

Laboratory Data Review Checklist Guidance

The following is the Minnesota Pollution Control Agency's (MPCA) informal checklist that may be used to review data. The information follows the general format of the National Functional Guidelines which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work.

Preservation

The validity of the analytical results is based on sample condition and the holding time from sample collection to sample analysis. The requirements for sample preservation and holding times are found in the specific method or in the Code of Federal Regulations.

How to check: Review the Chain of Custody Form, the Sample Condition on Receipt Form, and the report narrative to determine if the samples were preserved and arrived at the laboratory in the proper condition. If there is no indication in the narrative, the Chain of Custody Record, or the Sample Condition on Receipt Form that there was a problem, the integrity of the samples can be assumed to be acceptable. If problems are noted, the integrity of the samples has been compromised and professional judgment must be used to evaluate the impact on the sample results.

Look at the date of sample collection on the Chain of Custody Form and compare this to the date of sample preparation and/or to the date of analysis. The number of days must be less than or equal to the required technical or Program holding times. If the samples were not analyzed within the technical holding time, the results may be impacted. For most analyses, professional judgment must be used. Detects should be qualify as estimated ("J") and non-detects as either estimated ("J") or unusable ("R").

Calibration

Calibration information may not be available for review by the data user. Calibration information can be checked by reviewing the report narrative. The calibration process consists of an initial calibration and continuing calibration verifications. Requirements for initial calibrations are specified in the cited method reference or by the MPCA Quality Control (QC) policy. Acceptance criteria are defined in the method or in the MPCA QC policy.

How to check: Look at the report narrative or any attached data discussing calibration. Look at the data report and check any flagged data that indicates there was a calibration issue. If you find these, consider the impact on the affected compounds in making decisions.

Blanks

There are numerous types of blanks that are analyzed by the laboratory. These include field blanks, trip blanks, and laboratory blanks. These blanks are analyzed to determine the existence and magnitude of contamination resulting from field, transportation, or laboratory activities. The method blank is used to determine the levels of contamination associated with the processing and analysis of samples. There must be one method blank reported for each batch of samples prepared by the laboratory for most analyses. Instrument blanks are analyzed to determine if there is any carry-over from the analysis of the previous sample. The concentration of each target analyte in the method blank must be less than the associated report level. If the method blank is contaminated, measures must be taken by the laboratory to eliminate the problem.

How to check: Look at the blank analysis results and the narrative. If any target analyte is detected above the report level in the blanks, sample results may need to be qualified to indicate the problem. All concentration levels for the affected target analyte which are less than 10 times the concentration in the blank should be qualified with a "B" to indicate that the sample results may contain a bias related to the blank contamination. Concentrations of the affected analyte which are above ten times the blank contamination will not need to be qualified. If a compound of concern on a site is flagged due to blank contamination, the result may be a false positive and care must be used in making site decisions.

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Surrogates

Surrogates are compounds that are not normally found in the environment. Surrogates are added to every sample and all batch QC samples to monitor laboratory performance normally for organic compound tests. Laboratories develop surrogate recovery limits based on recoveries from submitted samples. Acceptance criteria are defined in the method or in the MPCA QC policy.

How to check: Review the recoveries of the surrogates for each method where surrogates are added. Acceptance criteria must be listed in the report per the MPCA QC policy. If any recovery is outside of specifications, qualify the associated data as follows:

For any recovery greater than the upper acceptance limit, qualify the detected associated analytes as estimated ("J") and do not qualify any associated non-detects.

For any recovery below the lower acceptance limit, qualify any detected associated analyte as estimated ("J") and qualify any associated non-detects as either estimated ("J") or as unusable ("R").

Laboratory Control Sample/Laboratory Control Sample Duplicates (LCS/LCSDs)

Data for LCS and LCSD are generated to monitor accuracy and precision of the analytical process on a matrix-free sample.

How to check: Review the recoveries and Relative Percent Difference (RPD) for each method. Acceptance criteria must be listed in the report per the MPCA QC policy. If any recovery or RPD is outside of specifications, qualify the associated data as follows:

- a. For any recovery or RPD greater than the upper acceptance limit, qualify the detected associated analytes as estimated ("J") and do not qualify any associated non-detects.
- b. For any recovery below the lower acceptance limit, qualify any detected associated analyte as estimated ("J") and use professional judgment to qualify any associated non-detects.

Matrix Spike/Matrix Spike Duplicates (MS/MSDs) or Matrix Spike/Sample Duplicates (MS/DUPs)

Data for MS and MSD or MS and DUPs are generated to monitor accuracy and precision of the analytical process on the sample matrix. For organic analyses, MS/MSDs are prepared and analyzed at a five percent rate while, for inorganic analyses, MS/DUPs are prepared and analyzed at a 10 percent rate.

How to check: Review the recoveries and RPDs for each method. Acceptance criteria must be listed in the report per the MPCA QC policy. If any recovery or RPD is outside of specifications, qualify the associated data as follows:

- a. For any recovery or RPD greater than the upper acceptance limit, qualify the detected associated analytes as estimated ("J") and do not qualify any associated non-detects.
- b. For any recovery below the lower acceptance limit, qualify any detected associated analyte as estimated ("J") and use professional judgment to qualify any associated non-detects.

Method Detection Limits/Report Limits

Method Detection Limits (MDLs) and Report Limits (RLs) are determined initially and repeated at a minimum of every two years, or after a major change to the instrument conditions. The MDLs are determined per the procedure defined in 40 CFR 136, Appendix B. The RLs should be at least three times the MDLs. For each analyte, at least one of the calibration standards must correspond to a sample concentration at or below the reporting limit. Report limits depend on program needs. They can change as new information becomes available. Report limits are verified after each calibration and at least monthly. Contact the MPCA Project Manager for required report levels for each target analyte. Report limits and method detection limits can vary between laboratories performing the same test method and within the same laboratory from one year to another as new MDL studies are performed.

