Laboratory Quality Control and Data Policy

The Minnesota Pollution Control Agency's (MPCA) Laboratory Quality Control and Data Policy provides data quality objectives to ensure that analytical data submitted to the MPCA is at a known level of quality to make defensible decisions and applies to all laboratory data submitted to the MPCA. Reference Tables 1, 2, and 3 to determine the analytical frequency and maximum acceptance criteria necessary for different quality control samples.

Table 1. QC Criteria required for any volatile organic compound data submitted to the MPCA

Table 2. QC Criteria required for any semi volatile organic compound data submitted to the MPCA.

Table 3. QC Criteria required for inorganics and/or metal data submitted to the MPCA.

Methods and laboratory accreditation

The MPCA requires laboratories to have accreditation or certification, if available, with a recognized authority such as the Minnesota Environmental Laboratory Accreditation Program (MNELAP) or the MPCA Wastewater Laboratory Certification Program. The laboratory scope must accurately reflect the method and version performed and reported, when available. If an MPCA program requires use of new analytes or updated methods, the laboratory may wait until the next accreditation renewal period to update their scope of accreditation.

It is required to use methods that are approved or allowable for the programs that they are being used for. These include the Clean Air Act, the Clean Water Act and the Resource Conservation and Recovery Act. If you are unaware of what methods are appropriate for your program, reach out to the applicable program coordinator. This includes sample collection, preservation, handling, preparation methods, and quality control. Exceptions to approved or allowed methods must be reviewed and approved in writing by the MPCA project manager/hydrologist and the MPCA Data Quality Unit prior to use. This includes use of alternate methods or method modifications that change the underlying chemistry and determinative technique. Contact the data quality unit for more information on using an alternate method or method modification by email at qa.questions.mpca@state.mn.us.

Method detection limits

Perform initial method detection limit (MDL) studies and verify on an on-going basis following the procedure outlined in Appendix B to 40 CFR 136 for all applicable test methods. An initial study is also required where changes to a test method may affect sensitivity of the analysis.

Proficiency testing

Laboratories must use a Proficiency Test (PT) provider approved by their accreditation body. If a PT analyte failure occurs, order and analyze a new PT within 30 days. A laboratory must not fail the same analyte within a field of testing on two consecutive PT studies, even if the field of testing is deemed to have passed as a whole. Submit corrective action to the environmental data quality unit at qa.questions.mpca@state.mn.us, for any PT failures impacting MPCA data.

Quality documents

The laboratory must conduct a formal review of all quality documents, including the laboratory quality assurance manual and standard operating procedures (SOP)s, at least annually. Quality documents must be kept up to date when changes are implemented. Outdated quality documents must be retained for a minimum of five years. Laboratories producing data for the MPCA will provide their quality assurance manuals and analytical SOPs upon request.

Data review

Laboratories must have a documented procedure for data review and validation to ensure data quality objectives and data defensibility requirements are met prior to issuing the final report. Data review procedures include a primary review performed by the bench analyst and a secondary review performed by a qualified laboratory staff member that is someone other than the original reviewer. The secondary reviewer must be able to follow the decision-making process of the primary reviewer. Secondary review requires examination of the analytical results in 100% of the analytical batches produced by the laboratory unless otherwise specified in program guidance and/or associated quality assurance documents such as QAPPs/QAPrPs.

Refer to any relevant QAPPs and/or other applicable quality documents for specific details relating to data review, validation, and usability responsibilities. Consultants or third parties submitting data from laboratories to the MPCA are responsible for reviewing laboratory reports to correct errors, omissions and ensuring project quality goals are met prior to data submittal to the MPCA. Refer to the agency's quality control criteria listed in Tables 1, 2, and 3 at the end of this document when project specific criteria are not available.

Corrective actions

Corrective actions related to MPCA projects must be available for review upon request. The documentation submitted for review must include the out-of-control event, the cause, and action taken to correct and prevent the event from reoccurring. Include qualifications to any data reported for situations where the sample reprocessing is not possible.

Sample handling and receipt

To ensure data integrity, the laboratory must have procedures for sample handling throughout the life of the sample. Sample condition upon receipt must be documented and the individual designated on the Chain of Custody must be notified within two business days if there is question to the suitability of a sample for testing. The laboratory will have a procedure to confirm appropriate preservation of the samples and notify the individual(s) designated on the COC if there are deviations. Samples received by the laboratory on the same day they were collected can be above the required temperature range if evidence of cooling is present. This would include the presence of an appropriate cooling material such as loose ice. If any deviations from method preservation requirements are noted, the laboratory must document the problem and notify the client to verify whether the sample will still be useable per the project's data quality objectives. Client authorization to proceed with the analysis must be documented and, where applicable, the samples must be qualified appropriately. The laboratory must provide sufficient bottles to the sample collector so that method batch QC requirements can be met, and the samplers must provide the appropriate amount of sample volume required for analysis. If the necessary amount of sample volume is not provided, the data must be qualified appropriately.

Reagents, standards and reference materials

Use standards and reagents that meet the requirements of the reference method. Obtain reference materials, when available, that are traceable to the National Institute of Standards and Technology (NIST). This includes standards supplied by NIST directly or purchasing the standards through manufactures who supply the traceable materials. Other options include the U.S. Environmental Protection Agency (EPA), or an appropriate international standard setting organization approved by the MPCA and/or MNELAP. Retain records for all stocks, standards, reagents, reference materials, and bacteriological media that allow for traceability throughout the process.

Sample analysis requirements

The laboratory SOP must specify the criteria used for qualitative identification. Examples include retention time, qualifier ion presence, ion ratios, signal to noise, secondary column confirmation, etc. Both qualitative and quantitative identification requirements must be routinely met at and above reporting limits.

Isomers must be resolved based on retention time. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25 percent of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs and must be reported as co-eluting.

If sample dilution is required, the dilution factor shall be the lowest required to obtain an instrument response within the range of the initial calibration. Analyze samples that require dilution due to high analyte concentration at a lesser dilution for multi-analyte methods, when possible.

Calculated Values, Rounding, and Significant Figures

The MPCA doesn't have agency wide requirements for calculated values, significant figures and rounding. However, Laboratories must have a documented and consistent system for values reported to the MPCA and must follow any procedures found in program specific guidance, or any other project/program related quality assurance document.

Records and reporting requirements

Retain all analytical data in a retrievable and reproducible format for a minimum of five years unless the program requires a different record retention period. The retained data must include all information required for the historical reconstruction of the data (SOPs, analytical results, calibration curves, standard and sample prep information, sample receiving information, QC data, and the final report with narrative/data qualifiers). All records must be available when requested by the MPCA during an audit or data review.

Analytical reports must include the minimum specifications according to accreditation requirements, in addition to the following information:

- A copy of the chain of custody received with the samples.
- Clear identification of subcontracted or satellite laboratory, if applicable. Include a copy of the subcontracted or satellite original report, with all sample related information including batch QC.
- Clear identification when referenced analytic methods are performed with approved alternate procedures. This only applies to methods/analytes for which the MPCA requires accreditation.
- Dilution factors and the adjusted Reporting Limit (RL)/Method Detection Limit (MDL) values for samples requiring dilutions.
- All sample result data qualifiers must be present on the same page as the sample results to which they apply and must be clearly linked to the analyte(s) of interest. All batch QC data qualifiers need to be present on the same page as the QC results to which they apply. The qualifier definition can be on a subsequent page.
- If the program requests data reporting between the method detection limit (MDL) and the RL, qualify the result with an estimate or "J" flag. When reporting a non-detect result, qualification must include a comment noting that the target analytes were not identified between the MDL and the RL, unless the results are clearly reported as less than the MDL.
- Include method-required batch QC including spiking levels, recoveries, precision, and QC limits.
- Only report sample data that fall outside instrument calibration limits (or range) with approval from the MPCA project manager or when reasonable effort to obtain the majority of the sample results within the calibration range for multi-analyte methods. Include proper qualification as an estimate along with the reason.
- Clearly identify amended or revised reports along with the date and reason for the revision.

- Consultants must not excerpt data from the report for their own summary tables without providing a copy of the entire report including batch QC information and data qualifiers to any data recipient.

Analytical Quality Control Requirements

Tables 1, 2, and 3 designate the different quality control samples associated with the different analytical groups along with the required frequency and acceptance criteria for any data provided to the MPCA. The tables must be followed unless different frequencies and criteria are noted in the reference method, or any project/program specific quality assurance documentation. Methods that use performance-based criteria for setting quality control limits cannot exceed the limits listed in Tables 1, 2, and 3.

Table 1. QC criteria required for any volatile organic compound data submitted to the MPCA.

Analytical group: Volatile Organic Compounds (VOCs)

Quality control type	Minimum frequency	MPCA acceptance criteria	Measures and/or exceptions[2]
Laboratory method blank	Every batch of 20 or less	< Reporting Limit (RL)	Sample concentration 10x blank concentration or a sample non-detect
LCS containing all analytes	Every batch of 20 or less	70-130%	High biased with sample non-detect
Matrix spike[1]	Every batch of 20 or less	70-130%	Qualify After Reasonable Effort.
Matrix spike duplicate or sample duplicate[1]	Every batch of 20 or less	Recovery: 70-130% RPD: 30%	Qualify After Reasonable Effort.
Surrogates	Every standard and sample	70-130%	Qualify After Reasonable Effort.
Internal standards	Every standard and sample	Method limits	None
Calibration standard recovery requirements	Within method requirements	RL: 50-150% Others: 70-130%	None
Secondary source standard containing all analytes	Every calibration	70-130%	None
Reporting limit check	Monthly	60-140%	High biased with sample non- detects
Continuing calibration verification	Within method requirements	80-120%	High biased with sample non- detects

Table 2. QC Criteria required for any semi volatile organic compound data submitted to the MPCA.

Analytical Group: Semi Volatile Organic Compounds (SVOCs)

Quality control type	Minimum frequency	MPCA acceptance criteria	Measures and exceptions[2]
Laboratory method blank	Every batch of 20 or less	< RL	Sample concentration 10x blank concentration or a sample non-detect
LCS containing all analytes	Every batch of 20 or less	50-150%	High biased with sample non-detect
Matrix spike[1]	Every batch of 20 or less	50-150%	Qualify After Reasonable Effort.
Matrix spike duplicate or sample duplicate[1]	Every batch of 20 or less	Recovery: 50-150% RPD: 30%	Qualify After Reasonable Effort.
Surrogates	Every standard and sample	50-150%	Qualify After Reasonable Effort.
Internal standards	Every standard and sample	Method Limits	None
Calibration standard recovery requirements	Within method requirements	RL: 50-150% Others: 70-130%	None
Secondary source standard containing all analytes	Every calibration	70-130%	None
Reporting limit check	Monthly	60-140%	High biased with sample non- detects
Continuing calibration verification	Within method requirements	80-120%	High biased with sample non- detects

Table 3. QC Criteria required for inorganics and/or metal data submitted to the MPCA.

Analytical Group: Inorganics/Metals			
Quality control type	**Minimum frequency**	**MPCA acceptance criteria**	**Measures and exceptions[2]**
Laboratory method blank	Every batch of 20 or less	< RL	Sample concentration 10x blank concentration or a sample non-detect
LCS containing all analytes	Every batch of 20 or less	80-120%	High biased with sample non-detect
Matrix spike[1]	Every batch of 20 or less	80-120%	Qualify After Reasonable Effort.
Matrix spike duplicate or sample duplicate[1]	Every batch of 20 or less	Recovery: 80-120% RPD: 20%	Qualify After Reasonable Effort.
Internal standards	Every standard and sample	Method limits	None
Calibration standard recovery requirements	Within method requirements	RL: 60-140% Others: 70-130%	None
Secondary source standard containing all analytes	Every calibration	90-110%	None
Reporting limit check	Monthly	60-140%	High biased with sample non- detects
Continuing calibration verification	Within method requirements	90-110%	High biased with sample non- detects

1. If the final Matrix Spike/Matrix Spike Duplicate result doesn't fall within the calibration curve but, the percent recovery meets acceptance criteria, the data is considered usable but must be qualified as estimated. If the percent recoveries of the set don't pass, they need to be diluted and reanalyzed.

2. Exceptions must be appropriately qualified

Frequently Asked Questions.

Q: What is the definition of reasonable effort?

A: Reasonable effort depends on the situation. This may include dilution and/or reanalysis until you no longer have enough sample to perform analysis, or the sample is going to exceed holding time. If you have any questions contact the appropriate project manager.

Q: Where no method criteria exists, will laboratories be audited and accredited to the quality control limits presented in this document?

A: Yes. The laboratory should review each quality control criteria and must determine and follow program/client requirements, where applicable. If criteria are found within the reference method and/or associated project/ program quality assurance document(s), follow the most stringent criteria.

References:

- MN Rules 2100 Subp. 3. D, Subp. 4. C, Subp. 5. D, Subp.6. D 4740.2080 QUALITY ASSURANCE PRACTICES; ALL TEST CATEGORIES. Parts 4740.2087, 4740.2089, and 4740.2095 to 4740.2099

Q: Why, under accreditation requirements, is it listed as "a recognized authority such as the Minnesota Environmental Laboratory Accreditation Program or MPCA Wastewater Laboratory Certification Program."

A: This document is for all data submitted to the PCA, including data from public laboratories.

Q: What if a lab has concerns with the achievability of the acceptance criteria in Tables 1, 2, and 3?

A: The MPCA will periodically check trends in acceptance criteria in tables 1, 2, and 3 to determine if updates and or exceptions to the criteria are necessary. Please direct your concerns to the qa.questions.mpca@state.mn.us.