Minnesota Pollution Control Agency

Laboratory Quality Control and Data Policy

The following requirements apply to all laboratory data submitted to the Minnesota Pollution Control Agency (MPCA) except where a project specific approved Quality Assurance Project Plan (QAPP), program guidance or other appropriate systematic planning document takes precedence. Failure to follow this Policy may result in data being unusable for MPCA programs, the report rejected, and, if applicable, the invoice contested.

It is the goal of the MPCA's Quality Assurance (QA) program to ensure that all data submitted to the MPCA be scientifically valid, defensible, and of known precision and accuracy. The data should be of sufficient known quality to withstand scientific and legal challenge relative to the use for which the data are obtained.

The MPCA requires the use of the most current analytical method final version unless an older version is allowed or required by a program, permit, or QAPP. Laboratories performing SW-846 methods must add the most current final versions of these methods, dated February 2007, to their scope of certification on their next renewal date following July 1, 2011. The MPCA will announce when future updates are required.

When the **MPCA** adds additional analytes or methods to program requirements, laboratories will be able to wait until their next certification renewal to add newly available analytes or methods to their scope of certification. If the **laboratory** chooses to expand their capabilities by adding a new method to their analytical services for which certification is required, they must be approved for certification before they can begin accepting client samples.

The following minimum quality control requirements apply unless otherwise specified in the determinative method.

Quality Control Requirements

The MPCA fully expects laboratories to follow approved, final methods, including calibration and quality control requirements, or receive an Alternate Test Procedure (ATP). The modification of methods is acceptable if approved as either a regulator reviewed ATP or within a standard operating procedure (SOP) reviewed and accepted by the laboratory's accreditation authority. Method changes that vary the chemistry or intent of the method are not acceptable without an ATP, including drastic changes to chromatographic conditions that may potentially compromise the integrity of the method required separation.

Sample Handling and Receipt

Samples should be received and maintained in a condition that ensures their integrity for the analytical method intended. Samples can be protected from breakage during shipment using any materials shown to be adequate for the purpose and that do not cause contamination or interferences. The laboratory must verify that the sample is received in the proper container with any applicable preservative in time to comply with the method or Program holding time requirement.

For volatile organic compounds (VOCs) and Gasoline Range Organics (GRO), the laboratory should be able to verify the sample is received in a hydrochloric acid (HCl) preserved vial but will not be able to check the pH until after sample analysis. If the sample does not meet the preservation criteria and is not analyzed within seven days, the data will need to be qualified.

The temperature of samples requiring thermal preservation must be checked and must be within the required temperature range. Samples that are hand delivered directly to the laboratory on the day they were collected may not meet this specification. To be considered acceptable, the samples must show evidence that the cooling process has begun, such as arriving on ice. If any deviations from method 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.

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Standards

Reference standards used in the laboratory must be obtained, when available, from 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.

The laboratory must retain records for all stocks, standards, reagents, and bacteriological media.

The records must include:

- identification of the manufacturer or vendor
- certificate of analysis or purity
- lot number
- date of receipt or preparation
- preparer's initials, if applicable
- method of preparation, when prepared in the laboratory
- storage conditions and location, in compliance with determinative method
- expiration date after which the material may be used for qualitative purposes only (the expiration date of a diluted child standard may not exceed the expiration date of the parent stock standard)
- an unique identifier that will enable traceability to all related records and components

Calibration

The following minimum calibration requirements apply unless otherwise specified in the determinative reference method.

For organic analyses:

The initial calibration must contain a minimum of five levels for each analyte and surrogate of interest for methods requiring multi-level calibrations. For each analyte, at least one of the calibration standards must correspond to a sample concentration at or below the reporting level.

The criterion for linearity of an initial calibration curve based on the average of the response factors is a relative standard deviation (RSD) of $\leq$20 percent for each compound that is included in the calibration standard(s) and is considered to be a target analyte.

A linear or non-linear calibration model based on a least squares regression may be employed when allowed by the determinative method. This approach also may be used for analytes that do not meet the RSD requirement for the average response factor, as long as the calibration verification criteria can be met. For linear and non-linear calibration curves based on a least squares regression (LSR) model construction coefficients which describe correlation as equal to 1.00 when representing the best curve fit must be $\geq$ 0.99. Examples of coefficients that describe correlation are the correlation coefficient (r), the coefficient of determination (COD), and r^2. They must all be $\geq$ 0.99.

Non-linear calibration must not be used to compensate for detector saturation or to avoid proper instrument maintenance. Non-linear calibration should not be employed for analytes typically shown to exhibit linear calibration. The method SOP must specify the compounds for which a non-linear fit may be used. A quadratic (second order) model requires six levels of standards, and a third order polynomial requires seven standards.

Once the calibration model has been selected, all calibration levels must be regenerated using the calibration curve. The absolute value of the percent difference between these two amounts for every calibration level, except the lowest, must be $\leq$ 20 percent or tighter, when tighter criteria are specified by the method. The percent recovery of the reporting limit verification standard must be within $\pm$ 40 percent of the true value. The reporting limit must be verified each time the instrument is calibrated, or monthly at a minimum. The concentration of the verification standard must be $\leq$ the reporting limit.

For metals analyses:

For the initial and daily instrument operation, calibrate the system according to the instrument manufacturer's guidelines using individual or mixed calibration standards. The calibration curve should be analyzed daily with a minimum of a calibration blank and a single standard at the appropriate concentration to effectively outline the desired quantitation range. The resulting curve should then be verified with mid-level and low-level (reporting limit) initial calibration verification standards.

Alternatively, the calibration curve can be analyzed daily with a minimum of a calibration blank and three non-zero standards that effectively bracket the desired sample concentration range. A standard corresponding to the reporting limit must be analyzed with each analytical run sequence. The percent recovery of the reporting limit verification standard must be within $\pm$ 40 percent of the true value, unless otherwise specified in the referenced method. Follow the initial and daily calibration acceptance criteria specified in the determinative method.

The linear dynamic range must be verified at a frequency established by the method, or yearly, if the laboratory reports data between the highest calibration point and the upper limit of the linear dynamic range. Background correction applied to the curve must be as directed by the method referenced.

For all other analyses:

Follow the calibration requirements of the cited reference method.

All calibration curves must be generated using standards that are traceable and verified.

All instrument calibrations must be verified with a standard obtained from a second source. Traceability must be to a national standard, when available. The suggested acceptance limits for this secondary source initial calibration verification analysis is 70 -130 percent but can be determined by the laboratory using charting. The limits must be reasonable as defined by the method.

Calibration Verification

Sample quantitation must be based on the most recent initial calibration. The method referenced must be followed for calibration verification. Generally, unless an instrument is fully calibrated according to the method prior to sample analysis, calibration verification must be performed to ensure calibration curve stability before and after a batch or at a frequency specified by the reference method.

For organic analyses:

The calibration must be verified at least every twelve hours unless otherwise specified by the determinative method. The twelve- hour time period begins with injection of the calibration verification standard or Mass Spectral (MS) tuning check standard. The last sequence injection must occur within twelve hours of the first sequence injection. The calibration verification standard should be near the mid-range of the calibration curve. Additional analyses of the mid-point calibration verification standard during the twelve-hour analytical shift are strongly recommended for methods involving external standard calibration. The calibration verification results must be within ± 20 percent of the response calculated during the initial calibration, or per method requirements. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., >20 percent, and the analyte was not detected in any of the previous samples during the analytical shift, the sample extracts need not be reanalyzed. Data associated with this type of exceedance must be flagged or narration included in the report to show that the laboratory recognized the exceedance and made a conscious decision to report the data.

All sample analyses performed using external standard calibration must be bracketed with acceptable calibration verification standards, unless otherwise specified by the determinative method.

Metals analysis:

Instrument calibration must be verified according to the method at the interval specified in the method or according to criteria that meet or exceed source method criteria.

For all other analyses:

Follow the calibration verification requirements of the cited reference method.

Internal Standards

For organic analyses:

For chromatography methods employing an internal standard calibration, the retention times of the internal standards in the calibration verification standard must be evaluated. If the retention time for any internal standard changes by more than 30 seconds, unless otherwise specified by the method, from that in the mid-point standard level of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions, corrections made, and samples reanalyzed.

For GC/MS methods, if the extracted ion current profile (EICP) area for any of the internal standards in the calibration verification standard changes by more than a factor of two (-50 percent to + 100 percent) from that in the mid-point standard level of the most recent initial calibration sequence, the detector must be inspected for malfunctions, corrections made, and samples reanalyzed.

Sufficient internal standards should be distributed throughout the chromatographic range of the analysis so that the relative retention time (RRT) for the majority of target compounds will be in the range of 0.80 to 1.20.

For inorganic analyses:

The intensities of all internal standards must be monitored for every analysis. If the intensity of any internal standard in a sample falls below the recommended method limits or 70 percent of the intensity of that internal standard in the initial calibration standard, a significant matrix effect must be suspected. If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. This procedure must be repeated until the internal-standard intensities rise to the minimum 70 percent limit. Reported results must be corrected for all dilutions.

Qualitative and Quantitative Identification

The laboratory SOP must specify retention time window criteria for determining a positive identification. For MS methods, criteria for determining a spectral match must be documented in the method SOP.

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. The method resolution requirements must be specified in the laboratory SOP.

When a secondary confirmation column is used, the method SOP must specify the agreement needed to determine a positive identification and the criteria for determining which value is reported. If the value reported does not agree with the SOP specified criteria, the report must fully explain the reason for the deviation and include an explanation of why the reported value was chosen.

For analyses with multi-point calibration:

Samples with concentrations that exceed the calibration range must be diluted to fall within the calibration range. Values exceeding the calibration range may only be reported and flagged after a reasonable effort has been made to obtain values within the calibration range unless there is documented approval of the MPCA project manager or of the party for whom the data are being produced, except when the lab has specific knowledge that the results will still meet the project's data quality objectives.

If sample dilution is required, the dilution shall be the lowest required to obtain an instrument response within the range of the initial calibration. Data from diluted samples that are routinely analyzed at only one dilution with no attempt to analyze the samples at a higher concentration, resulting in unnecessarily elevated reporting limits of non-detected compounds within a multi-analyte method, may be rejected.

An example would be, a sample being analyzed for a multi-analyte method that is diluted 1:100. If the only positive hits from this analysis are near the reporting limit, the sample should be analyzed at a higher concentration so that lower reporting limits can be obtained for the non-detected compounds, unless there are documented extenuating circumstances adequately explained by qualifiers or narration.

For ICP and ICP-MS analyses:

Dilute and reanalyze samples that exceed the calibration range or linear dynamic range, whichever is applicable, for an analyte (or species needed for a correction) or use an alternate, less sensitive line for which quality control data are already established. The linearity at the alternate mass or wavelength must be confirmed by appropriate calibration and all calibration verification QC. Alternatively, apply solid phase chelation chromatography to eliminate the matrix.

If a high level of target compound is present in a sample and is also found in subsequent samples, the analyst must demonstrate that the presence of the target compound is not due to carryover.

Method Blanks

Before analyzing any samples, the analyst must demonstrate that all equipment and reagent interferences are under control. At least one method blank must be prepared per batch of 20 environmental samples or as specified by the method, and prepared on the same day by the same method. For samples requiring extraction, the associated batch method blank must be analyzed with samples from that batch. If there are more samples than can be analyzed in one analytical sequence, a solvent blank may be analyzed with the remaining samples to demonstrate the system is free from interferences.

For samples analyzed for volatiles by purge-and-trap, one method blank must be analyzed with each batch of up to 20 environmental samples analyzed on the same instrument during the same analytical shift or 12 hour time period. Because the preparation is equivalent to the analysis, all other batch QC must also be included in the same analytical shift or 12 hour time period.

The method blank should be analyzed after the calibration verification standard and before sample analysis, unless the determinative method specifies otherwise. The batch method blank must be subjected to the same preparation and clean up procedures as the samples.

When elevated baselines are observed during blank and standard analyses, the chromatographic system should be considered contaminated. Such contamination is unacceptable and should be addressed as specified in the SOP. Samples produced with questionable baselines must be annotated as such in the data report submitted to the client or MPCA.

The target analyte concentration in the method blank should be less than the reporting limit. If the concentration of any target analyte is at or above the reporting limit in the method blank, the associated samples must be reanalyzed or re-extracted if there is any impact on the sample results. If this is not possible, the results must be flagged and the source of the contamination investigated and explained in the report narrative. The impact of the contamination on the sample results must be discussed and an explanation provided if the results are deemed acceptable.

Laboratory Control Samples

A laboratory control sample (LCS) must be included with each preparation batch as required by the reference method. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same solution at the same concentration as the matrix spike (MS), when appropriate, and is processed in the same manner as the samples. All target analytes must either be included in the LCS/MS/MSD spiking solution or, for methods that contain a large number of analytes, the lab may vary the spike components so that all target analytes are spiked at some point during the course of a calendar year.

Unless an analyte is being checked for compliance at a specific action level or a program or project specific quality document specifies otherwise, the LCS should be spiked at a concentration near the middle of the calibration range. The LCS must never be spiked outside the calibration range.

If there are no recommended acceptance limits for the LCS included in the method, the laboratory should use 70-130 percent as interim acceptance criteria for recoveries of spiked analytes until in-house LCS limits are developed. In-house LCS recovery limits should be within the limits calculated for MS recoveries. In-house limits must be examined for reasonableness. Except for very specific circumstances, LCS recovery limits should be within 50-150 percent for semi-volatile organics, 70-130 percent for volatile organics, and 80-120 percent for inorganics.

Samples shall be re-prepared and re-analyzed if the batch LCS is out of control unless there is insufficient sample for re-preparation. If re-preparation is not possible, or deemed unnecessary due to careful and thoughtful analysis of the data by the laboratory, an explanation must be provided in the report as to why the data were reported. All analyte spiking concentrations must be within the calibration range of the instrument.

The LCS result, spiking level, percent recovery, and acceptance limits must be included in the final laboratory report. The MPCA does not recognize the concept of "marginal exceedance" as referenced in the TNI 2009 Standard.

Matrix Spikes/Matrix Spike Duplicates/Duplicates

To assess the effect of the matrix on method performance, the laboratory must analyze at least one matrix spike/matrix spike duplicate (MS/MSD) pair with each preparation batch of up to 20 environmental samples of the same matrix processed together, unless otherwise specified by the determinative method. If samples are expected to contain the target analytes of concern, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample as an alternative to the MS/MSD pair, if allowed by the method. The matrix spikes must be prepared from samples contained in the batch as required by the method and if enough sample is supplied by the sampler. Should inadequate sample be included and an LCSD used to obtain batch precision data, this shall be annotated on the report. The laboratory must provide sufficient bottles to the sample collector so that method batch QC requirements can be met.

The MS/MSD must be another aliquot of the source sample at approximately the same weight or volume spiked with the same solution at the same concentration as the LCS and processed in the same manner as the batch samples. The MS/MSD must never be spiked outside the calibration range. All target analytes must be included in the LCS/MS/MSD spiking solution or, for methods that contain a large number of analytes, the lab may vary the spike components so that all target analytes are spiked at some point during the course of a calendar year.

Calculate the concentration of each analyte in the matrix spike and matrix spike duplicate. Show both the spiked and recovered values for each analyte and include the percent recovery, precision and QC limits in the final report. Alternatively, report the recovery results from the matrix spike and precision results from the sample/sample duplicate pair. Except for very specific circumstances, in an uncontaminated matrix the MS/MSD recoveries should be within 50- 150 percent for semi-volatile organics, 70-130 percent for volatile organics, and 80-120 percent for inorganics.

If any MS or MSD percent recovery falls outside the above-referenced recovery limits, the laboratory should determine if there is a matrix effect or a laboratory performance problem.

Data submitted to the MPCA that contain QC failures must include an explanation for the failures and an assessment of the impact on sample results

Surrogates

The laboratory must develop surrogate recovery limits using submitted samples and the samples must be spiked within the surrogate calibration range. If recoveries are not within in-house surrogate recovery limits, the laboratory must assess the source of the problem. If no instrument problem is found, the sample should be re-extracted and re-analyzed. If, upon re-analysis, the recovery is again not within limits, report the data as an estimated concentration. If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. If the holding time for the method has expired prior to the re-analysis, provide both the original and re-analysis results to the data user and note the holding time problem.

It is recommended that laboratory surrogate limits be greater than 50 percent with a minimum of 30 percent recovery for semi-volatile samples. The maximum recovery limit should be no more than 150 percent.

Surrogate recovery limits for volatiles analyses should be closer to 70-130 percent for most analytes. Follow the determinative method for guidance on the number of surrogates required for an analysis and recommendations for relevant compounds. Because surrogates are supposed to mimic the behavior of the target analytes, surrogate failures should be flagged and an assessment included in the final report of the possible impact of the failure on the sample data reported.

The final report must include surrogate recovery information, including acceptance limits, when surrogates have been used by the laboratory for process control, even when surrogates are not expressly required by the source method.

LCS/MS/MSD/DUP/Surrogates

In-house laboratory limits for accuracy and precision must be updated annually. Fixed limits are acceptable until in-house limits are generated. In-house QC limits must be examined for reasonableness.

Secondary Data Review

All laboratory data reported to the MPCA must undergo secondary data review. Secondary review means that raw data are reviewed, not just the transfer from the instrument data system to the laboratory information management system (LIMS). The analyst's decision-making process must also be reviewed. When reviewing MS data, extracted ion current information needs to be reviewed. The laboratory Quality Manual must specify the level of expertise required to qualify as a secondary data reviewer. Comments or concerns by the reviewer must be addressed prior to publishing of the final report.

Performance Test samples

Laboratories must use a Proficiency Test (PT) provider approved by their accreditation body. A laboratory must generate a written corrective action report within 30 days of receiving notification from an approved provider that a PT sample result for any reported field of testing or analyte is unacceptable. The laboratory must order another PT sample from an approved provider within 30 days after receiving the notification of unacceptable results for a field of testing. 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. This type of failure requires immediate corrective action including the generation of a corrective action report and the ordering and analysis of another PT sample containing the failed analyte. If the laboratory fails the same analyte in two consecutive PTs their results for that analyte will be considered unacceptable to the MPCA until they can demonstrate they have taken corrective action and can produce an acceptable PT result.

Analysis of a PT for an emerging or "experimental" contaminant must pass the same criteria as those for established methods.

Results and corrective actions for all PT samples must be provided directly to the MPCA.

Standard Operating Procedures

The laboratory must conduct a formal review of all SOPs at least biennially with changes incorporated and SOPs updated when they are implemented. Outdated SOPs must be retained for a minimum of five years. Laboratories must have written SOPs detailing the specifics of the method as applied to their facility. Copies of reference methods are not sufficient.

Initial Demonstration of Capability

Each analyst must demonstrate the ability to generate an acceptable initial demonstration of capability (IDC) along with acceptable results according to method recommendations and stated project data quality objectives.

Each laboratory must demonstrate initial capability with each combination of sample preparation and determinative methods that it utilizes, by generating data of acceptable accuracy and precision for the target analytes in a clean matrix. If the spiking solution is prepared by the laboratory, the stock standards used must be independent of those used for calibration. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. Follow the guidance contained in the determinative method for preparing the reference sample concentrate and the reference sample, when available. In the absence of recommended acceptance criteria for the initial demonstration of capability, the laboratory should use recoveries of 70-130 percent as guidance in evaluating the results. If no guidance is provided, document the process in the laboratory SOP. All records must be available for examination during an audit or upon request by the MPCA.

Method Detection Limits

Method detection limit (MDL) studies must be performed for all applicable test methods and instruments initially and each time there is a change in the test method that may affect how the test is performed or when a change in instrumentation occurs that affects the sensitivity of the analysis or at a minimum of every two years to ensure that MDLs are still accurate as equipment ages. Some examples of changes that would require a new MDL study are, but are not limited to, changes in a chromatography column phase, initial column length or diameter, major changes in the temperature program effecting compound separation, changes in mobile phase composition, changes in sample volumes or weights, changes in the calibration range, changes in sample preparation or analysis between manual and automated systems, etc. The laboratory must follow the procedure outlined in 40 CFR 136, Appendix B.

For analyses where multiple instruments perform the same test methods, reporting limits must be established for each instrument. As an alternative, the highest reporting limit for each like instrument and for each specific analytical method may be used for all like instruments.

Analytical Data

All analytical data must be retained in a retrievable and reproducible format for at least the timeframe specified by the data end user. Currently, the MPCA data retention requirement is five years. 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).

Report Requirements

Laboratories must include the following information in their reports:

- Laboratory name, address, and certification number.
- Laboratory contact name and telephone number.
- Client and project name, if applicable.
- Subcontracted or satellite laboratory information, if applicable, clearly identified on the cover page or first narrative page of the report, or the sample data page. Provide a copy of the original laboratory report including batch QC if all information, including batch QC cannot be provided by the contracting laboratory.
- Tests performed for which the laboratory does not hold MPCA-recognized certification, listed on the report cover or first narrative page and the reporting rationale. Annotation shall be made for analytes, test methods or methods with an ATP. This only applies to methods/analytes for which the MPCA requires certification.
- Sample identification, condition, date and time of collection and receipt, date and method of preparation and analysis.
- The reporting limit for each sample with appropriate units of measurement, and the counting error for each radiochemistry sample.
- For sample results requiring adjustment for dilutions, the dilution factors.
- If the laboratory is asked to report data between the method detection limit (MDL) and the reporting limit (RL), positive results should be indicated with an estimated "J" flag. If **no** positive results are reported, a statement should be added to the report narrative noting that the target analytes were looked for between the MDL and the RL and not identified unless the results are clearly reported as <MDL.
- All method required batch Quality Control (QC) elements need to be present in the report including method blanks, laboratory control spikes/laboratory control spike duplicates, matrix spikes/matrix spike duplicates, sample duplicates and surrogates. Spiking levels, recoveries, precision and QC limits must be calculated and reported against the original sample concentration with no blank adjustment or background correction unless explicitly authorized by the cited method.
- All sample 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. Matrix spike source samples need to be flagged when MS/MSD results are outside control limits. All samples in the batch need to be flagged when an LCS is outside control limits.
- Sample results may not be reported when exceeding the instrument calibration limits (or range) without the permission of the MPCA project manager, or the party for whom the data are being produced, or a reasonable effort on the part of the laboratory to obtain the majority of the sample results within the calibration range. If sample results are "E" flagged, the report must contain the reason results are being reported beyond the instrument calibration range.
- The name, function, and signature, or equivalent electronic identification, of the person authorizing the test report and the date of issue. Amended or revised reports need to clearly indicate this information along with the date and reason for the revision.
- A copy of the chain of custody received with the samples.
- A statement that the report must not be reproduced, except in full, without the written approval of the laboratory. 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.

- Deviations from the standard operating procedure, such as failed quality control, additions to, or exclusions from the test method and information on specific test conditions, such as environmental conditions and any nonstandard conditions that may have affected the quality of results must be fully flagged and/or narrated so that the data user understands the impact on the test results and the reason the laboratory chose to report the results.
- When the laboratory analyzes samples by a procedure other than as written, the laboratory record must include sample identification traceable to client, the modification to the procedure, the reason for the modification, and the client's authorization or acknowledgment of the modification.

Audit Requirements

Laboratories providing data submitted to the MPCA must supply raw data to the Agency in support of the laboratory results, upon request. The laboratory must also provide any technical assistance required to answer questions related to the data production.

Laboratories must provide both internal and external audit reports, as requested, during an MPCA data audit or review.

Laboratories must keep all raw data and information related to data production associated with any report submitted to the MPCA for a minimum of five years.

Other requirements for MPCA Programs

A laboratory found to be producing results that are in error will investigate the cause of the error and determine the timeframe during which erroneous results were reported. All clients receiving results produced in error will be notified and will receive amended reports with the corrected data within 30 calendar days of the error being discovered. If the amended report will not be received within 30 days, an estimate of when the report can be expected will be provided to the client. The MPCA will also be notified of the data error and provided with the corrective action report. Laboratories found to continue to produce data under conditions or by a method knowingly incorrect and not providing notification to effected clients will not be considered acceptable to perform work for MPCA programs for a minimum of one year and could face criminal or civil prosecution. Note: Laboratories discovering an error but in negotiations with a certification authority are still required to notify all clients in Minnesota within 30 days of discovery of the error, not at the end of a case or upon completion of a case with the State.

Consultants submitting reports to the MPCA containing laboratory data are responsible for reviewing the contents of the laboratory reports and making sure errors and omissions are corrected before the data are submitted to the MPCA.

A laboratory found to be out of compliance with this guidance must remedy any deficiencies and provide documentation of the correction to the MPCA QA Unit. Within 30 days of notification of the deficiencies, the laboratory must submit documentation of corrective actions planned and taken. If all corrective actions cannot be completed within 30 days of notification, an estimate of the time required to complete the corrective actions will be included in the documentation and the final documents showing proof of corrective action will be submitted when the corrective actions are completed. The case will not be closed until all documentation of corrections have been completed and submitted to the MPCA QA Unit.

If the laboratory does not provide any documentation of corrective actions within 30 days, MPCA programs will be notified not to accept data from the laboratory for the analysis or area of the laboratory in question until further notice.

If the MPCA determines that data from a laboratory should not be accepted by MPCA Programs, the laboratory will be notified in writing. Data will continue to be accepted for fields of testing or analytes not affected by the noncompliance.

Audits conducted by accreditation authorities in support of MPCA programs are required to review representative laboratory data packages to monitor the laboratory products including raw data, calibrations, tracking of QA limits, noncompliance procedures, training of analysts, method updating, as well as the quality process. Bench level staff must be interviewed as part of the audit to access their familiarity with the reference methods, laboratory SOPs and equipment for which they are responsible. The MPCA will only recognize primary accreditation authorities who perform on site audits of laboratories doing work for Minnesota programs. Audits must be performed at a minimum of every three years.