Laboratory Data Review Instructions

April 1, 2025

The following is the Minnesota Pollution Control Agency's (MPCA) informal data review instructions which are usable for all programs. The instructions follow 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. Note that the qualifiers referenced in the document are the validator qualifiers found in the National Functional Guidelines and are intended as examples on how to qualify based on the sections 1-10. The qualifiers will not match laboratory data qualifiers and may be different from other internally generated validator qualifiers. Data validation qualifiers included in laboratory data validation packets/checklists are meant to identify the impact on the quality of the data when there are failures to meet method, project, program, or contractual requirements. If you have questions, please contact qa.questions.mpca@state.mn.us.

1. Preservation

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. The integrity of the samples is assumed to be acceptable if there is no indication in the narrative, Chain of Custody or Sample Condition on Receipt Form. Use professional judgement to evaluate the impact on the sample results if problems are noted.

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. Use professional judgment to evaluate the impact on the sample results. Detects should be qualified as estimated ("J") and non-detects as either estimated ("J") or unusable ("R").

Any problem with the condition of the sample, preservation of the sample, or not meeting holding times must be described in the report narrative and/or properly qualified on the results for the specific sample.

2. Calibration

Calibration details may not be available for review by the data user, but the laboratory must qualify the data associated with calibration failures. 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 and acceptance criteria are specified in the cited method reference, project quality documentation, or in the MPCA Laboratory Quality Control (QC) and Data Policy.

HOW TO CHECK: Look at the report narrative or any attached calibration data. Look at the data report and check any flagged data that indicates there was a calibration issue. Consider affected compounds and sample results when making decisions using impacted data.

3. Blanks

There are numerous types of blanks that are analyzed by the laboratory. Common types are method blanks, instrument blanks, field blanks, trip blanks and equipment blanks. Blanks are analyzed to determine the existence and magnitude of contamination resulting from field, transportation, or laboratory activities. Method blanks are used to determine the level 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 or be below project data quality objectives.

HOW TO CHECK: Look at the blank analysis results and the narrative. Sample results must be qualified if any target analyte is detected in the blank above the method specific requirements or the report level. All concentration levels for the affected target analyte, which are less than ten 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 above ten times the blank contamination will not need to be qualified with a validator qualifier. If a compound of concern on a site is flagged due to blank contamination care must be used when making site decisions with impacted data.

4. Surrogates

Surrogates are compounds added to every sample and batch QC sample to monitor laboratory performance and are typically used for organic analysis. Surrogates are compounds not expected to be found in the environment. Laboratories develop surrogate recovery limits based on recoveries from submitted samples. Acceptance criteria are defined in the method or in the MPCA Laboratory Quality Control and Data Policy.

HOW TO CHECK: Review the recoveries of the surrogates for each method where surrogates are analyzed. Acceptance criteria must be listed in the report per the MPCA Laboratory Quality Control and Data Policy. If any recovery is outside of specifications, qualify the associated data as follows:

a. 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.

b. 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").

5. Internal Standards

Most organic methods and some inorganic methods use internal standards for quantification which normalize fluctuations during preparation and analysis. High internal standard recoveries may cause a low bias to the analytes and low internal standard recoveries may bias results high. Internal standards are typically reported for PFAS analysis but are not reported for other methods. Acceptance criteria is method defined or found in the MPCA Guidance for per-and polyfluoroalkyl substances. Results connected to internal standard failures should not be used.

HOW TO CHECK: Internal Standards may be listed as surrogates in laboratory reports. Review the recoveries of the internal standards for each method where applicable. Acceptance criteria must be listed in the report per the MPCA Laboratory Quality Control and Data Policy. Results associated with failing recoveries should not be used or qualified as unusable ("R").

6. 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 Differences (RPDs) between the LCS and LCSD for each compound and each method. Acceptance criteria must be listed in the report per the MPCA Laboratory Quality Control and Data Policy. The laboratory should re-analyze the samples and associated QC if any recovery or RPD is outside of specifications. The data is qualified as follows if the second analysis confirms the original analysis:

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.

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

Data for MS/MSD or MS/DUPs are generated to monitor accuracy and precision of the analytical process in the sample matrix. The frequency of MS/MSD samples are documented in the project quality documentation or in the MPCA Laboratory Quality Control and Data Policy. Note: Labs do not always choose samples from your project to run as MS/MSDs. On the Chain-of-Custody (COC), you may supply additional sample volume/mass and request that the lab choose your sample for QC purposes.

HOW TO CHECK: Review the recoveries and RPDs for each method. Acceptance criteria must be listed in the report per the MPCA Laboratory Quality Control and Data 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.

8. Method Detection Limits/Report Limits

The report limits (RLs) should be at least three times the method detection limit (MDLs). Report limits depend on program needs and can change as new information becomes available. 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. Program-required reporting limits must be met for each analysis. If the reporting limits have been raised, the laboratory must provide an explanation in a footnote or the report narrative. In the case of multiple analyses, the laboratory must report the concentration for an analyte from the least dilute analysis with passing QC results.

9. Sample information

The laboratory must ensure that all field sample IDs are cross-referenced to laboratory sample IDs and that this information is clear to the data user.

HOW TO CHECK: Review that the sample specific information found on the work orders chain of custody matches what is found within either a sample summary or with each individual samples.

10. Lab reports

The laboratory must fully explain all issues with sample analysis and discuss any decisions made on the reported data. This can be accomplished with a case narrative that accompanies each analytical report or by qualifying the data and adding additional text for clarification. The explanation should provide clear and objective information for the data user to decide on the data useability.