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1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate inorganic data generated by SW-846 Method 6010B for the 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 SW-846 Method 6010B 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. In addition, the usability of the inorganic data provided by the project laboratories will be determined based on the general guidance provided in the "US EPA Contract Laboratory National Functional Guidelines for Inorganic Data Review" (2/94; National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by Contract Laboratory Program (CLP) protocol and are not directly applicable to validation of data generated by SW-846 Method 6010B; 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 for 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 Rev 1-01.xls
- Inorganic triplicate comparison Rev 1-01.xls
- Total versus dissolved comparison Rev 1-01.xls

Chemistry Applications:

- FIT
- Methods Database

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (2/94).
- SW-846 Method 6010B.
- Region I, EPA – New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).
- Region II, Evaluation of Metals Data for the Contract Laboratory Program (CLP) (1/92) Validation of Inorganics.

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- 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 inorganic data based on evaluation of information presented in the data package deliverables. Compliance with SW-846 Method 6010B 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 Section of the QAR. Each deficiency and comment discussed in the QAR will indicate any subsequent impact on the usability of the data or will identify 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 certain deficiencies prior to submittal of the QAR (if feasible and sanctioned by General Electric Company). At a minimum, corrections required to allow 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 require a significant amount of the data reviewer's time to correct. In addition, the data reviewer should contact the project laboratories if feasible to request the correction of all correctable deficiencies that impact sample results or that the data reviewer was unable to correct prior to the submittal of the QAR, if time allows. Any laboratory resubmittals as a result of such requests will be discussed in the comments subsection of the QAR and will be included as an attachment of the QAR.

4.2 DETERMINATION OF DATA USABILITY

The data reviewer will determine the usability of the inorganic data based on an evaluation of the information presented in the data package deliverables. The findings of the inorganic data usability assessment will be presented in terms of data qualifications 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 will indicate that the affected sample result(s) has been flagged with a 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, unusable results (R/UR), estimated results (J/UJ), field duplicate comparison, and a general qualifier for all results reported below the quantitation/reporting 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 inorganic data and the resultant qualifications will be as stipulated on the attached Table for the Validation of Metals Data Generated by SW-846 Method 6010B. It should be noted that the Project Manager should be consulted when "professional judgement" use is indicated on the attached table.

Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Temperature and Conditions Upon Receipt	Aqueous samples should be preserved with nitric acid to pH \leq 2. Solid/soil samples should be preserved to 4 \pm 2°C.	If pH is >2 and the laboratory did not adjust the pH and allow the sample to sit for 16 hours before digestion, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). Solid/soil samples should not be qualified due to out-of-criteria temperature upon receipt.
Technical Holding Time	All matrices should be analyzed within 6 months of sample collection.	If holding time is exceeded, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). If holding time is grossly exceeded (>1 year from date of sample collection), qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“UR”).
Initial Calibration	Should be established with a minimum of one blank and one standard.	Use professional judgement if the minimum number of standards was not used or if instrument was not calibrated daily and/or not calibrated each time set up.
Instrument Performance (See Note #1 for additional information.)	%D or %RSD between replicate exposures should be \leq 20%. Samples should not display negative results >2 \times the instrument detection limit (IDL).	If %RSD or %D>20%, qualify positive results greater than the reporting limit as estimated (“J”) and do not qualify “not-detected” results. If a negative result >5 \times IDL, qualify the “not-detected” result as unusable (“UR”). If an analyte displays a negative result >2 \times IDL, qualify the “not-detected” result as estimated (“UJ”).
Initial Calibration Verification (ICV)	For accuracy, use recovery limits of 90-110%.	Qualify samples for an entire analytical sequence. If an analyte recovery is >110% but \leq 125%, qualify positive results as estimated (“J”) and do not qualify “not-detected” results. If an analyte recovery is <90% but \geq 75%, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). If an analyte recovery is >125%, qualify positive results as unusable (“R”) and do not qualify “not-detected” results. If an analyte recovery is <75%, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“UR”).

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Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Continuing Calibration Verification (CCV)	For accuracy, use recovery limits of 90-110%.	<p>Qualify samples analyzed before and after a non-compliant CCV.</p> <p>If an analyte recovery is >110% but ≤125%, qualify positive results as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If an analyte recovery is <90% but ≥75%, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If an analyte recovery is >125%, qualify positive results as unusable (“R”) and do not qualify “not-detected” results.</p> <p>If an analyte recovery is <75%, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“UR”).</p>
CRDL/CRI Standard (not required, but frequently analyzed.) (See Note #2 and Note #8 for additional information.)	For accuracy, use recovery limits of 85-115%.	<p>Qualify samples analyzed before and after a non-compliant CRDL/CRI standard.</p> <p>If an analyte recovery is >115%, qualify positive results ≤3× the spike level as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If an analyte recovery is <85% but ≥50%, qualify positive results ≤ 3× the spike level as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If an analyte recovery is <50%, qualify positive results ≤3× the spike level as estimated (“J”) and qualify “not-detected” results as unusable (“UR”).</p> <p>If an analyte recovery is >150%, qualify positive results ≤ 3× the spike level as unusable (“R”), qualify positive results >3× the spike level but ≤5× as the spike level estimated (“J”), and do not qualify “not-detected” results.</p>

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Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Initial Calibration Blank (ICB)/Continuing Calibration Blank (CCB)/Preparation Blank (PB)/Field Blank/Equipment Blank (See Note #3 and Note #8 for additional information.)	The highest positive result (greater than the IDL) associated with a sample should be summarized and utilized for evaluation of contamination.	For ICBs and CCBs qualify samples per analytical sequence; for PBs, field blanks, and equipment blanks qualify per batch and for SDG. If an analyte is detected in the blank but not in the associated samples, no action is required. If a sample result is >MDL/DL but ≤5× blank result, qualify the positive result as “not-detected” (“U*”). If a sample result is >5× blank result, qualification is not required. If a blank has a negative result with an absolute value >2× IDL, qualify positive results ≤5× the absolute value of the blank result as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).

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Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
ICP Interference Check Sample Analysis (ICSA/ICSAB) (See Note #4 for additional information.)	For accuracy, use recovery limits of 80-120% for ICSA/ICSAB. The absolute value of analytes not present in ICSA solution should be <2× IDL.	Qualify samples analyzed before and after ICSA/ICSAB standard. Sample data are acceptable if the concentrations of interferents (<i>i.e.</i> , Al, Ca, Fe, and Mg) in the samples are ≤ 50% of the respective ICSA concentrations. For samples with concentrations of interferents (<i>i.e.</i> , Al, Ca, Fe, and Mg) >50% of the respective concentrations in the ICSA, qualify as follows: If an ICSAB recovery is > 120%, qualify positive results as estimated (“J”) and do not qualify “not-detected” results. If an ICSAB recovery is 50-79%, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). If an ICSAB recovery is <50%, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“UR”). If positive results are observed in the ICSA for non-ICSA analytes that are >2× DL, qualify positive results up to 5× ICSA concentration in samples with high (>50% ICSA interferents) interferents as estimated (“J”) and do not qualify “not-detected” results. If negative results with an absolute value >2× DL are observed in the ICSA for non- ICSA analytes, qualify positive results up to 5× the concentration observed in the ICSA in samples with high (>50% ICSA interferents) interferents as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).

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Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Laboratory Control Sample (LCS) (See Note #5 for additional information.)	For accuracy, use recovery limits of 80-120% for aqueous samples and 70-130% for solid samples.	<p>For aqueous samples, if a recovery is >120% but ≤150%, qualify positive results as estimated (“J”) and do not qualify “not-detected” results.</p> <p>For aqueous samples, if a recovery is <80% but ≥50%, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>For aqueous samples, if a recovery is >150%, qualify all positive results as unusable (“R”) and do not qualify “not-detected” results.</p> <p>For aqueous samples, if a recovery is <50%, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“UR”).</p> <p>For solid samples, if a recovery is >130%, qualify positive results as estimated (“J”) and do not qualify “not-detected” results.</p> <p>For solid samples, if a recovery is <70% but ≥30%, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>For solid samples, if recovery is <30%, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“UR”).</p>
Matrix Spike/Matrix Spike Duplicates (MS/MSD) (See Note #6 and Note #8 for additional information.)	<p>For accuracy, use recovery limits of 75-125%.</p> <p>For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.</p>	<p>Data should not be qualified due to %Rs (or RPDs calculated using %Rs) that are outside of criteria if the original concentration of an analyte is >4× the spiking level for that analyte. RPDs calculated using MS/MSD results can be used to evaluate precision.</p> <p>If a recovery is >125%, qualify positive results in all associated samples as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If a recovery is <75% but ≥30%, qualify positive results in all associated samples as estimated (“J”) and qualify “not-detected” results in all associated samples as estimated (“UJ”).</p> <p>If a recovery is <30%, qualify positive results in all associated samples as estimated (“J”) and qualify “not-detected” results in all associated samples as unusable (“UR”).</p> <p>If the precision exceeds the RPD criterion, qualify positive results in all associated samples as estimated (“J”) and do not qualify “not-detected” results.</p>
ICP Serial Dilution Analysis	%D<10% if original undiluted concentration is >50× IDL.	If %D is >10%, qualify positive results as estimated (“J”) and do not qualify “not-detected” results.

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Table for the Validation of Metals Generated by SW-846 Method 6010B

Quality Control Item(s)	Usability Criteria	Action(s)
Field Duplicate/Laboratory Duplicate (See Note #6, Note #7 and Note #8 for additional information.)	Use default limits of 20% RPD (%RSD for triplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate analyses) for solid samples when sample results are $\geq 5 \times$ RL. Use default limit of \pm RL for aqueous samples and $\pm 2 \times$ RL for solid samples when at least one sample result is $< 5 \times$ RL.	If the criteria are not met, qualify positive results for non-compliant analyte in original sample and its duplicate as estimated ("J") and qualify "not-detected" results as estimated ("UJ").
Total vs. Dissolved Comparison (See Note #8 for additional information.)	When the dissolved result is greater than the total result: use default limits of \pm IDL if at least one result is $< 10 \times$ IDL. Use default limit of percent difference $< 10\%$ if both results are $\geq 10 \times$ IDL.	If the criteria are not met, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ"). If at least one result is $< 10 \times$ IDL and the difference is $> 5 \times$ IDL, qualify positive results and "not-detected" results as unusable ("R/UR"). If both results are $\geq 10 \times$ IDL and the percent difference is $> 50\%$, qualify positive results as unusable ("R").
Percent Solids	Solid samples with $< 50\%$ solid content require qualification.	If a solid sample has a percent solid content $< 50\%$ but $\geq 10\%$, 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 not qualified based on the QC previously discussed. Write a brief narrative to give the user an indication of the analytical limitation of the data. If sufficient information of the intended use and required quality of the data is available, the reviewer should include the assessment of the usability of the data within the given content.

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**Notes for the Validation of Metals Data
Generated by SW-846 Method 7470A/7471A**

1. Due to the nature of trace ICP data, IDL may be very low such that $5\times$ the IDL may be below the laboratory reporting limit. If the IDL is very low, use professional judgement to determine if the reporting limits should be qualified due to negative sample results.
2. If the spike concentration of the CRDL/CRI standard is $<$ the IDL/MDL, do not utilize the results for qualification. Use professional judgement if the spike concentration of the CRDL/CRI standard is \geq IDL but $<$ the reporting limit.
3. Generally, if 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 of a contaminant. When evaluating blank contamination, sample weights, volumes, and initial dilution factors should be taken into account. Sample results should not be blank corrected.

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 (if only one blank was collected for a several-day sampling event, results would be applied to all samples in the SDG).

4. When comparing ICSA results to sample results, the units of each should be the same (*i.e.*, if the sample results are in mg/kg and the ICSA results are in $\mu\text{g/L}$, convert the ICSA results to mg/kg before comparing the results.) If the negative interference in the ICSA solution is comparable (similar level) to the negative values observed in the CCBs,

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**Notes for the Validation of Metals Data
Generated by SW-846 Method 7470A/7471A**

the negative ICSA values should not be utilized for qualification. If the negative interference in the ICSA solution is not comparable to the negative values observed in the CCBs, the negative ICSA values should be utilized for qualification.

5. The spike level for the solid LCS should be comparable to the detection limit. Use professional judgement if the spike level is not comparable to the detection limit.
6. The laboratory may choose to analyze an MSD instead of a laboratory duplicate. The laboratory may include a post-digestion matrix spike (PDS) analysis. These results are not utilized for qualification; however, the results are utilized to evaluate the MS/MSD recoveries.
7. Duplicate samples may be collected 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 that measure only laboratory performance. Field duplicate sample results should only be applied to the original sample and its field duplicate. Laboratory duplicates 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.
8. The use of RL/DL in evaluating laboratory quality is as follows:
 - when evaluating negative values, total versus dissolved results, and the ICSA (non-spiked) compounds, the DL should be used.

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**Notes for the Validation of Metals Data
Generated by SW-846 Method 7470A/7471A**

- when evaluating field duplicates and laboratory duplicates, the RL/QL should be used.

The DL is defined as the number that the positive results are reported down to; therefore, the DL may be the IDL, MDL, or RL.

The RL is defined as the quantitation limit or project reporting limit. If the laboratory did not provide the RL, the IDL or MDL should be used.

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