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PCB Analysis
SW-846 Method 8082

PCBs are a class of chemicals with 1 to 10 chlorine atoms attached to biphenyl and a general chemical formula of $C_{12}H_{10-x}Cl_x$. This substitution of chlorine atoms at various positions on the basic chemical structure leads to 209 distinct compounds known as congeners. PCBs are man-made compounds and commercial mixtures of congeners are known as Aroclors. The data validation of PCB analyses is complicated by the various analytical procedures that are used by laboratories for quantification. PCBs can be quantified either as Aroclors or as individual compounds known as congeners using SW-846 Methods 8082 and 8270C respectively. The initial calibration procedures, surrogate standards and the use of internal standards depends on the whether PCBs are quantified as congeners or Aroclors.

1.0	**PCB Data Review - Initial Calibration**	
1.1	**Are PCBs quanitifed as Aroclors or as congeners?** Note: Aroclors are commercial PCB mixtures that are distinguished by a number (e.g., Aroclor 1254) which is based on the number of carbon atoms (usually 12) and the average percentage by weight of chlorine atoms in the chemical mixture. PCB congeners are designated with a distinct chemical name and CAS number. *Action: Record how PCBs are quantified.*	
1.2	**Is dual column confirmation used or some other means of compound confirmation?** Note: Method 8082 (Section 1.5) requires dual column confirmation or confirmation by another mode of analysis. However, Aroclors can be quantified by method 8270C (GC/MS) which does not require dual column confirmation. Action: If confirmation is required (Method 8082) but no information is present, contact the lab for an explanation. If no confirmation data is forthcoming, qualify all detected compounds as estimated and flag the data with a “J”.	
	If the analysis was for Aroclors proceed to question 1.3. If congeners were analyzed for proceed to question 1.10.	

1.3	**If dual column confirmation is used as required for Aroclors (Method 8082), is calibration information given for both columns?** *Action: If data for both columns are not found contact the laboratory for the missing information. If the data is not forthcoming, qualify all data as estimated and flag the detected compounds with a "J" and non-detected compounds with a "UJ". If other information is missing and/or there are other data validation issues, the data may be determined to be unacceptable and consequently rejected and flagged with an "R".*	
1.4	**Was a five point calibration performed using a mixture of Aroclors 1016 and 1260?** Note: The five point calibration mixture contains many of the peaks found in other Aroclors and is analyzed to demonstrate the linearity of the calibration. *Action: If data for both columns are not found, contact the laboratory for the missing information. If the data is not forthcoming, qualify all data as estimated and flag the detected compounds with a "J" and non-detected compounds with a "UJ". If other information is missing and/or there are other data validation issues, the data may be determined to be unacceptable and consequently rejected and flagged with an "R".*	

1.5	**Are the Relative Standard Deviations (%RSD) of the initial Calibration Factors (CFs) of the individual Aroclors in the 1016/1260 calibration mixture below 15% (Method 8082)?** **Alternately, is the average %RSD for all Aroclor standard (1016/1260) peaks 15 percent or less?** Note: The %RSD criteria demonstrates that the calibration is linear over the range of concentrations of the standards. If it is not linear, one or two standards may be dropped from consideration (with a corresponding adjustment in linear range or in quantitation limit) to meet the requirements. The lab may also choose to perform a non-linear calibration. *Action: If the linearity criteria is not met and there is no evidence that an Aroclor was quantified using a non-linear equation, then detected results for that Aroclor are qualified as estimated and flagged with a "J" and undetected results may be qualified with a "UJ" or "R" using professional judgement.*	
1.6	**Was initial calibration data for the other Aroclors included with the report?** Note: Standards of the other Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been used to determine the Instrument response. *Action: If data is absent, contact the laboratory for the missing information. If the data isn't available, the results should be qualified is not forthcoming, qualify all data as estimated or rejected using professional judgement and the DQOs of the project.*	

1.7	**Was a minimum of 3 peaks (preferably 5 peaks) chosen to identify each Aroclor other than Aroclors 1016 and 1260?** Note: The peaks must be characteristic of the Aroclor in question. Peaks are chosen in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. At least five peaks should be used for Aroclors 1016 and 1260. These peaks must be unique to these Aroclors. *Action: Check the number of peaks. If possible, record the peak area (height or response) for each characteristic Aroclor peak to be used for quantitation.*	
1.8	**Optional: Were the Mean Retention Times (RTs) of each of the five major peaks of Aroclors 1016 and 1260 and the Retention Time (RT) of the surrogates correctly determined from the five-point initial calibration?** Note: An RT Window must be calculated as ±0.07 minutes for each of the five Aroclor peaks used to identify Aroclors 1016 and 1260. Tetrachloro-m-xylene (TCX) must use a RT window of ±0.05 minutes and decachlorobiphenyl (DCB) must use an RT window of ±0.10 minutes. *Action: If RT windows were not determined correctly or did not meet the acceptance criteria, the identification of the Aroclor is suspect. Review the data and initially qualify the Aroclor(s) in question as estimated (flag data as "J "or "UJ"). If additional data quality problems are encountered then, using professional judgement, data may be rejected and flagged with an "R".*	

1.9	**Optional: For the other Aroclors, 1221, 1232, 1242, 1248, 1254 (sometimes 1262 or1268), the relative RTs of each of the three to five major peaks and the relative RT of the surrogates are determined from the single-point standard initial calibration. If Aroclors 1221, 1232, 1242, 1248, 1254 (sometimes 1262 or 1268), are detected in a sample, the relative RTs of each of the three to five major peaks and the RT of the surrogates are determined from the five-point initial calibration.** Note: An RT Window must be calculated as ±0.07 minutes for each of the three to five Aroclor peaks and ±0.05 and ±0.10 minutes for the surrogates tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB), respectively. *Action: If the RT windows were not determined correctly or did not meet the acceptance criteria, the identification of the Aroclor is suspect. Review the data and initially qualify the Aroclor(s) in question as estimated ("J" or "UJ"). If additional data quality problems exist then, using professional judgement, reject the data.*	Proceed to Section 2.0
	PCBs determined as congeners	
1.10	**Was the instrument calibrated using at least five concentration levels of PCB congener standards using the internal standard Decachlorobiphenyl (DCB)?** *Action: If 5 standards were not used, contact the lab for an explanation. Results may be qualified using best professional judgement. Usually, analyses are acceptable if three standards are used which include the low level standard. If the low level standard is not used, then all non-detected congeners should be qualified as estimated and flagged "UJ" or rejected "R" depending on the calibration standards that were used and the DQOs of the project.*	
1.12	**Do the average response factors (RFs) determined from the 5 initial calibration standards meet the minimum ratio for quantification?** Note: This criteria is not defined in the method. However, method 8000 requires a minimum RF of 0.05. *Action: quantify all congeners that do not meet the minimum response factors as rejected and flag the results with an "R".*	

1.14	**Are the percent Relative Standard Deviation (%RSD) of the RFs for <u>each</u> congener in the initial calibration standard below 20% (method 8000)?** **Is the <u>average</u> %RSD for <u>all</u> congeners 20 percent or less?** Note: The %RSD criteria demonstrates that the calibration is linear over the range of concentrations of the standards. If it is not linear, one or two standards may be dropped from consideration (with a corresponding adjustment in linear range or in quantitation limit) to meet the requirements. The lab may also choose to perform a non-linear calibration. *Action: If the linearity criteria is not met and there is no evidence that a congener was quantified using a non-linear equation, then detected results for that congener are qualified as estimated and flagged with a "J" and undetected results may be qualified using professional judgement.*	
1.15	**Were relative retention time (RRTs) windows calculated for each congener, internal standard and surrogate?** *Action: If the data is missing contact the laboratory for results. If the data is not forthcoming, do not qualify data at this time. However, if retention time information is not present with continuing calibration data, qualify results as estimated (flag data with a "UJ" or "J"). The omission of this data is a data quality objective concern and consequently further qualification may be necessary.*	

2.0	PCB Data Review - Continuing Calibration Verification (CCV)	
Questions 2.1 to 2.4 pertain to the analysis of Aroclors. Precede to Question 2.5 if the analyses quantify PCBs as congeners.		
2.1	**Was a CCV analyzed within twelve hours of the analyses of samples being validated?** Note: A CCV must be performed at the beginning (opening CCV) and end (closing CCV) of the analytical sequence for method 8082. The opening and closing CCVs consist of an injection of a blank followed by an injection of the mid-point concentration Aroclor 1016/1260 standard mixture. If an Aroclor other than 1016 or 1260 is detected in any sample, that detected Aroclor must have a mid-point concentration standard analyzed as part of the opening and closing CCV *Action: If a CCV was not analyzed within 12 hours of the samples, then qualify data according to professional judgement. All data analyzed outside of 14 hours of the CCV analysis could be regarded as rejected and flagged with and "R".*	
2.2	**Check the retention time data for the Aroclors analyzed in the CCV and determine whether they fall within the criteria set from the initial calibration (see question 1.8 for criteria)**. *Action: If the retention time of the Aroclors fall outside of the retention time criteria, the reviewer should flag each non-detected as estimated ("UJ"). The data should be used to check for false positives. If a peak is found to lie close to the new retention time window, that positive result should be tentatively considered a false positive and the results flagged with a "U".*	
2.3	**Were the Percent Difference (%D) of each Calibration Factor (CF_V) from the five peaks in the CCV (mid-level 1016/1260 mixture) within ±15% of the mean CFs from the initial calibration?** Note: $\%D = \left(CF_V - \overline{CF}\right) \div \left(\overline{CF}\right) \times 100$ *Action: If the Percent Difference is not within ±15%, qualify associated detections as estimated and flag the data with a "J". Non-detections are considered estimated and data should be flagged with an "UJ".* According to Method 8082, if the CCV is outside of the 15% criteria, the laboratory should have performed corrective action.	

2.4	**Dual column confirmation is a requirement of Method 8082. Were the retention time windows and %D of each identified Aroclor within criteria (e.g. RT within ± 0.07 minutes of the initial calibration and %15 difference between columns**). *Action: If either of these requirements are outside of the criteria, the confirmation of the Aroclor is suspect. Review the chromatograms and determine whether the Aroclor was correctly identified. If not, qualify the questionable result as undetected (flag the data with a "U" or an "UJ") or based upon professional judgement qualify a detected result from a single column as estimated and flag the result with a "J".*	
2.5	**For congener quantification, was the initial calibration verified (CCV) within 12 hours of sample analysis?** *Action: check the run log and determine if the samples being verified were analyzed within 12 hours. If not, samples analyzed between 12 and 14 hours should be qualified as estimated and flagged with a "U" or "UJ". Samples analyzed outside of 14 hours should be qualified as rejected and flagged with an "R".*	
2.6	**Was the response (area counts) of the internal standards in the CCV within ±50% of the average internal standard in the initial calibration?** *Action: if the internal standard response is outside of the criteria, the results should be rejected and all data flagged with an "R".*	
2.7	**Was the relative retention time (RRT) of the congeners and internal standards within 0.06 relative retention units of the initial calibration?** *Action: If the relative retention times are outside of the criteria, all analyses should be initially determined to be estimated and the data should be flagged with a "U" or "UJ". The chromatogram should be checked and determined if there is a likelihood of a false positive or negative. After this analysis, the data may be further qualified based upon professional judgement.*	

2.8	**Were the Percent Difference (%D) of each Response Factor (RF_v) from the CCV within ±15% of the mean RFs from the initial calibration?** Note: $\%D = (RF_v - \overline{RF}) \div (\overline{RF}) \times 100$ *Action: If the Percent Difference is not within ±15% qualify associated detections as estimated and flag the data with a "J". Non-detections are considered estimated and data should be flagged with an "UJ". According to Method 8082, if the CCV is outside of the 15% criteria, the laboratory should perform corrective action.*	

3.0	PCB Data Review - Blanks	
A method blank must be extracted each time samples are extracted using the same procedures and reagents. A method Blank should be analyzed after the 12-hour CCV and prior to sample analysis. In addition, a method blank should be analyzed per every batch of 20 samples. Some samples may need cleanup procedures such as sulfur cleanup. These procedures are referenced in SW-846 3600C. If these cleanup procedures are used, a specific cleanup blank must be reviewed and the results verified.		
3.1	**Was a Method Blank analyzed prior to sample analysis and are the results reported?** *Action: Check the results to determine whether a method blank was analyzed and that the results of this blank are reported. If the data is not present, contact the laboratory for the information. If the data is not forthcoming, do not qualify non-detected Aroclors. Detected Aroclors may provisionally be qualified as estimated based and flagged with a "J" based upon professional judgment.*	
3.2	**Is there an indication that the samples associated with that blank were diluted?** Note: The dilution factor can be found in the data report (a dilution factor of 1 indicates no dilution).	
3.3	**Do any method/field/trip/rinsate/cleanup blanks have any positive results for any Aroclors or congeners? Were the same congeners or Aroclors found in the samples? List those analytes and the results that are both found in the blanks and samples. These analytes are subject to qualification.** Note: A list of samples associated with each of the contaminated blanks should be prepared. Field blank results should be used to qualify data. Trip blanks are used to qualify samples based on potential contamination during shipment, and are not required for non-aqueous matrices. *Action: Follow the directions in the table below to qualify sample results due to blank contamination. Use the largest value from all of the associated blanks. If any blanks are grossly contaminated, all data associated may be qualified as "R", based upon professional and the project's DQOs.*	

3.4 **For those Aroclors or congeners identified in question 3.3, follow the directions in the following table to qualify data.** Note: If analytes are detected in a blank but not in the sample of interest, then no qualification is necessary. Use the information from 3.2 to determine whether a dilution factor should be used to determine qualification. When a dilution is applied to samples, the contaminant concentration in the samples are divided by the dilution factor, then use the criteria listed in Table to qualify blanks and sample data.	

Criteria	Action:
Sample Conc. > Detection Limit but < 5x Blank Result	**Identify the sample result "U" undetected**
Sample Conc. < Detection Limit & < 5x Blank Result	**Report the detection limit and qualify result as estimated, "UJ", undetected**
Sample Conc. > Detection Limit & > 5x Blank Result	**No qualification is necessary**

4.0 PCB Data Review - Laboratory Control Sample (LCS)	
Laboratory Control Sample **An LCS should be included with each batch of samples (approximately 20). The LCS consists of an aliquot of a clean (control) matrix similar to the matrix type of the sample with the same weight or volume. The LCS is spiked with the same Aroclors or congeners at the same concentration as the matrix spike (i.e. mid-calibration concentration). When the results of the matrix spike indicate a potential problem due to the sample matrix itself, the LCS verifies that the laboratory can perform analyses in a clean matrix (Method 8000C).**	
4.1 Was an LCS prepared, extracted, analyzed and reported once per batch or every twelve hours (which ever comes first)? Note: This information should be included in the report provided by the lab. If not, contact the laboratory and request that the information be submitted to the agency. This information should be found in the run log. *Action: If LCS information cannot be found, consult the facility for re-submittal of the data package. If LCS information is not present, qualify all positive results as estimated and flag the data with a "J". If warranted, the Data Validator may reject all results as unacceptable.*	
4.2 Does the LCS contain the following compounds and surrogates? **Aroclor 1016, Aroclor 1260** **Tetrachloro-m-xylene (TCMX; surrogate)** **dichlorobiphenyl (DCB; surrogate).** **If congeners are quantified, the same set of congeners used in matrix spike are used in the LCS.** Note: Method 8082 and 8000C calls for the LCS to be spiked at the same level as the matrix spike. When specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for spiking in the LCS (Method 8082, Section 8.4.1). *Action: If the specific Aroclors or congeners are not contained in the LCS, contact the laboratory for an explanation. Use the information in applying professional judgement to qualify the sample results.*	

4.0 PCB Data Review - Laboratory Control Sample (LCS)	
4.3 **Do the percent recoveries (%R) meet the QC limits set by the laboratory?** Note: The Aroclors 1016 and 1260 should have recovery criteria between 70% to 130% (Method 8000) unless specific laboratory criteria is established. *Action: If the LCS recovery is greater than the upper acceptance limit, then positive sample results for the affected compound(s) should be qualified as "J+". If the LCS recovery is lower than the lower acceptance limit, associated target compounds should be qualified as estimated and flagged as "J-". Recoveries which are much lower than lower acceptance limit may warrant rejection and data should be flagged with an "R".* *For congener analyses, if the mass spectral criteria are met, but the LCS recovery is less than the lower acceptance limit, then the associated detected target compounds should be qualified as estimated and flagged with a "J-", and the associated non-detected target compounds should be qualified as rejected and flagged with an "R".* *If more than half of the compounds in the LCS are not within the recovery criteria, then all of the associated detected target compounds should be qualified as estimated and flagged with a "J" and all associated non-detected compounds should be qualified as rejected and flagged with an "R."*	**List compounds and sample IDs that do not meet QC limits:**
4.4 **Verify the calculations for at least one %R.** ***%R = found/true X100*** *Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgement to determine if the lab should be contacted for re-submittal or if the data should be flagged.*	

5.0 PCB Data Review - Matrix Spike/Matrix Spike Duplicates	
Matrix Spike/Matrix Spike Duplicates **Matrix spike and matrix spike duplicates (MS/MSD) are performed to assess the influence of the matrix on accuracy and precision for PCB analyses. Matrix spikes and duplicates are required for every batch of samples (i.e. every 20 samples). The validator should be aware that the MS/MSD are batch specific, not sample specific. For example, the MS/MSD information may be performed on any sample in the batch, but not necessarily a sample being validated. Because of this, matrix spike and matrix spike duplicate data alone are not used to qualify results, but the information is used with other QA/QC data to qualify data.**	
5.1 **Is matrix spike/matrix spike duplicate recovery data present?** *Action: If any matrix spike/spike duplicate data is missing, the laboratory should be contacted for a re-submittal.*	
5.2 **Do the spikes used for the matrix spike/spike duplicate contain the same compounds and same spiking concentration as used for the LCS?** *Action: If the same compounds were not used or if the concentrations of the spikes are different, then contact the laboratory for an explanation. If the explanation is unsatisfactory continue with the MS/MSD review and assign qualification to the data if necessary.*	
5.3 **How many Aroclor or congener spike recoveries are outside the QC limits set by the laboratory?** *Action: review the LCS data and quantify data based upon the LCS results. See question 4.3.*	**Record the spike recovery and control limits:**
5.4 **How many RPDs for matrix spike and matrix spike duplicate recoveries are outside the QC limits?** Note: The MS/MSD results may be used in conjunction with other QC criteria to determine the need for data qualification. Outliers should be identified.	**Record the recovery data out of criteria and control limits. Review surrogate and LCS data to determine if qualifiers are necessary:**

6.0 PCB Data Review - Surrogate Recovery		
PCB Surrogate Compound Recovery **Surrogate compounds are added to every sample, standard and blank. The recovery of surrogate compounds allows an assessment of accuracy in a given sample. PCB surrogate recoveries are used with other QA/QC data to qualify sample results and to justify laboratory re-analysis.**		
6.1	**Is surrogate recovery data for Aroclors or congeners present?** Note: Two surrogates, tetrachloro-m-xylene (TCMX) and dichlorobiphenyl (DCB) should be added to every standard, blank, LCS, MS/MSD and sample and data should be provided for all samples. *Action: If no data is present, contact the laboratory for an explanation and re-submittal of the data. If data is not forthcoming, initially qualify the data as estimated. Professional judgement may be used to reject sample results.*	
6.2	**Were surrogate outliers marked correctly (based upon the laboratory's criteria)?** *Action: Mark, circle or highlight the suspected outliers.*	**List the sample ID(s), matrix(-ces) and parameter(s):**
6.3	**If any surrogate compounds were out of compliance was re-analysis performed to confirm a matrix interference?** Note: Check the report narrative for an indication of re-analysis. *Action: If a surrogate is above the upper control limit, all positive results should be qualified as estimated and flagged "J+". Results listed as non-detected should not be qualified.* *If any surrogate recovery is less than the lower criteria, but greater than or equal to 10% recovery, all detected compounds should be qualified as estimated and flagged with a "J-" and all non-detected compounds flagged with a "UJ".* *If any surrogate compound recovery is less than 10%, all detected compounds should be qualified as estimated and flagged with a "J-" and all non-detected compounds should be rejected and flagged with an "R".*	**List sample ID(s) for surrogate compounds out of compliance and criteria:**