December 16, 2015

Daniel P. Hautman, TSC Deputy Director
USEPA, OW, OGWDW, SRMD
Technical Support Center (MS140)
26 W. Martin Luther King Dr.
Cincinnati, OH 45268

RE: Reporting of Compliance Data With Qualifiers That Do Not Impact Data Usability

Dear Mr. Hautman:

The Environmental Laboratory Advisory Board (ELAB or Board) is a standing Federal Advisory Committee Act committee that advises the U.S. Environmental Protection Agency (EPA or Agency). The Board's Charter states that it is to provide consensus advice, information and recommendations on issues related to EPA measurement programs and facilitate operation and expansion of a national environmental accreditation program.

Several months ago, an issue was raised to the Board regarding the reporting of qualified data for compliance purposes. In some states, the mechanisms currently in place when reporting data either do not allow for data qualifiers to be included or those areas are required to be left blank. This creates a difficult situation for both data users and laboratories.

ELAB suggests the following recommendations that the Agency could consider for future implementation:

- Develop and implement a data-reporting document that would provide guidance to laboratories on:
  - Which types of "out of control" situations can be accepted and which ones would invalidate data from being reported and
  - Suggested data qualifiers or narratives that should accompany such data.

- Encourage consistency among state programs on the use of qualified data and the types of allowable qualifiers.

- Allow laboratories to accurately report the condition of data to data users.

These recommendations were based on research conducted by members of the Board in response to concerns raised by members of the laboratory community. Some of the resources that the Board found useful are attached to this letter. The Board believes that these could be useful to the Agency in developing a guidance for data usability/acceptability. The Board shares the Agency's concerns on the protection of public health in addressing data usability issues and qualifiers and thinks that this is best addressed by clear and consistent guidance to laboratories.

ELAB appreciates the opportunity to bring this matter to the Agency, and the Board looks forward to assisting with the development of a guidance should the Agency move forward with that recommendation. If any additional information is needed, please let us know.

Sincerely,

Patricia M. Carvajal
Chair, Environmental Laboratory Advisory Board

Attachments:

- Commonwealth of Pennsylvania Department of Environmental Protection Bureau of Laboratories – Instructions for Completion: Request to Report Qualified Drinking Water Sample Results Chemistry
- Florida Department of Environmental Protection Process for Assessing Data Usability

7/2015

pennsylvania
DEPARTMENT OF ENVIRONMENTAL PROTECTION

COMMONWEALTH OF PENNSYLVANIA
DEPARTMENT OF ENVIRONMENTAL PROTECTION
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Instructions for Completion
Request to Report Qualified Drinking Water Sample Results - Chemistry

The Department's Bureau of Safe Drinking Water ("BSDW") manages the Pennsylvania Drinking Water Information System ("PADWIS"). PADWIS is the electronic storage system for all drinking water compliance data generated for the Commonwealth of Pennsylvania. Environmental laboratories report drinking water compliance data to PADWIS through the Department's Drinking Water Electronic Reporting System ("DWELR"). The Department's Laboratory Accreditation Program ("LAP") manages and oversees the accreditation program for environmental laboratories generating SDWA compliance data for the Department. In accordance with the Federal and State Safe Drinking Water Acts ("SDWA"), the Department's regulations mandate that all SDWA compliance data be generated by PA-DEP accredited laboratories and in accordance with all regulatory, method, and permit requirements.

It is expected that SDWA compliance results reported to DWELR meet all regulatory and method acceptance criteria, and that reported data are not associated with sample collection, preservation, analysis, or quality control ("QC") failures. In addition, DWELR and PADWIS do not have a mechanism to accept data qualifiers. The Department understands that in select and specific situations qualified data might be considered valid for compliance purposes, and the Department has developed a mechanism for laboratories to request to report qualified data to PADWIS. **This system should be used as a LAST RESORT and only when the results could be considered valid.** Laboratories cannot use this system to avoid corrective action, instrument maintenance, re-calibration, re-analysis, re-collection, etc.

Before You Submit Your Request:

The Department will only approve data that can be legally defended as valid SDWA compliance results. The laboratory supervisor should review all requests before submission to the Department and make a good-faith effort to determine if the results might be considered valid and must provide a justification to the Department explaining why he or she believes the data should be accepted by the Department. If you cannot provide a valid justification for why the data should be accepted, DO NOT submit the request.

The Department will **NOT** accept any data associated with any of the following situations:

- Unacceptable Initial Calibration
- Sample analysis or preparation performed out-of-hold
- Samples collected in inappropriate containers or with unacceptable preservation
- Failure of QC with high bias and sample results above the minimum quantitation limit
- Failure of QC with low bias (including surrogates) and sample results below the MCL or action level
- Sample analysis performed by a laboratory that does not hold accreditation with the PA-DEP

For Matrix Spike Failures (assuming acceptable results for all other QC measures):

- Inorganic Testing: The laboratory must re-prepare the matrix spike sample and analyze the re-prepared sample. If the second analysis indicates matrix interference then the laboratory may submit the Request Form.
- Organic Testing: The laboratory must verify that any surrogates and/or internal standards are within method acceptance criteria for the particular sample and matrix spike. If the surrogates, internal standards, and all other sample acceptance criteria are acceptable, then the laboratory may submit the Request Form.
- DO NOT submit the form requesting to report matrix interference if you have not performed the steps above.

Completion of the Request Form:

A laboratory seeking to obtain permission from the Department to report qualified SDWA compliance results must complete the Request Form. Complete the Request Form as follows:

- Print or type all information.
- Complete ALL items on the form, including the pH and residual chlorine measurements. Even if the samples do not require pH preservation or removal of residual chlorine, the Department still requires this information to determine if the sample might be considered valid.
- Do not include ambiguous information, such as requesting to report results as "<RL". The laboratory must provide an actual numerical result and units of measurement, such as "<0.005 mg/L".
- Use one form per batch of samples analyzed by the same method that demonstrate the same QC failure type.
- For requests that encompass more than one analyte and/or more than one sample, provide the specific information for each analyte (analyte name and results, including units) that are referenced to the specific laboratory sample ID number on a separate sheet.
- An approved laboratory supervisor or QA Officer must sign and date the form.

Required Attachments:

In addition to the Request Form, the laboratory must provide the following attachments:

- Sample and QC Summary: The laboratory must provide a summary of the sample and batch specific QC information for each analyte and QC measure associated with the analytical test performed. The information provided by the laboratory should not be copies of the analytical data (instrument print-outs). The information that must be submitted should be a short summary, possibly in a table form that contains at least the following information, depending on the required QC:
  - Initial Calibration Range, including concentration of the calibration standards.
  - True Value and % recovery of the QC measure (including but not limited to LCS, MRL check, CCVs, ICVs, etc.)
  - Performance of the results of the Matrix Spikes, Duplicates, Method Blank(s), Surrogate(s), and Internal Standard(s)
- Copy of the Chain of Custody or other sample receiving documentation utilized by the laboratory to verify and document sample collection and receipt information. If samples were received as a subcontracted work agreement, the receiving laboratory must indicate to which laboratory the samples were originally submitted.
- Investigations into the Failure: Describe the investigations undertaken by the laboratory to determine the cause of the failure, also known as a root cause investigation.
- Corrective Actions Resulting from the Failure: Explain the corrective actions, if any, that are or will be implemented to avoid this failure in the future.
- Justification: Provide an explanation or justification as to **WHY** you believe the data is valid and the Department should accept the qualified sample results for drinking water compliance. This justification must directly relate to the validity of the data in question and cannot relate to a monitoring period, inability to recollect a sample, etc.
- Other Comments or Information: Provide any additional comments or information that you feel the Department will need to make the decision.

NOTE: Please attempt to limit the amount of documentation that you provide to the Department. The required attachments should not result in more than one page per document. If your submission is longer than 5-10 pages please contact your accreditation officer to ensure that you are providing the correct information.

Submission of the Request Form:

The Request Form and attachments must be provided to the general e-mail account for the Department's Laboratory Accreditation Program at eplabaccredit@pa.gov. Please submit one request form and required attachments in a single e-mail submission.

Use the following format in the e-mail subject line for single sample submissions:

DEP Laboratory ID#, Method Name, Laboratory's Sample ID#
For example: 68-01234, EPA 505, 546899

Use the following format in the e-mail subject line for multiple sample submissions:

DEP Laboratory ID#, Method Name, Date of Analysis (# of Samples in the Request)
For example: 68-01234, EPA 505, 5-15-2015 (12)

Evaluation of the Request Form:

The LAP will review the submission and coordinate with the BSDW to make a final decision. The LAP will notify you if the submission requires additional information, documentation, or correction. Failure to provide the requested documentation or submission of inaccurate information will delay the processing of the request. The LAP will notify the laboratory of the Department's final decision via e-mail to the e-mail address(s) that are included on the original submission. Questions regarding completion and submission of the Request Form should be directed to the laboratory's accreditation officer or to the Laboratory Accreditation Program at (717) 346-7200 or eplabaccredit@pa.gov.

DO NOT report any SDWA compliance data associated with unacceptable sample collection, handling, quality control, etc. without approval from the Department.

Process for Assessing Data Usability

DEP-EA 001/07

Florida Department of Environmental Protection
2600 Blair Stone Road
Tallahassee, FL 32399-2400

Bureau of Standards and Special Projects
Environmental Assessment Section

March 31, 2008

Table of Contents

Process for Assessing Data Usability
DEP-EA 001/07

1. **Introduction** - The intent of this document is to outline the process to be used by the Department when evaluating Data Quality Indicators (DQI) and determining the usability of analytical data.

1.1. **The Data Assessment Process** - Determining if data are usable for a particular purpose is a complex task, requiring a reasonable and balanced evaluation of many factors. The procedural components of the usability assessment must be performed by auditors with sufficient scientific expertise in environmental data verification and validation, and include, but are not limited to: Understanding the purpose for auditing the data (project or program data quality objectives provide the context for the audit);

- Identifying the set of data to be audited, the types of analytes or parameters in the data set and the reported values (e.g., concentration) for the analytical results for all components;
- Determining the relationship between each analytical result, the associated decision or action level (e.g., water quality standard or clean up target) and the laboratory's quantitation limit;
- Evaluating the documented calibration, quality control and other supporting data against designated Data Quality Indicators;
- Establishing the pattern, frequency, and magnitude of any failures or other deficiencies associated with the results;
- Determining the extent to which the audited data set fulfills the Data Quality Objectives of the project or Program;
- Evaluating corroborative data (e.g., performance tests, data from other laboratories);
- Providing usability recommendations to the Program data users.

1.2. **Evaluating Data Quality Indicator Failures** - Exceeding the acceptance criteria for one or more Data Quality Indicators does not necessarily mean the data are unusable. The factors mentioned in 1.1 above must be systematically evaluated before a usability decision can be made

1.2.1. The purpose for which the analytical data were collected can vary widely, and may include such diverse activities as: initial screening or scoping studies, permit compliance monitoring, assessing waters for Total Maximum Daily Load development or determining whether a permitted waste facility has met "clean closure" site contamination assessment requirements.

1.2.2. A Data Quality Indicator failure that is acceptable for a screening study may not be acceptable for declaring a site free from contamination.

1.2.3. In practical terms, it is not possible in this document to discuss each potential scenario that might be associated with a data usability recommendation for a particular purpose. Therefore, the factors affecting this decision (outlined in 1.1 and 1.2, above) and the thought process applied to the data usability assessment are addressed by the general principles and examples listed below.

2. **Data Quality Indicators (DQI)** - The following Data Quality Indicators (Sections 3.0 – 16) are targets used in the context of assessing data usability. Depending on a given situation, not all DQIs are applicable to the specific assessment project.

> *Example: To be considered usable, a data submittal to DEP consisting only of high sample concentrations (with no values near the PQL) need not be associated with records demonstrating evaluation or verification of the PQL.*

2.1. The application of DQIs to sample results presumes the random occurrence of non-routine criteria failures to be an expected fact associated with all analyses. The frequency of DQI failures are considered as part of the overall data usability assessment in order to determine those instances where routine or systematic failures indicate a significant data usability problem.

2.2. Use of DQIs with Other Published Criteria - Except as discussed otherwise in this document, sample results are evaluated for usability based on the procedural requirements and performance criteria established by the reported analytical methods, applicable project data quality objectives, applicable regulations, applicable NELAC Quality Systems standards and applicable DEP SOPs for field and laboratory activities (DEP-SOP-001/01 and DEP-SOP-002/01).

2.3. Use of Records to Assess Data Usability - As applicable to the data usability assessment process, any record associated with a reported sample result or set of sample results may be audited, per 62-160.240 & .340, FAC. Both original ("raw") and reduced or summarized versions of data records are inspected in order to determine acceptance of the procedures and performance criteria that are used to generate and evaluate the sample data and associated quality control activities.

2.4. The presence or absence of critical, archival records that support the sample results is considered when determining whether sufficient documentation is available to assess the usability of the data.

3. **Laboratory Control Sample or Spike (LCS)** –In order of preference, LCS data are evaluated rather than matrix spike data (MS), if both types of data are generated for the associated sample preparation batch. However, the MS data are also used to evaluate the recoveries of the analytes for the parent sample and may affect the usability of the parent sample result.

3.1. General Requirements

3.1.1. An LCS result is linked or associated with all applicable reported sample results for the same preparation batch or analytical sequence of 20 samples or less.

3.1.2. The LCS contains all of the analytes of interest for the method or project, or the mix of analytes in the LCS is rotated at a routine frequency as suggested in the NELAC Quality Systems standard in Appendix D.

3.1.3. If a preparation step (such as digestion or extraction) is required for the analysis method, the LCS is prepared identically with and at the same time as the associated samples in the preparation batch.

3.1.4. The LCS is analyzed or reanalyzed within a verified calibration bracket (analytical sequence see Section 8.2.2 for the definition).

3.1.5. All reanalyses are documented with a valid explanation of the systematic error that prompted the reanalyses.

3.1.6. The concentration of the LCS is analyzed to be within the calibration range for the instrument as established by the concentrations of the standards used for the initial calibration and continuing calibration verifications.

3.1.7. Where applicable, the LCS is prepared at the method-specified concentration or at a concentration appropriate for the project data quality objective.

3.2. **Evaluation of LCS Recovery** - Where applicable, the control limits for the LCS are established by the laboratory for a specified matrix at ± 3X the standard deviation of the mean recovery of the cumulative LCS analyses at a specified concentration. Alternatively, the recovery control limits are established by linear regression for a range of concentrations using cumulative recovery data. The mean recovery is calculated or regression analysis is applied after outliers are eliminated from the recovery data set using any appropriate statistical test for outliers and after outliers due to known systematic errors are also censored from the data set. The mean recovery is recalculated or linear regression is reanalyzed if trend monitoring of individual LCS recoveries indicates that a systematic bias has developed and corrective action is needed.

3.2.1. Alternatively, control limits are established by the laboratory using other technically justifiable and scientifically sound procedures in accordance with project data quality objectives.

3.2.2. Where applicable, criteria established by rule, reported method or project data quality objectives are used to evaluate the LCS recoveries.

3.2.3. If no other criteria are applicable, the following control limits are used to evaluate the LCS recoveries:

80% - 120% (water) for Nitrate, Nitrite, Nitrate + Nitrite, Ammonium, TKN, Total Phosphorus, Orthophosphate

85% - 115% (water) for Metals

Laboratory Control Limits for:

All Organic analytes for the LCS in any matrix

All other analytes amenable to spiking for the LCS in water

All analytes amenable to spiking for the LCS in non-aqueous matrices

3.2.4. When applicable, sample results reported with failed LCS recoveries are documented with a valid explanation of the systematic error that occurred.

3.2.5. Analytes or methods technically not amenable to spiking for recovery determinations are not evaluated.

4. **Matrix Spikes (MS)** - Method-specified criteria are used to evaluate the MS, where applicable. If no LCS data is available for the sample preparation batch or analytical sequence being evaluated, the MS data is used to evaluate method control for all of the associated samples in the batch or sequence.

4.1. General Requirements

4.1.1. The MS contains all of the analytes of interest for the method or project, or the mix of analytes in the MS is rotated at a routine frequency as suggested in the NELAC Quality Systems standard in Appendix D.

4.1.2. If a preparation step (such as digestion or extraction) is required for the analysis method, the MS is prepared identically with and at the same time as the associated samples in the preparation batch.

4.1.3. The MS is analyzed or reanalyzed within a verified calibration bracket (analytical sequence).

4.1.4. All reanalyses are documented with a valid explanation of the systematic error that prompted the reanalyses.

4.1.5. The concentration of the MS is analyzed to be within the calibration range for the instrument as established by the concentrations of the standards used for the initial calibration and continuing calibration verifications.

4.1.6. Where applicable, the MS is spiked at the method-specified concentration or at a concentration appropriate for the project data quality objective.

4.2. Evaluation of MS Recovery

4.2.1. The MS spike concentration is compared with the un-spiked, parent-sample concentration. The suitability of the spike concentration is evaluated on a case-by-case basis according to method specifications or project data quality objectives.

4.2.2. MS recovery data is evaluated if specified by applicable data quality objectives, but the recovery of the MS is only associated with the parent sample, unless no LCS is associated with the preparation batch or analytical sequence of 20 samples or less. In this case, the MS recovery is associated with all of the samples in the preparation batch or analytical sequence of 20 samples or less for evaluation of sample data usability.

4.2.3. Where applicable, the control limits for the MS are established by the laboratory for a specified matrix at ± 3X the standard deviation of the mean recovery of the cumulative MS analyses for the associated matrix at a specified concentration. Alternatively, the recovery control limits are established by linear regression for a range of concentrations for the associated matrix using cumulative recovery data. The mean recovery is calculated or regression analysis is applied after outliers are eliminated from the recovery data set using any appropriate statistical test for outliers and after outliers due to known systematic errors are also censored from the data set. The mean recovery is recalculated or linear regression is reanalyzed if trend monitoring of individual MS recoveries indicates that a systematic bias has developed and corrective action is needed.

4.2.4. Alternatively, control limits are established by the laboratory using other technically justifiable and scientifically sound procedures in accordance with project data quality objectives.

4.2.5. If the MS is used to evaluate all associated batch samples, the applicable LCS criteria are used to evaluate the MS recovery.

4.2.6. Where applicable, criteria established by rule, reported method or project data quality objectives are used to evaluate the MS recoveries.

4.2.7. If no other criteria are applicable, the following control limits are used to evaluate the MS recoveries:

80% - 120% (water) for Nitrate, Nitrite, Nitrate + Nitrite, Ammonium, TKN, Total Phosphorus, Orthophosphate

70% - 130% (water) for Metals

Laboratory Control Limits

All Organic analytes at any concentration

All other analytes amenable to spiking at any concentration

All analytes in non-aqueous matrices

4.2.8. When applicable, sample results reported with failed MS recoveries are documented with a valid explanation of the systematic error that occurred.

4.2.9. Analytes or methods technically not amenable to spiking for recovery determinations are not evaluated.

5. **Surrogate Spikes** - Surrogate spikes are evaluated according to method-specified requirements and the NELAC Quality Systems standards (Chapter 5 and Appendix D).

5.1. The concentration of the surrogate spike is analyzed to be within the initial calibration range established for the instrument.

5.2. When applicable, sample results reported with failed surrogate spike recoveries are documented with a valid explanation of the systematic error that occurred.

5.3. Analytes or methods technically not amenable to spiking for recovery determinations are not evaluated.

6. **LCS Duplicates or Replicates (LCSD) and Matrix Spike Duplicates (MSD)** - In order of preference, LCSD data are evaluated rather than MSD data, if both types of data are generated for the associated samples. However, the MSD data are also used to evaluate the precision of the analytes for the parent sample and may affect the usability of the parent sample result.

6.1. General Requirements

6.1.1. When applicable, replicate rather than duplicate LCS/MS data are evaluated for precision control.

6.1.2. An LCSD/MSD result is linked or associated with all applicable reported sample results for the same preparation batch or analytical sequence of 20 samples or less.

6.1.3. The LCSD/MSD contains all of the analytes of interest for the method or project, or the mix of analytes in the LCSD is rotated at a routine frequency as suggested in the NELAC Quality Systems standard in Appendix D.

6.1.4. If a preparation step (such as digestion or extraction) is required for the analysis method, the LCSD/MSD is prepared identically with and at the same time as the associated samples in the preparation batch.

6.1.5. The LCSD/MSD is analyzed or reanalyzed within a verified calibration bracket (analytical sequence).

6.1.6. All reanalyses are documented with a valid explanation of the systematic error that prompted the reanalyses.

6.1.7. The concentration of the LCSD/MSD is analyzed to be within the calibration range for the instrument as established by the concentrations of the standards used for the initial calibration and continuing calibration verifications.

6.1.8. Where applicable to the evaluation, the MSD spike concentration is compared with the un-spiked, parent-sample concentration. The suitability of the spike concentration is evaluated on a case-by-case basis according to method specifications or project data quality objectives.

6.2. Evaluation of Duplicates

6.2.1. Where applicable, the control limits for the LCSD/MSD are established by the laboratory for a specified matrix at ≤3X the standard deviation of the mean precision (absolute or relative) of the cumulative LCSD analyses for the associated matrix at a specified concentration. Alternatively, the precision control limits are established by linear regression for a range of concentrations for the associated matrix using cumulative precision data. The mean precision is calculated or regression analysis is applied after outliers are eliminated from the precision data set using any appropriate

6.2.2. Alternatively, control limits are established by the laboratory using other technically justifiable and scientifically sound procedures in accordance with project data quality objectives.

6.2.3. Where applicable, criteria established by rule, reported method or project data quality objectives are used to evaluate the LCSD/MSD precision.

6.2.4. If no other criteria are applicable, precision data for the LCSD/MSD are evaluated using the laboratory control limits, with a target precision of ≤20% RPD or RSD for water samples and ≤40% RPD or RSD for non-aqueous and solid-matrix samples.

6.2.5. When applicable, sample results reported with failed LCSD/MSD precision are documented with a valid explanation of the systematic error that occurred.

6.2.6. Analytes or methods technically not amenable to precision determinations are not evaluated.

7. Sample Duplicates (SD)

7.1. General Requirements

7.1.1. Sample matrix duplicates are evaluated for the analytes present in the sample if no LCSD/MSD data is available or if required by the method or project data quality objectives.

7.1.2. When applicable, replicate rather than duplicate sample data are evaluated for precision control.

7.1.3. If a preparation step (such as digestion or extraction) is required for the analysis method, the SD is prepared identically with and at the same time as the associated samples in the preparation batch.

7.1.4. If no LCSD/MSD is available for evaluation, an SD result is linked or associated with all applicable reported sample results for the same preparation batch or analytical sequence of 20 samples or less.

7.1.5. The SD is analyzed or reanalyzed within a verified calibration bracket (analytical sequence).

7.1.6. All reanalyses are documented with a valid explanation of the systematic error that prompted the reanalyses.

7.1.7. The concentration of the SD is analyzed to be within the calibration range for the instrument as established by the concentrations of the standards used for the initial calibration and continuing calibration verifications.

7.2. Evaluation of Sample Duplicates

7.2.1. Where applicable, the control limits for the SD are established by the laboratory for a specified matrix at ≤3X the standard deviation of the mean precision (absolute or relative) of the cumulative SD analyses for the associated matrix at a specified concentration. Alternatively, the precision control limits are established by linear regression for a range of concentrations for the associated matrix using cumulative precision data. The mean precision is calculated or regression analysis is applied after outliers are eliminated from the precision data set using any appropriate statistical test for outliers and after outliers due to known systematic errors are also censored from the data set. The mean precision is recalculated or linear regression is reanalyzed if trend

7.2.2. Alternatively, control limits are established by the laboratory using other technically justifiable and scientifically sound procedures in accordance with project data quality objectives.

7.2.3. Where applicable, criteria established by rule, reported method or project data quality objectives are used to evaluate the SD precision.

7.2.4. If no other criteria are applicable, precision data for the SD are evaluated using the laboratory control limits, with a target precision of ≤20% RPD or RSD for water samples and ≤40% RPD or RSD for non-aqueous and solid-matrix samples.

7.2.5. When applicable, sample results reported with failed SD precision are documented with a valid explanation of the systematic error that occurred.

7.2.6. Analytes or methods technically not amenable to precision determinations are not evaluated.

8. **Calibrations -** Except as discussed elsewhere in this document, all calibrations are evaluated according to method-specified requirements and applicable NELAC Quality Systems standards.

8.1. Initial Calibration

8.1.1. The number of standard concentrations specified by the method or project data quality objectives is used to perform the initial calibration of the instrument or technique.

8.1.2. Where applicable, the concentration values specified by the method or project data quality objectives are used to perform the initial calibration of the instrument or technique.

8.1.3. If not specified by the method or project objectives and when applicable to the analytical technology, at least two standard concentrations and a blank are used for a linear calibration curve.

8.1.4. The acceptance of the initial calibration is evaluated using the method-specified criteria or the criteria specified by the project data quality objectives.

8.1.5. If not specified by the method or project data quality objectives and when applicable to the analytical technology:

- The correlation coefficient for a regression is ≥0.995 when applied to the linear calibration curve.
- Higher-order calibration curve regressions have a coefficient of determination of ≥0.99, using 6 calibration points for a second order curve and 7 calibration points for a third order curve. The calibration points used for the curve include a result for the calibration blank.

8.1.6. Where applicable, the initial calibration range is extended by an accepted calibration verification.

8.1.7. Unless allowed by the method or project data quality objectives, applicable sample results whose analyzed concentrations fall outside of the calibration range established by the standard concentrations used for the initial calibration (excluding the calibration blank) and calibration verifications are not usable without further evaluation or qualification.

8.2. Calibration Verification (CV)

8.2.1. At least one verification standard is from a second source as indicated in the applicable NELAC Quality Systems standard for initial calibrations.

8.2.2. An acceptable calibration verification is analyzed at the beginning and end of the analytical batch (verified calibration bracket) or as otherwise required by the applicable NELAC Quality Systems standard for continuing calibration verifications.

8.2.3. The analytical batch is determined as defined by the applicable NELAC Quality Systems standard.

8.2.4. As applicable or when specified by rule or project data quality objectives, the calibration verification standard is analyzed at the method-specified frequency.

8.2.5. As applicable or when specified by rule or project data quality objectives, the calibration verification result is evaluated using the method-specified criteria.

8.2.6. In the absence of acceptance criteria specified by rule, project data quality objectives or method, and where applicable, the control limits for the CV are established by the laboratory for a specified matrix at ± 3X the standard deviation of the mean recovery of the cumulative CV analyses at a specified concentration. For this purpose, recovery is defined as the percent of expected concentration analyzed for the CV standard. The mean recovery is calculated after outliers are eliminated from the recovery data set using any appropriate statistical test for outliers and after outliers due to known systematic errors are also censored from the data set. The mean recovery is recalculated if trend monitoring of individual CV recoveries indicates that a systematic bias has developed and corrective action is needed.

8.2.7. Alternatively, control limits are established by the laboratory using other technically justifiable and scientifically sound procedures in accordance with project data quality objectives.

8.2.8. If a continuing calibration verification did not meet the acceptance criterion, but is immediately reanalyzed with acceptable results, the samples analyzed before the failed verification result are valid, provided that the verification previous to the failed verification and following the reanalyzed verification are also acceptable and comprise a verified calibration bracket or analytical batch.

8.2.9. Sample results that are not analyzed within a verified analytical batch or calibration bracket are reanalyzed or qualified as estimated values.

8.2.10. Alternatively, for purposes of assessing the usability of the sample data where verifications have failed and where the reported sample values are relevant to the regulatory action level, compliance limit or other project data quality objective, the failed continuing calibration verifications are evaluated for potential high or low bias according to the applicable NELAC Quality Systems standards for calibration verifications.

9. **Method Blanks or Other Analytical Blanks -** All method blanks and other types of analytical blanks use to control analytical contamination are evaluated for the presence of the analytes of interest for the project or method.

9.1. A blank result is linked or associated with all applicable reported sample results for the same preparation batch or analytical sequence of 20 samples or less.

9.2. If a preparation step (such as digestion or extraction) is required for the analysis method, at least one blank is prepared identically with and at the same time as the associated samples in the preparation batch.

9.3. The blank is analyzed or reanalyzed within a verified calibration bracket (analytical sequence).

9.4. All reanalyses are documented with a valid explanation of the systematic error that prompted the reanalyses.

9.5. Unless specified otherwise by rule, an applicable analytical method or project data quality objectives, it is expected that the concentration of analytes detected in the blank (above the reported MDL) will be less than 10% of the concentration in the individual samples of the associated preparation batch or analytical sequence (for the affected analytes).

9.6. Where samples are diluted for analysis, the evaluation of the blank results is considered with respect to the dilution factors associated with the samples.

9.7. BOD method blanks are evaluated against the method criterion.

9.8. No colonies are reported in bacteriological method blanks.

10. **Field Quality Control Blanks (Trip Blanks, Field Blanks or Equipment Blanks)** - Field QC Blanks associated with the samples for a specific sampling event are evaluated for contamination as indicated by the presence of the analytes of interest for the project or method.

10.1. If a preparation step (such as digestion or extraction) is required for the analysis method, the blank is prepared identically as the associated samples in the preparation batch.

10.2. The blank is analyzed or reanalyzed within a verified calibration bracket (analytical sequence).

10.3. Unless specified otherwise by rule, an applicable analytical method or project data quality objectives, it is expected that the concentration of analytes detected in the blank (above the reported MDL) will be less than 10% of the concentration in the associated field sample (for the affected analytes).

10.4. Where samples are diluted for analysis, the evaluation of the blank results is considered with respect to the dilution factors associated with the samples.

11. **Holding Times** - Holding times as specified in the tables in FS 1000 of the DEP SOPs are met.

11.1. For those analytes where there is no separate holding time specified for the extract, digestate or other processed sample, the holding period ends when the sample processing begins.

11.2. Holding times that are specified in “hours” are met if the sample processing (e.g., extraction, digestion, filtration, etc) or analysis, as applicable, begins within the last hour of the specified holding time, accounting for the time zone in which the sample was collected.

> *Example: For a sample with a 24-hour holding time that was collected at 14:15 on May 20, 2007, the sample is within holding time if the sample is processed before 15:15 on May 21, 2007.*

11.3. Holding times that are specified in “days” are met if the sample processing begins before 24:00 on the final day, accounting for the time zone in which the sample was collected.

11.4. Clarifications

11.4.1. For microbiology, the holding time is evaluated as the duration between the sample collection date and time and the date and time of the placement of the processed sample into or on the applicable growth medium.

11.4.2. For BOD or CBOD, the holding time is evaluated as the duration between the sample collection date and time and the date and time of the initial DO measurement for the test.

11.4.3. For toxicity testing, the holding time is evaluated as the duration between the sample collection date and time and the date and time of introduction of the last individual test organism into a test sample.

12. **Quality Control Check Samples** – The following criteria are used to evaluate specific quality control check samples for the indicated test.

12.1. BOD Analyses

12.1.1. For each preparation batch of 20 samples or less, glucose-glutamic acid (GGA) samples are analyzed to obtain values within the method-specified acceptance control limits.

12.1.2. Alternatively, per approved revisions to SM 5210 B, other compounds are used to control the method, when applicable to a specific wastewater effluent.

12.1.3. In order of preference, replicate check sample data are evaluated rather than sample replicate data, if both types of data are generated for the associated samples.

12.1.4. Precision data for the replicate check sample analyses are evaluated using the laboratory control limits, with a target precision of ≤20% RPD or RSD expected.

12.1.5. Where applicable, the control limits for the replicate check sample analyses are established by the laboratory at ≤3X the standard deviation of the mean precision (absolute or relative) of the cumulative check sample analyses. The mean precision is calculated after outliers are eliminated from the precision data set using any appropriate statistical test for outliers and after outliers due to known systematic errors are also censored from the data set. The mean precision is recalculated if trend monitoring of individual check sample results indicates that a systematic error has occurred and corrective action is needed.

12.1.6. Alternatively, control limits are established by the laboratory using other technically justifiable and scientifically sound procedures in accordance with project data quality objectives.

12.1.7. When applicable, sample results reported with failed check sample precision are documented with a valid explanation of the systematic error that occurred.

12.2. Chlorophyll Analyses

12.2.1. For each preparation batch of 20 samples or less, or at least once every three months, a chlorophyll extract solution in acetone is analyzed to evaluate recovery at the concentration for the extract.

12.2.2. A vendor-assayed source of chlorophyll is used to prepare the check sample extract.

12.2.3. The concentration of the check sample extract is such that when extrapolated to the volume of a nominal whole-water concentration, the extract appropriately represents the estimated reporting limit for the whole water sample.

12.2.4. The check-sample results at the extract concentration are evaluated using control limits established by the laboratory.

12.2.5. The control limits for the extract check-sample solution are established by the laboratory at ± 3X the standard deviation of the mean result of the cumulative check-sample analyses expressed as a percentage of the expected value of the check-sample concentration. The mean result is calculated after outliers are eliminated from the check-sample data set using any appropriate statistical test for outliers and after outliers due to known systematic errors are also censored from the data set. The mean result is recalculated if trend monitoring of individual check-sample results indicates that a systematic bias has developed and corrective action is needed.

12.2.6. Alternatively, control limits are established by the laboratory using other technically justifiable and scientifically sound procedures in accordance with project data quality objectives.

12.3. **Matrix-Specific Evaluation for Known or Suspected Interferences** - An example of typical interferences of concern to DEP is the analyses for trace metal concentrations in seawater.

12.3.1. When applicable to the project data quality objectives and when technically feasible, known or suspected interferences in the matrix specific to the project analytes and samples are evaluated using quality control (QC) samples of known matrix composition and analyte concentration.

12.3.2. The QC sample result is linked or associated with all applicable reported field sample results for the same preparation batch or analytical sequence of 20 samples or less.

12.3.3. The composition of the matrix for the QC sample is controlled for the analytes and interferences of interest and closely matches or approximates the matrix of the associated field samples.

12.3.4. A naturally derived or artificially formulated matrix is used to prepare the QC sample.

12.3.5. The QC sample is prepared by the analyzing laboratory using a second source standard for the analyte of interest. The second-source standard is selected as indicated in the NELAC Quality Systems standard for initial calibrations.

12.3.6. Alternatively, the QC sample is obtained from an external source such as a commercial vendor of QC-check samples or Standard Reference Materials.

12.3.7. Where applicable, the QC sample is prepared at the method-specified concentration or at a concentration selected according to project data quality objectives.

12.3.8. Where applicable, the concentration of the QC sample is evaluated against any data quality objectives for minimum quantitation level applicable to the data use and does not exceed 2X the minimum quantitation level.

12.3.9. If a preparation step (such as digestion or extraction) is required for the analysis method, the QC sample is prepared identically with and at the same time as the associated samples in the preparation batch.

12.3.10. The QC sample is analyzed or reanalyzed within a verified calibration bracket (analytical sequence).

12.3.11. All reanalyses are documented with a valid explanation of the systematic error that prompted the reanalyses.

12.3.12. The concentration of the QC sample is analyzed to be within the calibration range for the instrument as established by the concentrations of the standards used for the initial calibration and continuing calibration verifications.

12.3.13. As applicable, the recovery for the QC sample is evaluated using method-specified criteria, vendor-specified criteria, or control limits established by the laboratory.

12.3.14. Where applicable, the control limits for the QC sample are established by the laboratory for a specified matrix at ± 3X the standard deviation of the mean recovery of the cumulative QC sample analyses at a specified concentration. Alternatively, the recovery control limits are established by linear regression for a range of concentrations using cumulative recovery data. The mean recovery is calculated or regression analysis is applied after outliers are eliminated from the recovery data set using any appropriate statistical test for outliers and after outliers due to known systematic errors are also

censored from the data set. The mean recovery is recalculated or linear regression is reanalyzed if trend monitoring of individual QC sample recoveries indicates that a systematic bias has developed and corrective action is needed.

12.3.15. Alternatively, control limits are established by the laboratory using other technically justifiable and scientifically sound procedures in accordance with project data quality objectives.

12.3.16. When applicable, sample results reported with failed QC sample recoveries are documented with a valid explanation of the systematic error that occurred.

13. Sample Preservation Checks

13.1. The field records demonstrate positive indication that the samples are properly preserved (including thermal preservation).

13.2. The laboratory records demonstrate that proper thermal preservation was checked upon receipt at the laboratory, per the instructions in the NELAC Quality Systems standards.

13.3. The laboratory records demonstrate that samples that have been collected on the same day and hand-delivered are received at the laboratory in ice if the temperature check fails the applicable rule or method requirement.

13.4. The laboratory records demonstrate positive indication that the samples are checked for proper chemical preservation prior to or during sample preparation or analysis.

14. Evaluation of the Reported MDL

14.1. If no analyte is detected in the evaluated sample, the assessment of sample data usability includes establishing that the laboratory determined, evaluated and verified the reported MDL for the analyte according to the requirements in the applicable NELAC Quality Systems standards, regulatory requirement or reported method.

14.2. The MDL determination, evaluation and verification are considered when establishing the usability of the sample data where the reported MDL is relevant to the regulatory action level, compliance limit or other project data quality objective.

14.3. For the purposes of data usability evaluation, the DEP-defined MDL is equivalent to the NELAC-defined LOD.

15. Evaluation of the Reported PQL

15.1. If the concentration of the evaluated sample is below the concentration value of the reported PQL or is below the concentration value of the lowest initial calibration standard or continuing calibration verification standard associated with the sample, the assessment of sample data usability includes establishing that the laboratory determined, evaluated and verified the reported PQL for the analyte according to the requirements in the applicable NELAC Quality Systems standards.

15.2. The PQL determination, evaluation and verification are considered when establishing the usability of the sample data where the reported PQL is relevant to the regulatory action level, compliance limit or other project data quality objective.

15.3. For the purposes of data usability evaluation, the DEP-defined PQL is equivalent to the NELAC-defined LOQ.

16. Evaluation of Reversals (Parts vs. Whole Comparison)

16.1. Where applicable, sample results are evaluated to determine if the sum of reported parts or fractions for the associated sample analyte results exceed 120% of the corresponding reported or calculated whole.

16.2. Evaluation of reversals with respect to sample data usability is assessed on a case-by-case basis with more extensive validation of the specifically affected analyses conducted as needed.

Examples of reversals include the following:

Parameter Part	Parameter Whole
Total ammonia	TKN
Orthophosphate	Total phosphorus
Nitrate	Measured Total nitrite/nitrate
Nitrite	Measured Total nitrite/nitrate
Sum of nitrite and nitrate	Measured Total nitrite/nitrate
Sum of nitrite, nitrate and TKN	Measured Total nitrogen
Sum of measured total nitrite/nitrate and TKN	Measured Total nitrogen
Filtered sample results (e.g., dissolved metals)	Unfiltered sample results (e.g., total metals)
Methyl mercury	Total mercury
DOC	TOC

17. **General Principles of Data Quality Assessment** - The following principles will be used when evaluating sample data for data usability determinations using specified Data Quality Indicators. The guidelines discussed in this section are only applicable if specific data quality assessment directives have not been provided in any other Department Rule or reporting format. The examples given below are illustrative and simplistic in nature.

17.1. Supporting data that fail the target acceptance criteria for specific Data Quality Indicators will be evaluated against the affected sample result and the magnitude of the failure.

Examples:

A wastewater effluent, with a nitrate discharge permit level of 10 mg/L, was analyzed by a laboratory and found to have a nitrate concentration of 0.26 mg/L. For the recovery of the Laboratory Control Sample (LCS), the laboratory has established acceptance criteria of 80 – 120%. For this particular batch of samples, recovery for the LCS was 78%, and the laboratory properly qualified the result with a "J". Because the actual nitrate value was significantly lower than the permit limit, and the quality control failure was relatively minor, this result is judged to be usable.

A Class I waterbody was sampled for benzene to determine if concentrations complied with the water quality standard of 1.18 ug/L. A benzene analysis was performed with initial calibration standards ranging from 1 ug/L to 200 ug/L. All QC checks associated with this analysis were within the calibration range and within acceptance criteria. One of the project samples had a benzene concentration of 210 ug/L. The sample was qualified by the laboratory with a "J" and an explanation provided that the result exceeded the high level calibration standard. Because of the absolute magnitude of the

sample result (exceeding the water quality standard by nearly 209 ug/L), and the fact that it exceeded the high level standard by only 5%, the data is usable for the project.

A wastewater effluent sample, which was collected to determine compliance with a 10 ug/L arsenic permit limit, was found to have 9.5 ug/L of arsenic. While the Laboratory Control Sample acceptance criteria previously established by the laboratory was 75 – 125%, the LCS recovery associated with this sample was only 65%. These data are unusable to demonstrate compliance, due to the close proximity of the sample to the action level, and the low LCS recovery (well outside of the targeted acceptance criteria).

17.2. The magnitude of the sample result is considered when evaluating the consequences of the failed or absent Data Quality Indicators.

Examples:

An Everglades surface water sample, analyzed for compliance with a 10 ug/L total phosphorus criterion, was found to contain 21 ug/l of total phosphorus. The sample was properly preserved, but due to a laboratory mistake, the original sample had to be re-analyzed. The second analysis produced a total phosphorus result of 17 ug/L, but now the holding time was exceeded by 1 week. The value was qualified by the laboratory with a "Q" for exceeding the holding time. Despite a reduction in the phosphorus concentration that could have been associated with exceeding the holding time, the analytical result still exceeded the action level, and the datum is usable.

For a waste facility study, benzene was found in groundwater at a concentration of 5.7 ug/L. However, the reported method detection limit (MDL) for this particular benzene analysis was 1 ug/L, with a practical quantitation limit (PQL) of 4 ug/L. The Rule 62-777 FAC guidance document for routinely achievable PQLs indicates the target PQL for benzene is 1 ug/L. In this case, despite the elevated detection limit (indicating use of a less sensitive method), the sample result was above the reported PQL, and this result is usable.

A surface water sample was analyzed by membrane filtration for fecal coliform, to determine if the 800 Colonies/100 mL water quality standard was exceeded. The analytical result was calculated to be 1,200 Colonies/100 mL. Method blanks were run simultaneously with the surface water samples by filtering 100 mL of sterilized dilution water, which resulted in the presence of 2 – 5 target colonies in the blank samples. The laboratory did not use "V" (analyte detected in blank) to qualify the result. This result is usable despite the failure of the lab to properly qualify the samples, the high magnitude of the result exceedance, compared with the minor contamination in the blanks.

Biochemical Oxygen Demand analysis conducted on a surface water sample yielded a result of 5.5 mg/L. Dissolved oxygen depletion in the associated method blank was 0.3 mg/L, exceeding method criteria by 0.1 mg/L. The results were properly qualified with "V". The data is usable based on the magnitude of the exceedance compared with the quality control failure.

17.3. The evaluation of laboratory performance or the determination of usability for a data set will be based on a preponderance of Data Quality Indicator failures pointing to specific, systematic problems with the laboratory operation or the data set.

Examples:

The analytical data generated by a County surface water monitoring program were being audited to determine if the data could support Impaired Waters Rule listing decisions for the Total Maximum Daily Load (TMDL) program. Fifteen chlorophyll a samples, which had been analyzed over a five year period, were evaluated. Twice in this five year period, due to laboratory oversights, the 48 hour hold time for filtration was not met. Records demonstrated that the 48 hour hold time was the laboratory's target and that a quality system for meeting this objective was evident. Upon reviewing additional chlorophyll a quality control records, it was determined these holding time exceedances were isolated instances and that the 5 year period of data is usable.

Upon auditing a laboratory's quality control information associated with data submitted to the TMDL program, it was discovered that the majority of samples were analyzed beyond the accepted holding time, and that none of these results were qualified with "Q". When the laboratory manager was asked about the discrepancy, they indicated that the qualifiers were purposely suppressed so that the TMDL program would use all the data. Because these admissions indicated that the entire quality system was compromised, the data were deemed not usable.

17.4. Project management goals are taken into consideration when making data usability assessments. Specific Data Quality Objectives are established for certain analytical activities within a project, and these objectives will be evaluated when making a usability statement.

Examples:

A cleanup goal established for a waste remediation site stated that all analytical work must be sensitive enough to meet the Cleanup Target Levels (CTL) in Rule 62 – 777 FAC. During the project, a previously unknown area of contamination was discovered, and the Project Manager ordered some grab samples to delineate the aerial extent and magnitude of the newly found contamination. The lab used a method with a higher PQL (that did not meet the original cleanup objectives) but use of this method allowed a rapid turn-around time, enabling an effective adaptive management approach to better address the newly discovered contamination. These data are usable based on this set of objectives (delineating the new area of concern).

Four monitoring wells were sampled for trichloroethane (TCE) at a groundwater remediation site previously known to have TCE concentrations ranging from 20-30 ug/L. During this particular sampling, TCE measured at the four wells was found to be below 1 ug/L, and there were no laboratory quality control failures of any kind. However, sampling records indicated that large volumes of groundwater were purged with a centrifugal pump in a short time period, and that dissolved oxygen levels in the wells exceed 7 mg/L. Because the evidence indicates that improper sampling occurred, resulting in excessive aeration and de-gassing of volatile compounds, these data were determined to be unusable.

18. Procedure for Data Usability Determinations

18.1. Data auditors will review and evaluate the following information:

- The purpose for auditing the data;
- The reported values of the analytical results;

- The relationship between each analytical result and an associated decision or action level;
- The documented calibration, quality control and other supporting data compared with designated Data Quality Indicators in 62-160, F.A.C. or other specified Data Quality Objectives;
- The pattern, frequency, and magnitude of any failures or other deficiencies associated with the results;
- The extent to which the audited data set fulfills the Data Quality Objectives of the project or Program.

18.2. Based on the evaluation, the auditors will determine how the data can be used by the relevant Department programs.

19. Summary of the Data Usability Assessment Process

Based on the above inputs, the auditor will extrapolate audit findings to determine the overall performance of a laboratory for a period of record, and determine the usability of the data in question for a Department purpose. Usability assessments will evaluate the Data Quality Indicator results for the subject data set relative to DEP program or project objectives, and the follow the principles characterized in this guidance document to draw an "overall conclusion" concerning the usability of the data set. This conclusion will address individual samples or will express a "general assessment" based on examination of a representative sample set over a pre-determined time period, depending on the project objectives and the sample set being evaluated. This assessment will include, where applicable, the evaluation of data trends relative to laboratory corrective actions or laboratory events (e.g., laboratory instituted NELAC standards in November of 2004). The resulting usability determination would be characterized over certain segments of the audited period. Recommendations concerning usability of the data will be communicated with appropriate Department staff.