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 is submitted at a known level of quality to make defensible decisions. This policy applies to laboratory data submitted to the MPCA. Follow Tables 1, 2, and 3, for quality control frequency and acceptance criteria unless the reference method, quality assurance project plan (QAPP), sampling analytical plan (SAP), or required program document takes precedence. 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.
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 with a recognized authority such as the Minnesota Environmental Laboratory Accreditation Program or MPCA Wastewater Laboratory Certification Program. The laboratory scope must accurately reflect the method and version preformed 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.

The MPCA requires use of approved or allowed methods appropriate for the program such as the Clean Air Act (CAA), the Clean Water Act (CWA), and the Resource Conservation and Recovery Act (RCRA). This includes sample collection, preservation, handling, preparation methods, and quality control. Exceptions to approved or allowed methods must be reviewed and accepted by 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 as a whole is deemed to have passed. 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.

Data review

Laboratories must have a documented procedure for data review and validation to ensure data quality objectives and data defensibility are met prior to issuing the final report. Data review should include primary review by the bench analyst and secondary review by qualified laboratory staff that is someone other than the primary analyst. The secondary reviewer must be able to follow the process and decision-making of the primary analyst.

Consultants or third parties submitting data from laboratories to the MPCA are responsible/liable for reviewing laboratory reports to correct errors and omissions before the data are submitted. They are also responsible for reviewing that the project quality goals are met. If the project does not have specific quality criteria, follow the agency's quality control criteria listed in Tables 1, 2, and 3.

Corrective actions

Corrective actions must be available for review if requested. The documentation submitted for review must include the out-of-control event, the cause, and action taken to correct and prevent the event from occurring. 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 MPCA notified 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 MPCA if there are deviations. Samples collected and received on the same day, that are above the required temperature range, must show evidence that the cooling process has begun, such as arriving on 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 meet project data quality objectives. Client authorization to proceed with the analysis must be documented. 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), the U.S. Environmental Protection Agency (EPA), manufacturers that supply NIST standards or NIST traceable standards, or an international standard setting organization. Retain records for all stocks, standards, reagents, reference materials, and bacteriological media that allows for traceability throughout the process.

Sample analysis requirements

The laboratory SOP must specify the criteria used for qualitative identification, for example, 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.

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 for methods performed with an alternate method. This only applies to methods/analytes for which the MPCA requires accreditation.
- For sample results requiring adjustment for dilutions, the dilution factors and the adjusted RL value.
- All sample result data qualifiers must be present on the same page as the sample results to which they apply. 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 requires data reporting between the method detection limit (MDL) and the reporting limit (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.

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 exceptions (qualified appropriately)**
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	70-130%	High biased with sample non-detect
Matrix spike[1]	Every batch of 20 or less	70-130%	
Matrix spike duplicate or sample duplicate[1]	Every batch of 20 or less	Recovery: 70-130% RPD: 30%	
Surrogates	Every standard and sample	70-130%	None
Internal standards	Every standard and sample	Method limits	None
Calibration standard 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	50-150%	None
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 (Qualified appropriately)**
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%	
Matrix spike duplicate or sample duplicate[1]	Every batch of 20 or less	Recovery: 50-150% RPD: 30%	
Surrogates	Every standard and sample	50-150%	None
Internal standards	Every standard and sample	Method Limits	None
Calibration standard 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	50-150%	None
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 (qualified appropriately)**
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%	
Matrix spike duplicate or sample duplicate[1]	Every batch of 20 or less	Recovery: 80-120% RPD: 20%	
Surrogates[2]	Every standard and sample	80-120%	None
Internal standards[2]	Every standard and sample	Method limits	None
Calibration standard 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%	None
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. Surrogates and internal standards are only applicable to metals methods using ICP/MS or ICP/AES technology.