report to us within 30-days of receipt.

b. Repeating PTs
    1. If the first PT result is unacceptable, the laboratory must resolve the suspected cause and complete a second PT with 30-days of receiving the unacceptable result report.
    2. If the second PT is unacceptable, the laboratory must resolve the suspected cause, submit a corrective action report to us, and complete a third PT within 30-days of receiving the unacceptable result report.
    3. If the third PT result is unacceptable, the laboratory may not report analytical results for the unacceptable parameters and methods until proficiency is demonstrated by passing two consecutive PTs conducted at least 15-days apart. Submit corrective action report and passing PT results to us within 30-days of receiving the acceptable PT results.
    4. In the case of unacceptable results for multiple analyte group PTs, the previous steps apply but 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.

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. Identify at least one individual within the organization 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 maintain your laboratory and appropriate analytical equipment 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 or virtual evaluations roughly once every three years or as necessary. You must make the time and have all relevant records available for the laboratory evaluation.

e. Records: You are required to have procedures for proper documentation. You are required to maintain any analytical records needed to determine compliance with this manual. All records shall be made available to the client/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 or virtual evaluations to determine your ability to meet the requirements in this manual along with the requirements for the test methods. In most situations advance notice is given prior to an evaluation but unannounced on-site evaluations may occur if necessary. We will also conduct on-site evaluations if determined 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.

b. 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.

c. We will provide an evaluation report that documents deficiencies discovered during an on-site evaluation within 30 business days, and we will contact you if additional time is needed.

d. 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.

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, contact us for 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 procedural changes. The SOPs need to be reviewed each year and the quality manual every two years. Document summary of revisions and reviews in a historical log. 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. Procedures for training of laboratory personnel.
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 (MDL) studies or verifications and how reporting limits are determined.
9. The procedures for evaluating quality control samples, including (but not limited to) proficiency testing, 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.
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.
14. Any additional policies or procedures that define the quality system.

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 and Reporting Limits.
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, analysis, and calculations.
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 client or permit requirements. 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 in 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 returned to service 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 temperature must be traceable to NIST calibration. If the certificate supplied by the manufacturer includes a calibration expiration date, you are required to verify them prior to this date and annually thereafter. Thermometer verification must be completed with a thermometer traceable to NIST and recalibrated to NIST at least every five years. Alternatively, thermometers can be replaced with currently certified thermometers.
      (b) **Infrared (IR) Thermometers** used to measure sample temperature must be verified at least every six months against a NIST traceable thermometer over the range of use to include ambient (20-30^{o}C), iced (0-6^{o}C), and frozen (0 to –5^{o}C). IR thermometer must be checked against a bottle of water that contains a certified thermometer at the temperature of interest on days of use and must agree within 0.5^{o}C.
      (c) **Equipment** requiring temperature measurement:

      Temperature measurements apply to laboratory refrigerators, freezers, ovens, incubators, water baths, autoclaves, hot blocks or hot plates.

      Record the temperatures on days of use and ensure temperature requirements are met. A continual monitoring or minimum/maximum device may be utilized when

equipment is in use over multiple days in absence of lab staff. 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.

(d) **Balances:** You must check balances on days of use with at least 2 certified weights that bracket the range of use or per method requirement. The balance checks must meet the tolerances applicable to their use. Balances must be evaluated by a qualified vendor each year as an additional check on the balance and the weights.

(e) **Weights** used for 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.

(f) **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.

(g) **Glassware:** If you are using glassware that is Class A, you may use the manufacturer's certificate. Verify Class B glassware and plasticware by a gravimetric method to determine mass before first use 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 volume to ensure testing requirements are met. 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 clients/facilities, see below.

d. You must preserve samples correctly and check and document the preservation. If you only perform analysis 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, 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 another facility, industry, or samples in support of an MPCA water quality program , 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 client/facility submitting samples.
3. The dates of sample collection (and time of collection if holding times are 48 hrs. or less).
4. The person 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 or per method requirements. If the samples do not meet the required preservation, you need to discuss this with the client/facility. If the samples were collected and delivered on the same day, 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 for demonstration of capability (DOC) for each analyst required to perform the testing.
    3. Maintain a signature log for all lab personnel to include printed name, signature, and initials.
    4. 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. Unique sample description/ID code.
    3. Analysis and preparation dates and times.
    4. Preservation status of samples on arrival at the laboratory, where applicable.
    5. Measurements of laboratory support equipment associated with sample analysis and storage.
    6. Identity of laboratory personnel preparing and testing samples.
    7. Identification of the analytes or analyte groups analyzed in samples.
    8. Preparatory methods that are used for sample digestions and extractions.
    9. Methods of analysis used for samples.
    10. Results of sample analysis, including the units, dilution, and adjustment to reporting limit if applicable.
    11. Traceability of standards and reagents used to perform preparation and analysis.
    12. Initial and continuing calibration data associated with samples analyzed.
    13. Calibration verification information.
    14. Raw data for analytical instrument calibrations and samples.
    15. Results of quality control samples associated with samples analyzed.
    16. Corrective actions associated with samples analysis.
    17. Maintenance performed on laboratory support equipment and analytical instruments.
    18. Environmental conditions, when crucial to the test method, at the time samples are prepared or analyzed.
    19. 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. For stock standards and reagents document the following:
    - vendor/manufacturer
    - certificate of analysis
    - lot number
    - quantity
    - date of receipt
    - expiration date

    Include on the reagent or standard container, the date received and opened. If an expiration date is not supplied by the manufacturer contact them for the expiration date. If an expiration date cannot be provided the lab must assign an expiration date.

    For laboratory prepared reagents and standards document the following:

    - stock source
    - preparation date
    - method of preparation
    - initial and final quantity of each
    - preparer's initials
    - expiration date

    When reagents or standards are prepared each day of use, and not retained beyond the analysis day, the method of preparation may be included in the SOP, 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 another facility, industry, or water quality program, you must supply the following unless a signed written agreement is on file indicating that a report may be issued without all the contents. You are required to produce the information below, including the laboratory quality control results, if requested by us or by the client/facility.
    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 time.
h. Receipt date and time.
i. Parameter name, final result, reporting limit and unit of measure.
j. Analysis method used.
k. Analysis date and time.
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 client/facility requires that an MDL be reported, then report the MDL 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. Subcontract results. You should review reports supplied by subcontract laboratories to make sure the reports include the laboratory certification/accreditation 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. Calibrations establish a relationship between the instrument response and the analyte concentration and must include a sufficient number of non-zero standards that are 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 a need 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. 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. Use at least the minimum number of standard concentrations for calibrations, which is three 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 limit.
9. 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. If removing the lowest calibration point the reporting limit must be adjusted to the next quantifiable calibration standard.
12. 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, verify with at least three standards that cover the operational range.
13. All sample results must be generated after the calibration curve meets the acceptance criteria.
14. Complete calibration verification each day the instrument is used or with the frequency specified in the method. The standard recoveries must meet the method acceptance criteria.
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. 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 verify with at least three standards that cover the operational range.
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:

    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. Demonstration of capability (DOC)

You must have documented procedures for training and maintain records that demonstrate analysts are proficient in performing the method to obtain accurate results for each parameter. Demonstration of capability is required initially for each analyst prior to generating results and annually thereafter with continued demonstration. Follow the approved method for initial and ongoing demonstration of capability procedures and acceptance when available. If the approved method does not specify DOC requirements use one of the following procedures.

1. Initial Demonstration of Capability
   (a) Chemistry:
      i. Analysis of a reagent or method blank that is less than the lowest reporting limit, along with at least four consecutive laboratory control samples (LCSs) at one to four times the concentration of the reporting limit, which meet the method or laboratory acceptance criteria for accuracy and precision.
      ii. For methods where an LCS is not practical assess initial DOC by participation in an internal blind study that meets acceptance for precision or external proficiency test study.

   (b) Microbiology:
      i. Participation in a blind internal study that meets acceptance for a blank, a negative culture, and a positive culture for the target organism that meets acceptance for laboratory precision.
      ii. Participation in a blind external proficiency study for the target organism.
2. Continued demonstration of capability
   (a) Chemistry:

i. Analysis of four consecutive LCSs that are acceptable for accuracy and precision. You are allowed to assess LCS precision and accuracy from four consecutive batches performed in the past 12 months.

ii. Participation in a blind external proficiency study.

i. Performing another initial DOC.

(b) Microbiology:

i. Participation in a blind external proficiency study for the target organism.

ii. Participation in a blind internal study that meets the laboratory precision for the target organism.

iii. Duplicate sample analysis with a sample that produces a positive count for the target organism and meets laboratory acceptance for precision.

iv. Performing another initial DOC.

c. Method detection limit and reporting limit

1. Method detection limit

(a) Unless the MDL is not required for a specific test, you must establish the MDL for each test and instrument. Follow the procedure in 40 CFR Part 136, Appendix B unless otherwise specified.

(b) Tests where MDLs are not required: pH, DO, BOD, CBOD, titration tests, microbiology, and gravimetric tests. For these tests, the laboratory must establish the test's sensitivity and reporting limit.

(c) An initial MDL is required when there is a change in the method or instrumentation that has an effect on the sensitivity.

(d) You must include the sample processing steps in the MDL study.

(e) The MDL must be below the reporting limit.

2. Reporting limit

(a) You must establish a reporting limit (RL) for each test. Requirements for determining reporting limits may be published in a method.

(b) Generally, the reporting limit is the lowest level of an analyte that can be accurately quantified or measured and must be greater than or equal to the lowest acceptable calibration point.

(c) Where applicable verify the RL on at least a monthly basis and recover at least 40% of the true value, unless the method is more stringent.

(d) The terms level of quantitation, quantitation limit, minimum level, and minimum reporting level have the same meaning as reporting limit.

d. Batch (refer to the definitions in Appendix A)

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 less) 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. Exceptions include when the sample concentration is at least 10 times the method blank result or the analyte of interest is not detected in the sample.

   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.

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 reference Control Limits in section k.
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 reference Control Limits in section k.
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.
7. If you don't have enough sample, for methods that require an MS/MSD, you may substitute an LCS/LCSD for precision.

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 of the samples.

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 the laboratory may generate their own control limits and reference the MPCA Laboratory Quality Control and Data Policy for maximum 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. 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. Establish and follow procedures for communicating out-of-control events (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 continuing 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. While some of the requirements above do not apply, the majority of the Quality System requirements are applicable. These include using approved methods for the correct program; using written SOPs; proper sample handling; meeting hold times; assuring the laboratory 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.

Appendix A

Definitions

The following terms and meanings apply to the MPCA Laboratory Certification Manual, as amended. Since many terms are already explained or defined in the manual itself, this appendix is meant to address other terms.

1. “Acceptable” means those results that apply to proficiency testing samples that are within the specified acceptable limits as indicated by an approved vendor, which are used to determine if a laboratory has analyzed a proficiency test sample successfully.
2. “Agency” refers to the Minnesota Pollution Control Agency (MPCA).
3. “Agency program” means a program or rule administered by the agency that requires submission of water data from a certified laboratory, such as the wastewater or watershed program.
4. “Analyte” means the chemical substance, physical property or organism analyzed in a sample.
5. “Analyte group” means a set of analytes that can be determined using the same method or technology.
6. “Batch” means a set of samples prepared or analyzed together, under the same process, instrumentation, personnel and lot of reagents. A preparation batch refers to a batch of samples that are the same matrix. Preparation batch processing is required to be completed in a 24-hour period. The number of samples allowed in a batch is typically 20 (excluding the quality control samples).
7. “Calibration” means the process used to establish an observed relationship between the response of an analytical instrument and a known amount of analyte, or the process used to determine, by measuring or comparison with a reference standard, the correct value of each scale reading in an instrument, meter or measuring device.
8. “Calibration function” means the specific mathematical relationship established to relate calibration standards to instrument response.
9. “Certification” means that a laboratory has been granted certification by the MPCA laboratory certification program. The Certification Program Administrator (or their designee) completes the functions outlined in the Laboratory Certification Manual. The manual is written with plain language in mind, therefore terms of “we”, “us”, “our” refer to the MPCA staff responsible for the laboratory certification program.
10. “Chain of custody” means the procedures and records that document the possession and handling of samples from collection through disposal. A chain−of−custody (COC) form is used to document, with a signature, date and time, transfer of the sample from collector to transport/delivery service and then to the laboratory staff receiving the samples.
11. “Client” means an entity that has arranged with a laboratory to perform tests and analyses to meet the requirements of an NPDES or SDS permit or another agency program or regulatory requirement.
12. “Correlation coefficient” means a quantity that measures the degree of agreement between the points in a calibration curve and the linear function derived to connect them.
13. “Corrective action” means any measure taken to eliminate or prevent the recurrence of the causes of an existing nonconformity, defect or undesirable condition.
14. “Deficiency” means a documented or verifiable deviation from the requirements of this manual that is noted during an on−site evaluation or while reviewing analytical data produced by a laboratory.

15. “DMR” refers to the Discharge Monitoring Report; “eDMR” is the electronic format.
16. “EPA” means the United States Environmental Protection Agency.
17. “Falsified data or information” means data or information which has been made untrue by alteration, fabrication, omission, substitution, or mischaracterization.
18. “Field Parameters” for the purpose of this manual (as amended) include dissolved oxygen, pH, temperature, conductivity and total residual oxidants.
19. “Laboratory” means a facility that performs tests in connection with a program which requires data from a certified laboratory. The terms laboratory, or laboratories, includes laboratories that are certified or are seeking certification made available by Minnesota Statute 115.84, to which the requirements of this manual apply. The manual is written with plain English in mind, therefore when “you” is used, it refers to the laboratory, along with the laboratory staff responsible for meeting the requirements in the manual.
20. “Method detection limit” or “MDL” means the minimum concentration of an analyte that can be measured and reported with 99% confidence that the concentration is distinguishable from the method blank results as determined by the procedure specified at 40 CFR 136, Appendix B.
21. “MPCA” is the Minnesota Pollution Control Agency.
22. “Municipal” refers to the operation by a municipality, or other local government, of wastewater treatment facilities/plants.
23. “NIST” means the National Institute for Standards and Technology.
24. “On-site evaluation” means an assessment conducted by the agency at a laboratory that is maintaining or seeking certification to determine compliance with the requirements in this manual.
25. “Parameter” means the chemical substance, physical property or organism being measured.
26. “Quality System” means a structured and documented management arrangement describing the policies, objectives, principals, organizational authority, responsibilities, accountability, and implementation plan ensuring quality in its work processes, products and services.
27. “Reporting limit” means the lowest level of an analyte that can be accurately recovered from the matrix of interest, for example, the level of quantitation.
28. “Second source standard” means a standard procured from a supplier or manufacturer different from the supplier or manufacturer of a laboratory’s calibration standards, or a standard obtained from the same supplier or manufacturer of a laboratory’s calibration standards from a lot verifiably different from the lot of the calibration standards.
29. “Traceability of measurement” means the ability of relating a result or measurement to appropriate state, national or international standards through an unbroken chain of documented comparisons.
30. “Violation” refers to a continued unresolved deficiency or a serious verifiable deviation from the requirements in this manual.

Appendix B

Certification parameters

The following parameters are available for certification.

Oxygen utilization	
	Biological Oxygen Demand (BOD5)
	Carbonaceous biochemical oxygen demand (CBOD5)
Nitrogen	
	Ammonia (as N)
	Kjeldahl Nitrogen Total (as N)
	Nitrate (as N)
	Nitrate nitrite (as N)
	Nitrite (as N)
Phosphorus	
	Total phosphorus (as P)
	Orthophosphate (as P)
Physical	
	Residue Total (total solids)
	Residue filterable (dissolved solids)
	Residue Volatile (volatile solids)
	Residue non-filterable (Total Suspended Solids)
	Oil and grease
	Turbidity
	Total, Fixed, and Volatile solids in Solid and Semisolid samples
General I	
	Acidity (as CaCO3)
	Alkalinity (as CaCO3)
	Color
	Hardness-Total (as CaCO3)
	Silica-Dissolved
	Sulfite (as SO3)
	Surfactants
General II	
	Chemical Oxygen Demand
	Total Phenolic Compounds

	Cyanide-total
	Sulfide
	Sulfate
	Chloride
General III	
	Organic Carbon-Total
	Organic Carbon-Dissolved
Metals	
	Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Total Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Vanadium, and Zinc
	Hexavalent Chromium
	Mercury
Microbiology	
	E. coli
	Fecal coliform
	Coliform (total), number per 100 mL
Organics; Purgeable by Gas Chromatography or Gas Chromatography/Mass Spectrometry	
	Volatile Organic Compounds
	Acrolein, Acrylonitrile
Organics; Semivolatile by Gas Chromatography/Mass Spectrometry	
	Phenolic Compounds (acid-extractables) and Base/Neutral Extractable Compounds (excluding pesticides)
Organics; Organochlorine Compounds	
	Polychlorinated Biphenyls
	Organochlorine Pesticides

Appendix C

References

1. EPA Code of Federal Regulations 40, Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures, June 17, 2024.
2. EPA Code of Federal Regulations 40, Part 503, Standards for the Use or Disposal of Sewage Sludge, February 19, 1993.
3. Minnesota Administrative Rules, Chapter 7001.4310 through 7001.4390, Permits and Certifications (2015).
4. Minnesota Statutes, Chapter 115, Water Pollution Control; Sanitary Districts (2023).
5. Laboratory Quality Control and Data Policy, MPCA (December (2024).
6. American Public Health Association, American Water Works Association, Water Environment Federation. Lipps WC, Braun-Howland EB, Baxter TE, eds. *Standard Methods for the Examination of Water and Wastewater*. 24th ed. Washington DC: APHA Press; 2023.
7. EPA Manual for the Certification of Laboratories Analyzing Drinking Water. Fifth Edition.