` **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 <u>individual</u> Aroclors in the 1016/1260 calibration mixture below 15% (Method 8082)?** **Alternately, is the average %RSD for <u>all</u> 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 $\pm$15% of the mean CFs from the initial calibration?

Note: