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APPENDICES

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1.0 OBJECTIVES

This standard operating procedure (SOP) describes procedures that the Environmental Standards data reviewers will use to validate PCB data generated by US EPA Method 680 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 US EPA Method 680 and/or other reference documents (*e.g.*, analytical SOPs) as applicable to the General Electric Company's Hudson River Design Support Sediment Sampling and Analysis Program. In addition, the usability of the PCB data provided by the analytical laboratories will be determined based on the general guidance provided in the "US EPA Contract Laboratory National Functional Guidelines for Organic Data Review" (10/99; National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by the Contract Laboratory Program (CLP) protocol and are not directly applicable to validation of data generated by US EPA Method 680; 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 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 support documentation of the QAR.

2.0 EVALUATION TOOLS

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

- Organic field duplicate comparisons Rev1-01.xls
- Organic field quadruplicate comparison Rev1-01.xls
- Organic field triplicate comparison Rev1-01.xls

Chemistry Applications:

- FIT
- Methods Database
- Target version 4.1 data processing software

3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (10/99).
- US EPA Method 680 as presented in SOP GEHR680.
- Region I, EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses (12/96).

- Region II, Standard Operating Procedure for the Validation of Organic Data Acquired Using SW-846 Method 8082 (Rev 2. 12/96).
- Region III, Modifications to National Functional Guidelines for Organic Data Review (9/94).

4.0 PROCEDURE

4.1 EVALUATION OF METHOD COMPLIANCE

The data reviewer will assess the method compliance of the PCB data based on an evaluation of information presented in the data package deliverables. Compliance to US EPA Method 680 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 Organic 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 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 deficiencies prior to submittal of the QAR (if feasible and sanctioned by to General Electric Company). At a minimum, corrections required to allow for a full evaluation of

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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. 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 PCB data based on an evaluation of the information presented in the data package deliverables. The findings of the PCB 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 Organic Data Qualifier subsection of the QAR. Each qualification discussed in the QAR will indicate that the affected sample result(s) has been flagged with a representative qualifier code(s) to 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 (U*), unusable results (R/UR), estimated results (J/UJ), tentative identifications of target compound results (N), 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 PCB data and the resultant qualifications will be as stipulated on the attached Table for the Validation of PCB (Aroclor) Data Generated by US EPA Method 680 (SOP GEHR680). It should be noted

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that the project manager should be consulted when “professional judgement” use is indicated on the attached table.

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Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	4±2°C	If temperature is >10°C but ≤20°C, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). If temperature is > 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 < 8 hours has elapsed from collection to receipt at the laboratory to determine if qualification due to elevated temperature applies.
Technical Holding Time	Aqueous samples should be extracted within 7 days of sample collection. Solid/soil samples should be extracted within 14 days of sample collection. All matrices should be analyzed within 40 days after extraction.	If a holding time is exceeded, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). If a holding time is grossly exceeded (<i>i.e.</i> , > twice the holding time), qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“UR”).
GC/MS Tuning (DFTPP)	Ion abundances should meet the method acceptance criteria. (See Note #1 for criteria.)	If mass calibration was not performed, qualify all associated data as unusable (“R”). If mass assignment is in error, qualify all associated data as unusable (“R”). Use professional judgment if abundance criteria are not met. Use professional judgment if samples are analyzed more than 12 hours after a compliant tune and there is no evidence of a compliant tune following the samples.

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Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Initial Calibration (See Note #2 for additional information.)	Nine selected PCB congeners are used as calibration standards to represent each homolog group, the mono- through octachlorobiphenyls and decachlorobiphenyl. Decachlorobiphenyl is used as the calibration congener for both nona- and decachlorobiphenyl homolog groups. Five response factors (RFs) for each PCB calibration congener and surrogate must be calculated relative to chrysene-d ₁₂ . If interference or problems exist with chrysene-d ₁₂ then RFs will be calculated using phenanthrene-d ₁₀ . Each %RSD should be ≤20%.	If a PCB congener has 20% < %RSD ≤ 50%, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and do not qualify "not-detected" results for the associated homolog group(s) and total PCBs. If a PCB congener has 50% < %RSD ≤ 90%, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and use professional judgement to qualify "not-detected" results for the associated homolog group(s) and total PCBs. If a PCB congener has %RSD > 90%, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and qualify "not-detected" results for the associated homolog group(s) and total PCBs as unusable ("UR").
Continuing Calibration Verification (CCV) (See Note #3 for additional information.)	A CCV is required at the beginning and end of each 12-h period during which analyses are performed. The % difference (%D) for each PCB calibration congener and surrogate in each CCV should be ≤20%.	Qualification is for all samples on both sides of the out-of-criteria calibration standards. If a PCB congener has 20% < %D ≤ 90% with the response indicating a sensitivity decrease, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and qualify "not-detected" results for the associated homolog group(s) and total PCBs as estimated ("UJ"). If a PCB congener has %D > 20% with the response indicating a sensitivity increase, qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and use professional judgement to qualify "not-detected" results for the associated homolog group(s) and total PCBs. If a PCB congener has %D > 90% with the response indicating a sensitivity decrease qualify positive results for the associated homolog group(s) and total PCBs as estimated ("J") and qualify "not-detected" results for the associated homolog group(s) and total PCBs as unusable ("UR").

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Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
SIM PCB Data Performance Criteria for Calibration Standards	GC separation -- Baseline separation of PCB congener #87 (Cl ₅) from congeners #154 (Cl ₆) and #77 (Cl ₄), which may coelute.	If baseline separation is not observed and the unresolved congeners are observed in an associated sample, qualify positive results for the associated homolog groups and total PCBs as estimated ("J").
	MS sensitivity -- Signal/noise ratio of ≥ 5 for m/z 499 of PCB congener #209, Cl ₁₀ -PCB, and for m/z 241 of chrysene-d ₁₂ .	If the S/N ratio was <5 , use professional judgment to determine potential qualitative impacts.
	MS calibration -- Abundance of $\geq 70\%$ and $\leq 95\%$ of m/z 500 relative to m/z 498 for congener #209, Cl ₁₀ -PCB.	If the relative ion abundance ratio was not with the stated range, use professional judgment to determine the accuracy of qualitative identifications (both positive and "not-detected" results), focusing on chlorine cluster ions. Carefully evaluate sample ion ratios.
Internal Standards	The area measured for m/z 240 for chrysene-d₁₂ nor that for m/z 188 for phenanthrene-d₁₀ should not have changed by more than 30% from the area measured in the most recent previous analysis of a CCV standard or decreased by more than 50% from the mean area measured during initial calibration. Retention time (RT) of the internal standard should not vary more than ± 10 seconds from the RT of the internal standards observed in associated CCV standard.	If a sample area count is outside of the criteria (70-130% of associated CCV or $\geq 50\%$ of the associated ICV), qualify positive results for the homolog groups quantitated using that internal standard and total PCBs as estimated ("J") and qualify "not-detected" results for the homolog groups quantitated using that internal standard and total PCBs as estimated ("UJ"). If extremely low sample area counts ($<35\%$ of the associated CCV or $<25\%$ of the associated ICV) are reported, qualify positive results for the homolog groups quantitated using that internal standard and total PCBs as estimated ("J") and qualify "not-detected" results for the homolog groups quantitated using that internal standard and total PCBs as unusable ("R"). If an internal standard RT varies by more than 10 seconds and no peaks are observed in the sample chromatogram, qualification of data is not necessary. Use professional judgment if peaks are observed in the sample chromatogram.

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Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Retention Time (RT) Windows (See Note #4 for additional information.)	The time (scan number) for initiation of data acquisition with each ion set must be carefully determined from the RTs (scan numbers) of the RT congeners in the PCB Window defining mixture. Approximate relative RTs of calibration congeners and approximate relative RT windows for PCB isomer groups are shown on Table 7 of SOP GEHR680. Absolute RTs of PCB congeners #77, #104, #202, and #189 should not vary by more than ± 10 s from one analysis to the next of the PCB Window defining mixture. (RT reproducibility is not as critical for congeners #1 and #209 as for the other four congeners, which are used to determine when ion sets are changed.)	If the PCB Window defining mixture RTs are not within the specified RT windows, evaluate sample ion current profiles (ICPs) for false positives and false negatives. If a constant drift in RT is observed in the bracketing PCB Window defining mixtures, the direction of the RT drift should be applied to the sample ICPs.

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Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Blanks (See Note #5 and Note #10 for additional information.)	Summarize all results greater than the method detection limit (MDL) present in the blanks (identified by PCB congener). The highest positive result associated with a sample should be utilized for evaluation of contamination.	If a PCB congener is found in the blank but not in the associated sample(s), no action is required. If a PCB congener in a sample is $\leq 5\times$ the blank result, subtract the PCB congener concentration from the associated homolog group and total PCB results (and note in the validation report). If all PCB congeners of a homolog group result are $\leq 5\times$ the blank results, qualify the positive result for the homolog group as “not detected” (“U*”) and subtract the homolog group result from the total PCB result. If the positive result qualified “U*” is $< RL$, the RL should be used as reported. If the positive result qualified “U*” is $\geq RL$, the value of the positive result should be used as the revised RL. If a PCB congener in a sample is $> 5\times$ blank result, no action/qualification is required. If gross contamination exists (<i>i.e.</i>, saturated peaks on the GC/MS), qualify the positive results as unusable (“R”) due to interference.
Surrogates (See Note #6 for additional information.)	Use 60-140% as limits.	If the recoveries of one or more surrogates are $>$ upper limit, qualify positive results as estimated (“J”) and do not qualify “not-detected” results. If the recoveries of one or more surrogates are $<$ lower limit but $\geq 10\%$, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). If the recoveries of one or more surrogates are $< 10\%$, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“UR”).

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Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD) (IF REQUESTED)	Calculated for Total PCBs only. For accuracy, use recovery limits of 60-140%. For precision, use RPD limits of 20% for aqueous samples and 40% for solid samples.	Data should not be qualified due to %Rs (or RPDs calculated on %Rs) that are outside of criteria if the original concentration of Total PCBs is $>4\times$ the spiking level for Total PCBs. RPDs calculated using MS/MSD results can be used to evaluate precision. If the recovery is $>140\%$, qualify the positive total PCB results in the native sample as estimated ("J") and do not qualify the "not-detected" total PCB results. If the recovery is $<60\%$ but $\geq 10\%$, qualify the positive total PCB results in the native sample as estimated ("J") and qualify the "not-detected" total PCB results in the native sample as estimated ("UJ"). If the recovery is $<10\%$, qualify the positive total PCB results in the native sample as estimated ("J") and qualify the "not-detected" total PCB results in the native sample as unusable ("UR"). If the precision exceeds the RPD criterion, qualify the positive total PCB results in the native sample as estimated ("J") and do not qualify the "not-detected" total PCB results. If a field duplicate of the native sample was collected and analyzed, the field duplicate sample should also be qualified if an MS/MSD recovery or RPD is outside of criteria (as stated above for the native sample).
Laboratory Control Samples (LCS)	Calculated for Total PCBs only. For accuracy, use recovery limits of 60-140%.	If the recovery is $>140\%$, qualify positive total PCB results in all associated samples as estimated ("J") and do not qualify "not-detected" total PCB results. If the recovery is $<60\%$ but $\geq 10\%$, qualify positive total PCB results in all associated samples as estimated ("J") and qualify "not-detected" total PCB results in all associated samples as estimated ("UJ"). If the recovery is $<10\%$, qualify positive total PCB results in all associated samples as estimated ("J") and qualify "not-detected" total PCB results in all associated samples as unusable ("UR").

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Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Field/Laboratory Duplicate (See Note #7 and Note #10 for additional information)	Use precision limits of 20% RPD (%RSD for triplicate and quadruplicate analyses) for aqueous samples and 40% RPD (%RSD for triplicate and quadruplicate analyses) for solid samples when sample results are $\geq 5 \times$ RL. Use limit of $\pm$ RL ($\pm 2 \times$ RL for solids) when at least one sample value is $< 5 \times$ RL. (Use one-half the RL as a numerical value for any “not-detected” results in the RPD calculations). Compare both individual total homolog results and total PCB results separately.	If the criteria are not met, qualify positive results for the out-of-criteria total homolog group or total PCBs 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 $\geq 10\%$, 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 $< 10\%$.

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Table for the Validation of PCB Data Generated by US EPA Method 680

Quality Control Item	Usability Criteria	Action
Quantitation and Qualitative Identification (See Notes #4, #8, and #9 for additional information.)	Calculations should be performed in accordance with Section 12 of SOP GEHR680. Samples with results that exceed the instrument calibration range should be reanalyzed at a dilution. EICPs must be evaluated to determine whether the laboratory correctly identified the PCB congeners based upon the identification procedures and criteria defined in Sections 11.3 and 11.4 of SOP GEHR680.	If a PCB congener result exceeds the instrument calibration range, qualify the positive result from the associated homolog group as estimated (“J”). Use professional judgement to determine whether sample reanalyses and dilutions should be compared to the original analyses. If criteria (see field duplicate usability) between the original sample results and the reanalysis sample results are not met, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). Use professional judgement to determine whether qualitative identifications are accurate and whether data qualification is necessary.
System Performance (See Note #9 for additional information.)	Professional judgement should be used when assessing the degradation of system performance during analyses.	Use professional judgement to qualify the data if it is determined that system performance degraded during sample analyses.
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 limitations of the data. If sufficient information on the intended use and required quality of the data is available, include the assessment of the usability of the data within the given context.

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**Notes for the Validation of PCB Data
Generated by US EPA Method 680**

1. Criteria for DFTPP Spectrum

<u>m/z</u>	<u>Relative Abundance</u>
127	40-60%
197	<1%
198	100% (Base Peak)
199	5-9%
275	10-30%
365	>1%
441	Present and <m/z 443
442	>40%
443	17-23% of m/z 442

2. If the initial calibration curve %RSD>50%, the linearity of the first three initial calibration standards should be evaluated. If the first three initial calibration standards for the PCB congener are linear (*i.e.*, $r \geq 0.99$), do not qualify “not-detected” results. If the first three initial calibration standards for the PCB congener are not linear, qualify “not-detected” results as estimated (“UJ”).
3. If instrument instability (*i.e.*, several CCV standards with PCB congeners exhibiting both increasing and decreasing sensitivity throughout an analytical sequence) is observed in the analysis of sequential CCV standards, “not-detected” results may be qualified as estimated (“UJ”) due to instrument sensitivity of a CCV standard response that is greater than the initial calibration standard response (increase in instrument sensitivity).

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**Notes for the Validation of PCB Data
Generated by US EPA Method 680**

If the CCV standard is %D>20% in the direction of increased instrument sensitivity and it is determined that “not-detected” results should not be qualified, the data reviewer should note this within the QAR support documentation.

4. Use professional judgement when evaluating sample ion current profiles (ICPs) when an RT shift is observed. If the ICPs reveal peaks corresponding to PCB congeners of interest using expanded RT windows and the surrogate compounds do not display a similar shift in RT, the concentrations of the PCB congeners that are outside of the RT window are subtracted from the associated total homolog and total PCB results.

If the ICPs reveal peaks that interfere with potential detection of a PCB congener, qualify reported positive results for the associated total homolog group as unusable (“R”).

5. The frequency of equipment/rinse blanks is determined during the sampling event. The results of a equipment/rinse blank should be applied to all samples collected in the same day, unless only one blank was collected for a several-day sampling event. 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 of a contaminant.

If a sample result qualified “U*” is <RL and the laboratory did not report the RL on the data tables or Form I, the positive result (*e.g.*, 8 µg/L) should be replaced with the RL (*e.g.*, 10 µg/L).

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**Notes for the Validation of PCB Data
Generated by US EPA Method 680**

Instrument blank contamination should be applied to samples bracketing the contaminated instrument blank.

6. The surrogate recovery limits do not apply to samples analyzed at greater than five-fold dilutions. Qualification of the data is not necessary if the surrogate is diluted beyond detection. Generally, a greater than five-fold dilution will affect the ability to even detect the surrogate. If a sample was analyzed at a five-fold dilution or less and either surrogate was not detected in the sample, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”). Write a comment in the QAR addressing the issue that sample-specific method performance based on surrogate recoveries could not be evaluated due to the dilution required for sample analysis.
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. Laboratory duplicate results and field duplicate results apply only to the original sample and the laboratory/field duplicate. Soil duplicate results are expected to have greater variance than aqueous duplicate results.
8. If a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the RL (secondary dilution), do not utilize this result when comparing an original analysis and a diluted reanalysis.

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**Notes for the Validation of PCB Data
Generated by US EPA Method 680**

9. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:
- High background levels or shifts in absolute RTs of internal standards
 - Excessive baseline rise at elevated temperature
 - Extraneous peaks
 - Loss of resolution
 - Peak tailing or peak splitting that may result in inaccurate quantitation
10. The RL will be defined on a project-specific basis. If the project-required RL is less than the low calibration standard concentration, the Project Manager should be consulted for instructions about application of qualification related to the RL.