

Georgia Department of Natural Resources
Environmental Protection Division

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Inorganics Laboratory Sample and Data Validation

Access to this SOP shall be available within the laboratory for reference purposes; the official copy of this SOP resides on the official Georgia EPD website at <https://epd.georgia.gov/about-us/epd-laboratory-operations>. Printed copies of this SOP will contain a watermark indicating the copy is an uncontrolled copy.

1 Scope and Application

- 1.1 The Inorganics Laboratory has procedures to validate analytical data produced within each Laboratory. The procedures establish specific requirements for the review and validation of analytical data at the analyst, supervisor, manager and quality assurance staff levels. All data is fully validated prior to reporting. The validation of each sample is recorded in the LIMS.

2 Definitions

- 2.1 The following definitions are applicable to laboratory and sample validation.
- 2.1.1 **Continuing Calibration Blank (CCB)** — is an aliquot of reagent water, or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with other samples.
- 2.1.2 **Continuing Calibration Check (CCC)** — is a standard used to verify the current calibration. When it fails, a new calibration is required.
- 2.1.3 **Correlation Coefficient** — Relationship of value x to value y in a regression line. It is used as an evaluation of "goodness of fit" of a regression line.

- 2.1.4 **Initial Calibration Verification (ICV)** — is a second source used to verify the calibration curve.
- 2.1.5 **Initial Calibration Blank (ICB)** is an aliquot of reagent water, or other blank matrix that is treated exactly as a sample, including exposure to all glassware, equipment, solvents, filtration and reagents that is used with other samples.
- 2.1.6 **Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)** — are used validate the analytical batch with respect to accuracy and precision. The LCS and LCSD are prepared by spiking laboratory de-ionized water, or other matrix similar to sample matrix.
- 2.1.7 **Matrix Spike (MS) and Matrix Spike Duplicate (MSD)** — are utilized to evaluate matrix affect on the target analyte(s) with regards to accuracy and precision in a specific matrix. The spiking level is the same as chosen for the LCS and LCSD. Samples are spiked before sample preparation and carried through analysis as a sample in the batch.
- 2.1.8 **Method Detection Limit Spike (MDLs)** – is low level spike that is carried through the entire method prep and instrument analysis. A minimum of one MDL is required for each analytical batch. This provides for continuous analysis of Method Detection Limits for analytical methodology. The MDL is spiked equivalent to the lowest point on the calibration curve. This QC sample is reported in Labworks and statistically evaluated on a regular basis no less than once or twice per year, as specified by methodology.
- 2.1.9 **Method Detection Limit Blanks (MDLB)**-The MDLB is a method blank that is carried through the entire method prep and instrument analysis. A minimum of one MDLB is required for each analytical batch. GAEPD Lab uses the Method Blank as the MDLB. These QC samples are reported in Labworks and statistically evaluated on a regular basis, no less than once per year. The MDLB is used for comparison with the MDLS for statistically calculating the analytical method MDL for all analytes reported.
- 2.1.10 **Recovery %** — Recovery of spiked compound in appropriate matrix. It is used to assess accuracy.
- 2.1.11 **Relative Percent Difference (RPD)** — Measure of variability that adjusts for the magnitude of observations.

3 Interferences
Not applicable.

4 Safety
Not applicable.

5 Apparatus and Equipment
Not applicable.

6 Reagents

Not applicable.

7 Sample Collection

Not applicable.

8 Calibration

Not applicable.

9 Quality Control

Not applicable.

10 Procedure

10.1 Laboratory Scientist ensures all documentation for the analytical batch is complete and accurate and submits work in a data packet.

10.1.1 Includes a Data Review Checklist and Reagent List

10.1.1.1 Requires current CDF due date

10.1.1.2 Requires current MDL due date

10.1.1.3 Requires pipette number and pipette calibration date.

10.1.1.4 Requires Corrective Action # is applicable.

10.1.1.5 Requires a check if Second QC was added.

10.1.1.6 Requires all pages to be initialed and dated

10.1.1.7 Requires comments by supervisor and peer reviewer of errors noted.

10.1.1.8 Includes PMF review score

10.1.2 Includes QC batch sheets.

10.1.3 Includes standard and reagent sheet which lists reagent and standard names, chemical manufacturers, lot #s and expiration dates.

10.1.4 Includes graph and standard correlation when applicable.

10.1.5 Includes data from run.

10.1.6 Includes Digestion log when applicable.

10.1.7 Includes Digestion/Distillation block diagrams when applicable.

10.1.8 Includes copy of run log

10.2 Laboratory Scientist evaluates each data package for adherence with specific method requirements and compliance with laboratory data quality objectives.

10.2.1 Reviews correlation coefficient to make sure it is 0.995 or better.

10.2.2 Reviews ICV and ICB. The ICV must have a recovery of 90-110% and the ICB must be below the method PQL.

- 10.2.3 Reviews CCCs and CCBs. The CCC must have a recovery of 90-110% and the CCB must be below the method PQL.
- 10.2.4 Reviews LCS and LCS Duplicates for % Recovery and RPD. The LCS recovery and RPD must meet the acceptance criteria for the method.
- 10.2.5 Reviews Matrix Spike and Matrix Spike Duplicate for % Recovery and RPD. The Matrix Spike Recovery and RPD must meet the acceptance criteria limits for the method.
- 10.2.6 Reviews data for samples that need diluting.
- 10.3 Laboratory Scientist initiates a corrective action for data discrepancies requiring the final report to be commented, and for results that do not comply with laboratory data quality objectives.
- 10.4 Initiates corrective action for data that needs to be rerun. Ex: CCC % recoveries outside control limits, CCB's above PQL, Matrix spike and matrix spike duplicate off-scale, RPD for matrix spike and matrix spike duplicate outside control limits, ICV outside control limits, LCS % recovery outside control limits, and RPD for LCS outside control limit.
- 10.5 Initiates corrective action for data requiring final report to be commented. Ex: Matrix spike recovery outside control limits and Matrix spike relative percent difference outside control limit, insufficient sample to run matrix spike and matrix spike duplicate.
- 10.6 Laboratory Scientist evaluates the instrumentation or measurement system response for the influence of matrix introduced interference that could affect the accuracy of the reported sample result.
- 10.7 Laboratory Scientist submits completed packet to fellow scientist for peer review.
- 10.8 Peer reviewer ensures data quality objective requirements are complete and documented for each analytical data package.
 - 10.8.1 Reviews correlation coefficient to make sure it is 0.995 or better.
 - 10.8.2 Reviews ICV and ICB. The ICV must have a recovery of 90-110% and the ICB must be below the method PQL.
 - 10.8.3 Reviews CCCs and CCBs. The CCC must have a recovery of 90-110% and the CCB must be below the method PQL.
 - 10.8.4 Reviews LCS and LCS Duplicates for % Recovery and RPD. The LCS recovery and RPD must meet the acceptance criteria for the method.

- 10.8.5 Reviews Matrix Spike and Matrix Spike Duplicate for % Recovery and RPD. The Matrix Spike Recovery and RPD must meet the acceptance criteria limits for the method.
- 10.8.6 Reviews data for samples that need diluting and evaluates usage of correct dilutions.
- 10.8.7 Peer reviewer ensures all calculations required to produce final sample results are correct.
- 10.8.8 Reviews each page for initial and date.
- 10.8.9 Peer review initials and dates next to each batch requirement that the packet has been reviewed and corrected and notes any errors in the comment section.
- 10.9 Laboratory Scientist corrects noted errors and then enters results into Labworks LIMS System.
- 10.10 Laboratory Supervisor reviews all analytical data packages completed by Laboratory Scientists in respective laboratory and ensures that data packages include:
 - 10.10.1 Standards with lot #'s
 - 10.10.2 QC batch sheets
 - 10.10.3 Standard and Reagent list
 - 10.10.4 Data from analysis
 - 10.10.5 Ensures that correlation coefficient is included
- 10.11 Laboratory Supervisor ensures data quality objective requirements are complete and documented for each analytical data package.
- 10.12 Laboratory Supervisor evaluates data package for adherence with specific method requirements and compliance with laboratory data quality objectives.
 - 10.12.1 Reviews correlation coefficient to make sure it is 0.995 or better.
 - 10.12.2 Reviews ICV and ICB. The ICV must have a recovery of 90-110% and the ICB must be below the method PQL.
 - 10.12.3 Reviews CCCs and CCBs. The CCC must have a recovery of 90-110% and the CCB must be below the method PQL.
 - 10.12.4 Reviews LCS and LCS Duplicates for % Recovery and RPD. The LCS recovery and RPD must meet the acceptance criteria for the method.
 - 10.12.5 Reviews Matrix Spike and Matrix Spike Duplicate for % Recovery and RPD. The Matrix Spike Recovery and RPD must meet the acceptance criteria limits for the method.
 - 10.12.6 Reviews data for samples that need diluting and evaluates usage of correct dilutions.

- 10.13 Laboratory Supervisor ensures all calculations required to produce final sample results are correct.
- 10.14 Laboratory Supervisor reviews and approves corrective actions initiated by staff scientists and reviews comments on final reports.
 - 10.14.1 Signs corrective actions and comments on samples when needed.
 - 10.14.2 Ensures that original copy of corrective action is put in corrective action log.
 - 10.14.3 Ensures that scientist placed copy of corrective action in data package.
- 10.15 Laboratory Supervisor initials and dates data package when approved.
- 10.16 Laboratory Supervisor reviews result entry in Labworks LIMS system and validates analysis.
- 10.17 Lab Manager or supervisor prints out reports to review data input into Labworks LIMS system.
 - 10.17.1.1 For non-drinking water samples the following are also reviewed.
 - 10.17.1.2 Checks to see if BOD and TOC values are near expected values.
 - 10.17.1.2 Checks to see if Suspended Solids and Turbidity results are similar.
 - 10.17.1.4 Checks to see if COD values are near expected values.
 - 10.17.1.5 TKN must be greater or equal to NH_3 .
 - 10.17.1.6 Total Phosphorus must be greater or equal to Ortho-phosphorus.
 - 10.17.1.7 Alkalinity and Hardness values are similar.
 - 10.17.1.8 Lab pH and Conductivity values should match corresponding field values.
- 10.18 Laboratory Supervisor or Laboratory Manager validates the analysis and then validates the samples within Labworks LIMS.
- 10.19 Laboratory Supervisor or Laboratory Manager mails reports to appropriate person.
- 10.20 Laboratory Manager develops individual laboratory procedures for data validation.
 - 10.20.1 Assigns analysis validation responsibilities.
 - 10.20.2 Assigns project validation responsibilities.
 - 10.20.3 Establishes and requires adherence to method specific data quality objectives as required by each method and the EPD Laboratory Quality System.
 - 10.20.4 Sets control limits in Labworks LIMS for data quality objectives.
 - 10.20.5 Reviews and approves scientist or supervisor initiated corrective actions.
 - 10.20.6 Conducts enough additional reviews to ensure overall data quality.
 - 10.20.7 Exports USGS and WQ results on a monthly basis.
 - 10.20.8 Reviews SOPs each year and updates them as needed.

11 Calculations

Not Applicable

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References

12.1

EPD Laboratory Quality Assurance Plan, online revision.

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