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Organics 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 Organics Laboratory has a procedure to validate analytical data within the laboratory and comply with the Laboratory Quality Assurance Plan. The procedure establishes specific requirements for the review and validation of analytical data at the scientist, supervisor and manager levels. All data is fully validated prior to reporting. The validation of each sample is recorded in LIMS.

2 Definitions

2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Manual for quality control definitions. (SOP reference 13.1)

2.2 Laboratory Reagent Blank (LRB) – is an aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. GAEPD lab refers to this LRB as a Blank. Each blank is associated with an analytical batch or group of QC samples. Blank results should be less than the current RL for the associated method analysis.

2.3 Extraction Solvent Method Blank (ESMB) – is an aliquot of solvent prepared by transferring the extraction solvent into a flask just as an extracted sample. The purpose of this negative control is to demonstrate that the extraction solvent is free of interferences and contamination and that the glassware washing procedure is effective. ESMB results should be less than the current MDL for the associated method analysis.

2.4 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) – are used to evaluate the analytical batch for precision and accuracy. They are prepared by spiking a laboratory blank matrix with a known level of analyte.

2.5 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) – are used to evaluate a specific matrix effect on the target analytes for precision and accuracy. The spiking level is the same as for the LCS/LCSD. Samples are spiked prior to extraction and carried through the entire analysis.

2.6 Percent Recovery (%REC) – is used to access accuracy. It signifies the amount of recovery of spiked analyte(s) in an appropriate matrix.

2.7 Relative Percent Difference (RPD) – is a measure of precision of the absolute difference between two values of the same component in two discreet samples of the same category, i.e. the values of analytes between two quality control samples or two environmental samples.

2.8 Surrogate(s) – are compounds with similar characteristics to the analytes of interest that are added to method control samples and environmental samples to verify the effectiveness of the performance of the measurement system. *Note: In LabWorks and on chromatograms, Surrogates are often labeled as "SS" for "Surrogate Standard." This is not to be confused with the "SS" designation for "Secondary Source Standards."*

2.9 Initial Calibration Verification (ICV) – is a second source standard of a known concentration used to verify the calibration curve.

2.10 Continuing Calibration Check (CCC) – is a standard of a known concentration used to verify the current calibration.

2.11 Laboratory Performance Check (LPC) – is a solution of method analytes, Surrogate compounds and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

2.12 Internal Standard (IS) – is a compound purposely added to both samples and standards at a known concentration in order to provide a basis for comparison in quantitation.

2.13 Method Detection Limit (MDL) – is a statistical estimate of the lowest concentration at which there is a 99% chance that the concentration is greater than zero.

2.14 Correlation Coefficient (r) – is the relationship of value x to value y in a regression line. It is used as an evaluation of "goodness of fit" of the regression line of the calibration curve.

2.15 Primary Source Standard (PS) – is a standard used to make the calibration points of the curve.

2.16 Second Source Standard (SS) – is a standard made from a manufacturer other than the Primary Source Standard to verify the accuracy of the Primary Source Standard. It is typically used to make the ICV.

2.17 Primary Sample – is a sample that is collected simultaneously with another sample using two separate collection systems for comparative purposes. One sample is designated as "primary."

2.18 Collocated Sample – is a sample that collected simultaneously with another sample using two separate collection systems. The "collocated" sample is sampled simultaneously with the "primary" sample using a collection system other than the collection system used to collect the primary sample.

2.19 Duplicate Sample – is a sample that is collected simultaneously with an "original" sample using one collection system for comparative purposes.
2.20 Replicate Analysis – is the analysis of one discreet sample multiple times.

3 Interferences
3.1 Not Applicable

4 Safety
4.1 Refer to the Laboratory Chemical Hygiene Plan, online revision.

5 Apparatus and Equipment
5.1 Not Applicable

6 Reagents and Standards
6.1 Not Applicable

7 Sample Collection
7.1 Not Applicable

8 Calibration
8.1 Not Applicable

9 Quality Control
9.1 Not Applicable

10 Procedure
10.1 Personal Responsibility
10.1.1 Technicians – Technicians will review the SOP(s) and EPA Method(s), if applicable, for their assigned duties. Technicians will assist with the prepping and completion of sample analyses in varying capacities ranging from bottle prepping, sample prepping, shipping and receiving, glassware prepping and cleaning, etc.
10.1.2 Scientists – All Scientists will review the EPA Method(s) and Laboratory SOP(s) for their assigned method(s). Differences between the promulgated method reference and the laboratory SOP will be brought to the attention of the Laboratory Manager or Supervisor. They should verify all method requirements are met before submitting the data for validation. Each Scientist is responsible for ensuring that their training records (CDFs) are current.
10.1.3 Supervisors – Supervisors will review the data package to verify that all QC requirements are met. Completeness and accuracy of LabWorks entries will be checked and documented. Supervisors will carry out the requirements of Section 10.1.2 for Scientists in their absence. Supervisors will also ensure that MDLs and RT Studies are current for their assigned EPA Methods and instruments. Supervisors will retain custody of all MDL and RT data packages after approval by the QA Officer. Supervisors will

also retain custody of all IDC and CDC data packages submitted for their assigned methods.

10.1.4 Managers – Laboratory Managers will ensure all requirements of the SOP are met for each Supervisor, Scientist and Technician in the laboratory. Managers will carry out the requirements of Section 10.1.3 for Supervisors in their absence. Managers will also ensure that Control Charts and SOPs are current for their assigned EPA Methods. Managers will retain custody of the Control Charts after approval by the QA Officer. The Manager will also act as the SOP Custodian. The Manager will retain Employee Training Folders for each individual employee.

10.2 Scientist Review

10.2.1 During the analytical run, the analyst should review QC samples and standards, if possible, to ensure that all method requirements are met. Any failures should be addressed and reported to the Supervisor as soon as possible. A Corrective Action form is generated as necessary for sequences requiring re-runs, data failing quality objectives, insufficient sample to perform required quality control tests or any other action that occurs which deviates from the SOP.

10.2.2 Data packages submitted to the Supervisor must contain chromatograms for all sample data and applicable confirmation data, a completed sequence review form, chromatograms of all associated QC data, any applicable results sheets and calculated reports, copies of completed extraction sheets and run sequences, print outs of all associated curves with cal-plots, chromatograms and ICVs and any associated Corrective Actions.

10.2.3 Analyses that meet all QC requirements are transferred into LabWorks after approval by the Supervisor or Manager.

10.3 Supervisor Review

10.3.1 Supervisors evaluate data packages for technical merit, adherence to specific method requirements and compliance with data quality objectives.

10.3.2 The Calibration Curve, ICV and subsequent CCCs are reviewed for each analyte in the run.

10.3.3 LCS, LCSD, MS and MSD are checked for Percent Recovery and RPD compliance. Surrogate recovery is verified. Pesticide Breakdown, Internal Standards, LPCs, MDLs, RT updates, etc. are checked where applicable.

10.3.4 All Corrective Action forms, data comments and any flags generated by the Corrective Action are reviewed for completeness and accuracy.

10.3.5 The QA/QC Report is reviewed. Any entries appearing in **Bold** print are failures and the data must be flagged, commented and documented by a Corrective Action form before validation.

10.3.6 If any of the above checks are unacceptable, the data package is returned to the analyst for correction or re-analysis. If all the checks are acceptable, the Supervisor enters “Completed” for their validation code.

10.4 Manager Review

10.4.1 The Manager will review all data cited in Section 10.3 in the Supervisor’s absence.

10.4.2 The Manager will review all MDL, RT, Control Charts, IDFs, SOPs and any other QC data prior to submittal to the QA Officer for approval.
10.4.3 The Manager may review and approve CDFs in place of the QA Officer.
10.4.4 The Manager will review and ensure that the LabWorks test codes are accurate and up to date.

11 Calculations

11.1 Not Applicable

12 Waste Management

12.1 See GA EPD Laboratory SOP – EPD Laboratory Waste Management Standard Operating Procedures, SOP 6-015, online revision.

13 References

13.1 Georgia EPD Laboratory Quality Assurance Manual, online revision.
13.2 Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, August 1995, EPA/600/R-95-131.
13.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, December 1996.
13.4 Manual for the Certification of Laboratories Analyzing Drinking Water, 5th Edition, January 2005 or later.
13.5 Technical Assistance Document for the National Air Toxics Trends Stations Program, Revision 3, October 2016 or later.
13.6 EPA Clean Water Act Methods Update Rule for the Analysis of Effluent, August 2017, EPD-HQ-OW-2014-0797; FRL-9920-55-OW.

14 Reporting Limits (RLs), Precision and Accuracy Criteria and Quality Control Approach

14.1 Not Applicable

15 Associated LabWorks Test Codes

15.1 Not Applicable