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1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that Environmental Standards' data reviewers will use to validate total organic carbon (TOC) data generated by the Lloyd Kahn Method for General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. Validation will be performed to assess the compliance of the sample data to the Lloyd Kahn Method and/or other reference documents (*e.g.*, analytical SOP) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the TOC data provided by the analytical laboratories will be determined based on the general guidance provided in the "US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review" (2/94) (National Functional Guidelines). It should be noted that the National Functional Guidelines applies strictly to data generated by the Contract Laboratory Program (CLP) protocol. As such, it is not directly applicable to validation of data generated by the Lloyd Kahn Method; therefore, this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) that will be prepared from one or more sample delivery groups (SDGs). Copies of annotated analytical results summaries (Form I's), including any changes to the analytical results and data qualifier codes, or a data summary spreadsheet of the qualified analytical results will be included in the analytical results section of the QAR.

2.0 EVALUATION TOOLS

Excel forms available in R:/Templates/Chemistry/XCELForms:

Inorganic field duplicate comparison Rev1-01.xls
Organic field triplicate comparison Rev1-01.xls

Chemistry Applications:

FIT
Methods Database

3.0 REFERENCE DOCUMENTS

US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (2/94).

Northeast Analytical, Inc.- Standard Operating Procedure for the Determination of Total and Particulate Organic Carbon According to Tekmar Dohrmann Application Note TOC-011 (Rev. 3; 2/10/00).

Region II, Evaluation of Metals Data for the Contract Laboratory Program (CLP) (1/92) Validation of Inorganics.

Region I, EPA – New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

Region III, Modifications to Natural Functional Guidelines for Inorganic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the TOC data based on an evaluation of information presented in the data package deliverables. Compliance to the Lloyd Kahn Method and/or other reference documents (*e.g.*, analytical SOPs) as applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program (as directed by the Project Manager) will be evaluated as part of the assessment. In addition, the deliverables will be evaluated for reporting errors and inconsistencies. The findings of the method compliance assessment will be described in terms of deficiencies and comments about the data/deliverables. The deficiencies/comments will be presented in three subdivisions (*i.e.*, correctable deficiencies, noncorrectable deficiencies, and comments) of the Inorganic Data Evaluation Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or any certain aspect(s) of the data that could not be evaluated due to the deficiency.

The data reviewer should contact the project laboratories to request the correction of deficiencies prior to the submittal of the QAR (if feasible and sanctioned by General

Electric Company). At a minimum, corrections necessary for a full evaluation of the usability of the data should be requested. Such correctable deficiencies may include sample result errors, missing data deliverables, or calculation errors that would take a significant amount of the data reviewer's time to correct. Any laboratory resubmittals as a result of such requests will be discussed in the comments subdivision of the QAR and included as an attachment to the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the TOC data based on an evaluation of the information presented in the data package deliverables. The findings of the TOC data usability assessment will be described in terms of certain qualifications of the data that the project team should consider in order to best utilize the data. These qualifications will be presented in the Inorganic Data Qualifier subsection of the QAR. Each qualification discussed in the QAR will indicate that the affected sample result(s) has been flagged with representative qualifier code(s) in the General Electric Company's database to provide, at a glance, an indication of the quantitative and qualitative reliability of each analytical result. In general, the qualifier statements will be presented in the QAR in the following order: blank contamination, common contaminants that were not qualified, unusable results (R/UR), estimated results (J/UJ), field duplicate comparison, and a general qualifier for all results reported below the quantitation limit (if applicable to General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program).

The data reviewer's criteria for evaluating the usability of the TOC data and the resultant qualifications will be as stated in the attached Table for the Validation of Total Organic Carbon (TOC) Data Generated by the Lloyd Kahn Procedure. It should be noted that the Project Manager should be consulted when directed to use "professional judgement" in the attached table.

Notes for the Validation of TOC Data Generated by the Lloyd Kahn Procedure

Quality Control Item	Usability Criteria	Action
Temperature and Conditions Upon Receipt	4±2°C pH<2 for aqueous samples	If temperature >10°C, but ≤20°C, qualify positive results as estimated ("J") and qualify "not-detected results as estimated ("UJ"). If temperature >20°C, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). Note time of collection relative to receipt at laboratory. Professional judgement should be used if less than 8 hours had lapsed from collection to receipt to determine if the qualification above applies. If the aqueous samples have pH values >2, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Technical Holding Time	Soil samples should be analyzed within 14 days of sample collection. Aqueous samples should be analyzed within 28 days of sample collection.	If the aqueous analysis was performed >28 days but <56 days, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the soil analysis was performed >14 days but <28 days, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the aqueous analysis was performed >56 days, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the soil analysis was performed >28 days, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Initial Calibration (See Note #1 for additional information)	r (linear) or coefficient of determination (COD) (quadratic) should be ≥0.99.	Use professional judgement when evaluating correlation coefficients and coefficients of determination (r or COD). If r or COD <0.99 but ≥0.85, qualify positive results as estimated ("J") and do not qualify "not-detected" results. If r or COD <0.85, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").

Notes for the Validation of TOC Data Generated by the Lloyd Kahn Procedure

Quality Control Item	Usability Criteria	Action
Independent Calibration Verification Standard (ICV) and Continuing Calibration Verification Standard (CCV)	ICV and CCV Recoveries (%R) should be 85-115%	If the ICV or CCV %R>115% qualify positive results as estimated ("J") and do not qualify "not-detected" results. If the ICV or CCV 55%≤%R<85% qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If the ICV or CCV %R <55%, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("UR").
Blanks (Preparation and/or Continuing Calibration Blanks) (See Note #2 for additional information)	Summarize all results greater than the method detection limit (MDL) present in the blanks. The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a target analyte is found in blank but not in the associated sample(s) no action is needed. If sample >RL, but <5× blank result, qualify the positive results as "not detected" ("U*"). If sample is positive, but <RL and <5× blank result, qualify the positive result as "not-detected" ("U*"). If sample result >5× blank result no qualification is necessary.
Laboratory Fortified Sample Matrix Sample (MS)	For accuracy, use default recovery limits of 75-125%. For precision, use RPD limits of 20% for aqueous and 40% for solid.	Data should not be qualified due to %Rs (or RPD calculated on %Rs) that are outside of criteria if the original concentration of a compound is > 4 × spiking level for that compound. RPDs calculated using MS/MSD results can still be used to evaluate precision. If the recovery is >125%, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results. If the recovery is 30%≤%R<75%, qualify positive results in the all associated samples as estimated ("J") and qualify "not-detected" results in all associated samples as estimated ("UJ"). If the recovery is <30%, qualify positive results in all associated samples as estimated ("J") and qualify "not-detected" results as unusable ("UR"). If the precision exceeds the specified RPD, qualify positive results in all associated samples as estimated ("J") and do not qualify "not-detected" results.

Notes for the Validation of TOC Data Generated by the Lloyd Kahn Procedure

Quality Control Item	Usability Criteria	Action
Field/Laboratory Duplicate (See Note #3 for additional information)	Use limit of 20% RPD (%RSD for triplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate analyses) for solid samples for sample results ≥5× RL. Use default limit of ± RL for aqueous samples and ±2× RL for solid samples when at least one sample value is <5× RL. (Use one-half of the numerical value for comparison if the TOC was "not-detected")	If the criteria are not met, qualify positive results for the non-compliant analyte in the original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Percent Solids	Solid samples with less than 50% solid content require qualification.	If a solid sample has a percent solid content <50% but ≥10%, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). Use professional judgement if a solid sample has a percent solid content <10%.
Overall Assessment of Data	Assess overall quality of the data. Review available materials to assess the quality, keeping in mind the additive nature of the analytical problems.	Use professional judgement to determine the need to qualify data which were not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitations of the data. If sufficient information on the intended use and required quality of the data is available, the reviewer should include his assessment of the usability of the data within the given context.

Notes for the Validation of TOC Data Generated by the Lloyd Kahn Procedure

1. Use professional judgement when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive then the samples should be evaluated for false positives. If the concentration intercept is negative then the samples should be evaluated for false negatives.

2. The frequency of field/equipment/rinse blanks is determined during the sampling event. The results of a field/equipment/rinse blank should be applied to all samples collected using the same equipment (equipment/rinse blanks only) on the same day (unless only one was collected for a several-day sampling event; results would be applied to all samples in the SDG). In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration for a contaminant.

3. Duplicate samples may be taken and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; therefore, the results may have more variability than laboratory duplicates which measure only laboratory performance. Field duplicate sample results should only be applied to the original sample and its field duplicate. Laboratory duplicate should be applied to all samples in a batch. It is also expected that soil duplicate results will have a greater variance than aqueous duplicate results.