SOP 31	Revision: 11	Supersedes: Rev10
PREPARATION AND ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBS) BY METHOD 1668A/C		
Analyst Review:		
Management:		
Quality Assurance:		
Effective Date: 26 August 2013		

Revision	Description of Revision
0	Renumbered and reformatted.
1	Updated RLs and Calibration Levels in Tables 1 and 2. Inclusion of MS/MSD or SD for DOD QSM clients in Section 12.3.1.
2	Added Gas Chromatograph info to Section 8. Section 13.6.2 was reworded to include corrective action. Combined the preparation and analytical SOPs for this method. Update company name.
3	DoD criteria added to Table 1, clarified the CRS step in Section 11, delete section 3.2.7, update Table 6; added Sections 11.1.4 and 12.5; Added Figure 2
4	Method modification added 3.2.11, and 12; minor editing
5	Added procedure for extraction of aqueous samples containing sediment to Section 11
6	DI Water references have been changed to HPLC Water; updated method mods; solvent change in 11.1.2; PFK frequency and mass clarification; added the column; added Section 19
7	Cleanup column pre-elutions; SPB-Octyl column ICAL procedure; reagent kilning times and temperatures
8	Minor edits; deleted gravity filter section; revised aqueous with particulate extraction
9	Revised PCB-177 Structure; update coeluters in tables; add centrifuge option; instrument maintenance reference, expanded ICAL range and additional IS
10	Modified OPR/IS/RS list; revised final volume for aqueous, expanded calibration curve. Add DRBC requirements.

11	Section 3.2.17 has been modified to include chemist's professional judgment. Added Section 3.2.18 and 3.2.19. Revised GC column from DB-1 to ZB-1 in section 7.9. Added tetradecane to Section 8.2. Added Section 9.3: Storing frozen samples. Added Section 10.3: Thawing frozen samples. Changed 40 µl C_{14} to 10 µl C_{14} in Section 13.2.1. Minor spelling errors corrected. SPB-Octyl column criteria were added to Section 15.2.2. Added Section 15.3.6 resolution criteria. Section 15.4.1 injection volume will be the same for standards and samples. Deleted Section 15.7 and re-numbered following sections. Revised Section 15.8.1 and added Section 16.5 for the optional reporting of EMPC results. Revised Section 15.9.5. Added Section 20.7 to reference EPA Method 1668C. Replaced references to IUPAC naming to Congener Number to be consistent with EPA Method 1668C. Revised notes at the end of Table 1. Modified Fish /Tissue Quantitation Limits, changed amount extracted for fish/tissue Matrix to 10 grams, added Quantitation Limits for Serum (pg/g) and Serum (pg/g-Lipid) and final volume for all matrices in Table 1. Coeluting PCB compounds names added to all tables. Updated concentration of calibration standards for the dichlorobiphenyls in Table 2. Co-eluting compounds 118/106 order changed in all tables. Co-eluting PCB concentrations have been updated in Table 2. Compounds have been replaced to reflect the correct list of analytes in Table 2. Ratios and control limits for chlorine atoms 6, 7 and 10 have been revised in Table 3. QL for Water and final volume of 20 µl for water and tissue matrices have been revised in Table 4. Table 5 has been revised to reflect the correct analytes and limits. Added ion abundance criteria for the ^{13}C-Heptachlorobiphenyl and ^{13}C-Nonachlorobiphenyl internal standards. Co-eluting compounds names added to Table 7. Definitions: removed PAH and added PCB. Added Table 8: PCB Natives and Corresponding Labeled Compounds. Added Amendment for State of Wisconsin specified criteria

1. PURPOSE

1.1. This procedure describes the preparation and analytical techniques used for the analysis of samples for polychlorinated biphenyls (PCBs) by EPA Method 1668.

2. SCOPE

2.1. This method is for the determination of the polychlorinated biphenyl (PCB) congeners at the quantitation limits listed in Table 1 for water, soil, sediment, sludge, ash, tissue, and other sample matrices by gas chromatography/high resolution mass spectrometry/selective ion monitoring (HRGC/HRMS/SIM). The quantitation limits for this determination are provided in Table 4.

2.2. The initial calibration curve (ICAL) contains all 209 native PCB congeners. For sample analysis, those PCBs resolved as separate congeners (see Table 1) are quantified using the congener–specific relative response factor (RRF) from the ICAL. Co-eluting congeners are quantified using the RRF based on the sum of the co-eluting congeners as calculated from the ICAL.

2.3. The MDLs represent the individual congener being reported, unless the congener co-elutes with one or more other congeners. In those cases, the values represent the sum of the concentrations of the co-eluting congeners.

2.4. The reporting limits and quantitation levels are usually dependent on the level of interferences rather than instrumental limitations. The quantitation limits listed are levels at which the PCBs can be determined with only common laboratory interferences present.

2.5. Detection limits are sample-specific and congener-specific.

3. METHOD MODIFICATIONS

3.1. This method is "performance-based". Modifications to the method to overcome interferences or lower the cost of measurements are permitted, provided that all performance criteria in this method are met.

3.2. The following modifications to the method were made to improve efficiency and accuracy:

3.2.1. Vista’s initial calibration curve includes PCB congeners listed in Table 1, instead of the 27 congeners included in the method.

3.2.2. According to Section 12.2.1.1 in USEPA Method 1668A, a pre-filter GMF 150 is used to remove any particles. To prevent possible contamination, Vista does not use a pre-filter in disk preparation.

3.2.3. According to Section 12.2.2.1 in the method, the sample bottle is rinsed with reagent water prior to methylene chloride. Vista rinses the sample bottle with portions of methylene chloride.

3.2.4. Under the separatory funnel extraction, the separatory funnel is rinsed with 20 mL of methylene chloride. Then the methylene chloride is placed through the sodium sulfate into the concentrator. Vista eliminated the methylene chloride rinse since sufficient rinsing of the separatory funnel is performed.

3.2.5. The method specifies that the tissue and sodium sulfate is to equilibrate for 12-14 hours prior to soxhlet extraction. Vista uses a shorter equilibration time to prevent solidification of the thimble contents. The Dean Stark apparatus removes any excess water.

3.2.6. In the back-extraction with base and acid, Vista uses sodium hydroxide rather than potassium hydroxide.

3.2.7. The silica gel column is not pre-eluted with methylene chloride prior to hexane.

3.2.8. In the silica gel cleanup, the container is rinsed with methylene chloride instead of hexane due to the extensive preparatory cleaning of the silica gel.

3.2.9. Vista's CS-3 standard includes the PCB congeners listed Table 1; therefore, injection of the combined 209 congener solution (15.4.2.1) is not necessary.

3.2.10. Additional internal standards are added to the solutions for more accuracy in quantitation.

3.2.11. Soil samples are kept in the refrigerator rather than freezer to ensure glass containers do not break.

3.2.12. Standard vials are not marked to detect evaporation.

3.2.13. Acid alumina cleanup may be used for coplanar analysis.

3.2.14. Tetradecane is used as a final solvent for coplanar analysis only

3.2.15. Neither corn nor other oils are used as a blank matrix for tissues.

3.2.16. The pH of aqueous samples is adjusted to 2-3.

3.2.17. High pressure filtration procedure is used for aqueous samples

containing high levels of sediment/particulate based upon the chemist's professional judgment.

3.2.18. Method 1668A requires a signal to noise ratio of 10:1 for all CB's in the initial calibration. Vista's criteria is 2.5:1 for the Di-CB's.

3.2.19. Vista monitors (m)/(m+2) for ^{13}C-HeptaCB's and (m+4)/(m+6) for the ^{13}C-nona-CB's. There are less interferences observed for these masses.

4. DEFINTIONS

4.1. Definitions are presented in Glossary.

5. CONTAMINATION AND INTERFERENCES

5.1. All materials used in the analysis shall be demonstrated to be free from interferences by running reference matrix method blanks with each sample batch.

5.2. The reference matrix should simulate, as closely as possible, the sample matrix under test. Reagent water can be used to simulate water samples; playground sand or white quartz sand can be used to simulate soils; filter paper can be used to simulate papers or similar materials.

5.3. Interferences co-extracted from samples will vary from source to source, depending on the site being sampled. The cleanup steps can be used to reduce or eliminate such interferences.

6. SAFETY

6.1. Procedures shall be carried out in a manner that protects the health and safety of all Vista employees, including the appropriate use of Personal Protective Equipment and engineering controls.

6.2. Each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. Only highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks should handle all compounds or reagents.

6.3. Each chemical compound should be handled in well-ventilated, controlled access laboratories.

6.4. Additional health and safety information can be obtained from material safety data sheets (MSDSs) available to all personnel involved in these analyses.

6.5. In the event of a known or potential compromise to the health and safety of a Vista associate, all work must stop and the incident reported immediately to management.

7. APPARATUS AND MATERIALS

7.1. Amber glass bottles, 1 liter & 500 mL (Teflon-lined screw cap)

7.2. Analytical Balance, capable of reading to 0.0001 g

7.3. Aquacheck and pH strips

7.4. Compaq Personal Work Station 433/500

7.5. Neslab HX200 and HX500 Water Cooler

7.6. Micromass Autospec Ultima High Resolution Mass Spectrometer (M390, M467, M504, M529, M651)

7.7. Hewlett Packard 6890 Gas Chromatograph

7.8. CTC Autosampler Model A200S

7.9. ZB-1 (Phenomenex, or equivalent) column

7.10. Drying oven, VWR Model 1320 or equivalent

7.11. Glass columns

7.12. Glass wool

7.13. Organomation 24-Station N-Evaporator

7.14. Pre-cleaned Glass fiber thimbles - coarse

7.15. Rotary evaporator

7.16. Round bottom flasks: 50, 100, 250, and 500 mL

7.17. Separatory funnels, typically 250 mL to 2-L size

7.18. Soxhlet/Dean-Stark (SDS) Extractor

7.19. Teflon boiling chips

7.20. Test tubes plus Teflon lined caps, 16 mm x 125 mm

7.21. Whatman GF/C, GF/D, and GF/F filters

8. REAGENTS, SOLVENTS AND STANDARDS

8.1. Reagents (Highest purity available)

8.1.1. Activated Silica Gel, for ~5 hours at 550^{o}C

8.1.2. Anhydrous sodium sulfate (Na_2SO_4) kilned for ~5 hours at 550^{o}C, granular

8.1.3. Hydromatrix

8.1.4. Ottawa sand, kilned for ~5 hours at 550^{o}C

8.1.5. Sodium hydroxide, 10N

8.1.6. Sulfuric acid, concentrated

8.1.7. Ultra-pure nitrogen gas

8.1.8. Water, HPLC

8.2. Solvents (Highest purity available)

8.2.1. Acetone

8.2.2. Ethanol

8.2.3. Hexane

8.2.4. Methanol

8.2.5. Methylene chloride (DCM)

8.2.6. Nonane (C_9)

8.2.7. Toluene

8.2.8. Tetradecane (C_{14})

8.3. Standards

8.3.1. All analytical standards are obtained from a certified vendor.

8.3.2. See current spike sheet for spiking concentrations and solutions.

8.4. See SOP 15 for preparation of reagents, standards and documentation.

9. SAMPLE COLLECTION, PRESERVATION, STORAGE AND HOLDING TIMES

9.1. Extract samples within 1 year and analyze within 1 year

9.2. Store samples at 4^{o}C (except fish/tissue at <-10^{o}C). Store extracts at <-10^{o}C in the dark.

9.3. If applicable, samples to be stored frozen are checked for adequate headspace prior to storage in case expansion occurs during freezing.

9.4. If residual chlorine is detected in an aqueous sample, add 80 mg sodium thiosulfate per liter.

9.5. Adjust to pH 2-3 with sulfuric acid (aqueous only).

10. SAMPLE PREPARATION

10.1. Residual Chlorine Determination (aqueous only)

10.1.1. Obtain an Aquacheck strip and place it directly into a small amount of sample in a disposable weigh boat. Move the strip back and forth for 30 seconds.

10.1.2. Check the color on the strip against the color chart on Aquacheck container.

10.1.3. If there is chlorine present, add 80 mg of sodium thiosulfate.

10.1.4. Record procedure on extraction benchsheet

10.2. pH Determination (aqueous only)

10.2.1. Obtain a pH strip and place it directly into a small amount of sample in a disposable weigh boat. Move the strip back and forth for 30 seconds.

10.2.2. Check the color on the strip against the color chart on the pH container. Adjust the pH to 2-3 with sulfuric acid if necessary.

10.3. Thawing Frozen Samples

10.3.1. Remove the sample from the freezer and place under the hood under ambient temperature.

- If there is a concern that the sample container may break, place the container in a pre-cleaned secondary container.

10.4. Once the sample has thawed completely, proceed with sample preparation

10.5. Homogenization and Fillets

10.5.1. Remove any obviously extraneous materials and homogenize sample prior to sub-sampling for extraction. Homogenization may be as simple as shaking the container vigorously by hand to crushing, chopping, and use of a mechanical grinder. Air-dry and homogenize prior to sub-sampling for extraction

10.5.2. Fish Samples: Each whole fish filet is cut into dorsal/ventral strips ~2cm wide and shuffled prior to placing through a grinder to ensure proper homogenization unless the client requests otherwise. Once each fish filet is homogenized, the entire ground fish filet is mixed with the other designated ground fish filets to make a composite. The entire composite is placed in an amber glass jar for extraction. By client request, whole fish may be either homogenized or filleted prior to homogenization. All grinding parts and components are cleaned prior to homogenization and between each sample.

10.5.3. Wash with soap and water and rinse with HPLC water.

10.5.4. Solvent rinse in the following order: Acetone $\rightarrow$ toluene $\rightarrow$ hexane $\rightarrow$ methylene chloride

10.6. % Solids Determination

10.6.1. "ZERO" or "TARE" the balance.

10.6.2. Place a weigh boat on the balance and record the weight as "Boat Weight".

10.6.3. Samples are individually homogenized with a clean spoon, Scoupula or spatula. Add a portion of the sample (2 – 10 g) to the weigh boat and record the weight as "Wet Wt. + Boat Wt."

10.6.4. Place the weigh boat plus sample in an oven at 110±5^{o}C for at least overnight.

10.6.5. Remove the weigh boat plus sample from the oven and allow to come to room temperature.

10.6.6. "ZERO" or "TARE" the balance.

10.6.7. Place the weigh boat plus sample on the balance and record the

weight as “Residue + Boat Wt.”

10.6.8. Calculate the percent solids by the following formula:

$$\%\text{Solids} = \frac{(\text{Residue Wt.} + \text{Boat Wt.}) - (\text{Boat Wt.})}{(\text{Wet Wt.} + \text{Boat Wt.}) - (\text{Boat Wt.})} \text{ X } 100$$

10.6.9. For aqueous samples, if %Solid exceeds 1%, remove any rocks or stones and homogenize the sample prior to extraction. Weigh 10 grams dry-weight equivalent for extraction.

10.7. Compositing – by client request

10.7.1. Samples are individually homogenized, if necessary, with a clean spoon, Scoupula or spatula. Aqueous samples should be mixed and shaken to obtain a representative sample.

10.7.2. Weigh out approximately 50 grams, or amount designated by the client, from each individual sample and place into a pan.

10.7.3. Repeat the homogenization for each sample.

10.7.4. Place each individual sample into a new, separate container. Record the weight of each sample on the benchsheet.

10.7.5. Retain the original sample containers. The new container is given a new sample ID number and then processed through the appropriate extraction.

10.8. Sample Weight Determination

10.8.1. Volumetric: Allow sample to come to ambient temperature, mark the water meniscus on the side of the 1 L sample bottle. Once the sample has been transferred, fill the sample bottle to the mark with water and transfer to a 1000 mL graduated cylinder. Record the sample volume to the nearest 5 mL.

10.8.2. Gravimetric: Sample bottle including sample is placed on calibrated balance. The weight is recorded. The bottle is allowed to air-dry overnight and then re-weighed on a calibrated balance. This weight is recorded and percent solids are determined.

11. EXTRACTION PROCEDURES

11.1. Aqueous Samples Without Sediment/Particulates

11.1.1. Record the combined weight of the bottle, cap and sample for each sample to be extracted. After the sample has been removed from the bottle, allow it to drain overnight and reweigh it and the

cap to determine the amount of sample extracted.

11.1.2. For the method blank (MB) and OPR(s), transfer ~1 liter of HPLC water into a one liter bottle for each.

- Add the appropriate volume of Internal Standard (IS) solution to a test tube containing ~1 mL of acetone. Quantitatively transfer to the aliquot of matrix with small portions of the solvent used. Add the appropriate volume of Native Standard (NS) solution to a test tube containing the IS/solvent and then quantitatively transfer to the aliquot of matrix assigned as an LCS, OPR, MS or MSD. Allow the spiked samples to equilibrate for at least 1 hour before extraction.

11.1.3. Pour the sample filtrate into a 2-liter separatory funnel. Rinse the sample filtrate container with ~60 mL of DCM and add it to the separatory funnel.

11.1.4. Stopper each separatory funnel and shake vigorously, with frequent venting, for 2 minutes.

11.1.5. Allow the phases to separate (centrifugation or other mechanical means may be used to facilitate separation).

11.1.6. Drain the DCM extract through a funnel of Na_2SO_4 into a 500 mL round bottom flask.

11.1.7. Extract the aqueous phase with two more ~60 mL portions of DCM (shaking 1 minute each time) and pass the extracts through the Na_2SO_4 into a round bottom.

11.1.8. Concentrate the extract to approximately 10 mLs.

11.2. Aqueous Samples Containing Sediment/Particulate

Option 1

11.2.1. Record the combined weight of the bottle, cap and sample for each sample to be extracted. After the sample has been removed from the bottle, allow it to drain overnight and reweigh it and the cap to determine the amount of sample extracted.

11.2.2. For the method blank (MB) and OPR(s), use 5 grams of sand for each.

- Add the appropriate volume of Internal Standard (IS) solution to a test tube containing ~1 mL of acetone. Quantitatively transfer to the aliquot of matrix with small portions of the solvent used. Add the appropriate volume of Native Standard (NS) solution to a test tube containing the IS/solvent and then quantitatively transfer to the aliquot of matrix assigned as an

LCS, OPR, MS or MSD. Allow the spiked samples to equilibrate for at least 1 hour before extraction.

11.2.3. Pre-clean 125mm G/F-F and G/F-D filters using Buchner funnel and DCM.

11.2.4. Four solvent-rinse all parts of the HPF apparatus.

11.2.5. Assemble HPF apparatus using one each of DCM-cleaned 125mm G/F-F and G/F-D filters.

11.2.6. Turn main nitrogen valve on with the flow rate set at 40 psi.

11.2.7. Pre-filter with about 10-15 mLs of HPLC water and discard (check canister for leaks).

11.2.8. Place a labeled beaker under HPF spout, slowly add sample to canister using a funnel. Rinse sample container once with a-small amount of acetone, then 2 more rinses with HPLC water.

11.2.9. Remover beaker (save contents) and place sample container under spout.

11.2.10. Begin filtering, adjust filtering rate using valve on the HPF canister (main valve at 40 psi).

11.2.11. Add contents of beaker back to sample container with filtrate, replace beaker under spout, disassemble HPF apparatus, lift filters off the platform and collect any remaining water and combine with filtrate. Quickly transfer filters to soxhlet for toluene SDS extraction as in section 11.3

11.2.12. Pour filtrate into 2L separatory funnel and extract as in section 11.1

11.2.13. Combine aqueous and filter extracts and spike with CRS.

11.2.14. Continue with clean-up procedures.

Option 2

11.2.15. Record the combined weight of the bottle, cap and sample for each sample to be extracted. After the sample has been removed from the bottle, allow it to drain overnight and reweigh it and the cap to determine the amount of sample extracted.

11.2.16. For the method blank (MB) and OPR(s), use 1 gram of sand for each.

- Add the appropriate volume of Internal Standard (IS) solution

to a test tube containing ~1 mL of acetone. Quantitatively transfer to the aliquot of matrix with small portions of the solvent used. Add the appropriate volume of Native Standard (NS) solution to a test tube containing the IS/solvent and then quantitatively transfer to the aliquot of matrix assigned as an LCS, OPR, MS or MSD. Allow the spiked samples to equilibrate for at least 1 hour before extraction.

11.2.17. Pour entire sample into a solvent rinsed centrifuge bottle. Rinse the sample container with HPLC water, and then rinse sample container with a small portion of DCM.

11.2.18. Centrifuge the samples at 3,000 rpm for 15 – 20 minutes.

11.2.19. Pour the aqueous portion into a separatory funnel and extract as in Section 11.1 above.

11.2.20. Transfer the sediment portion into kilned thimbles. Rinse the centrifuge container 2-3 times with toluene and transfer to thimble.

11.2.21. Extract sediment portion with toluene in SDS apparatus as in Section 11.3 below for 16-24 hrs.

11.2.22. Combine aqueous and sediment extracts, and spike with CRS

11.2.23. Continue with Clean-Up procedures.

- For DRBC samples, extract 2-4 liters of sample.

11.3. Soil, Sediment, Solids, Clay

11.3.1. Samples are individually homogenized with a clean spoon, scoupula or spatula. Weigh the sample (nominal 10 g dry weight equivalent) directly into kilned thimble, carefully breaking up any large lumps of sample.

- Add the appropriate volume of Internal Standard (IS) solution directly to the thimble. Add the appropriate volume of Native Standard (NS) to the thimbles assigned as an LCS, OPR, MS or MSD.

11.3.2. Reassemble the pre-extracted SDS apparatus, and add a fresh charge of toluene to the receiver and reflux flask. Apply power to the heating mantle to begin refluxing. Adjust the reflux rate to match the rate of percolation until water removal lessens the restriction to toluene flow. Wrap soxhlet with bubble foil to increase temperature.

11.3.3. Reflux the sample for a total of 16-24 hours (18-24 for tissues).

Cool and disassemble the apparatus.

11.3.4. Remove the distilling flask. Drain the water from the Dean-Stark receiver.

11.3.5. Concentrate the extracts from the particles to approximately 5 mL using the rotary evaporator.

11.4. Sludge Samples

11.4.1. Weigh the sample (nominal 10 g dry weight equivalent) directly into a kilned thimble.

- Place a ball of DCM-rinsed glass wool at the bottom of the thimble and the soxhlet.
- Add the appropriate volume of Internal Standard (IS) solution directly to the thimble. Add the appropriate volume of Native Standard (NS) to the thimbles assigned as an LCS, OPR, MS or MSD.

11.4.2. SDS extract for 16 – 24 hours with toluene.

11.4.3. If the % solids is too low to accommodate a 10 g sample size into thimble, fill thimble half full of Hydromatrix.

11.4.4. Tare weight of thimble then fill thimble full of sludge.

11.4.5. Record the weight of sludge, spike and then transfer immediately to soxhlet.

11.5. Tissue

11.5.1. Mix 25 g of well ground fish/tissue with at least 60-70 g of pre-cleaned Na_2SO_4 in a beaker. (If 10 g of tissue are used, then 30-40 g of Na_2SO_4 is used.)

11.5.2. Stir frequently to remove any lumps.

11.5.3. Re-stir after about ½ hour and transfer the mixture to a kilned thimble.

- Add the appropriate volume of Internal Standard (IS) solution directly to the thimble. Add the appropriate volume of Native Standard (NS) to the thimbles assigned as an LCS, OPR, MS or MSD.

11.5.4. Soxhlet extract for 18-24 hours with 1:1 DCM:Hexane.

11.5.5. Concentrate the extract to < 100 mL.

11.5.6. If % Lipids are to be determined proceed to the next step. Otherwise proceed with the appropriate cleanup procedures.

11.6. % Lipids

11.6.1. Transfer to a 250 mL mixing cylinder, adjust the extract to a convenient volume (typically 100 mL) using 1:1 DCM:Hexane and mix well.

11.6.2. Transfer 10% of the solution to an aluminum dish that has been pre-weighed on an analytical balance.

11.6.3. Allow the extract to air dry completely and then place in a 50±5 °C oven overnight.

11.6.4. When the aliquot is dry, allow to cool to room temperature, re-weigh the dish on an analytical balance and record the weight. Calculate the % lipids using the following equation:

$$\%\,\text{lipids} = \frac{\text{lipid residue weight}}{10\%\ \text{of sample weight}} \text{x}100$$

11.6.5. Using the remaining 90% of the extract, proceed with the appropriate cleanup procedures.

12. CLEANUP PROCEDURES

12.1. Coplanar PCBs analyses

12.1.1. Add ~100 μL of C_{14} and the appropriate Cleanup Recovery Standard(s) (CRS) to the extract.

12.1.2. Exchange the extract solvent to hexane and concentrate by one of the following techniques.

- Using a rotary evaporator; Rotovap to the $C_{14,}$ add 50 – 100 mL of hexane and Rotovap to the C_{14}.
- Using a turbovap evaporator; evaporate to < 2 mL, add 50 – 100 mL of hexane, be sure to mix the solvent in the stem with the added hexane and evaporate to < 2 mL.

12.1.3. Proceed to the appropriate cleanup.

12.2. Total PCBs analyses

12.2.1. Do not add any C_{14}.

12.2.2. Add the appropriate cleanup recovery standard.

12.2.3. Rotovap to < 20 mL, add 50 – 100 mL hexane and Rotovap to < 20 mL, repeat once more. Do not let go to dryness.

12.2.4. Proceed to the appropriate cleanup.

12.3. Acid Partitioning (AP)

12.3.1. Using hexane, adjust the extract volume to ~100 mL and transfer to the separatory funnel.

12.3.2. Carefully add ~50 mL of concentrated H_2SO_4 to a separatory funnel.

12.3.3. Shake for approximately 30 seconds with periodic venting, allow the layers to separate (centrifugation may be necessary) and discard the acid layer. Add ~50 mL of HPLC water to the separatory funnel. Shake for approximately 30 seconds with venting and discard the aqueous layer. Repeat if sample still contains color.

12.3.4. Pass the organic layer through Na_2SO_4 (pre-rinsed with 2 ~15 mL aliquots of hexane) then concentrate to <10 mL.

12.4. Acid Base Silica Gel (ABSG)

12.4.1. Prepare the column as depicted in Figure 1.

12.4.2. All traces of solvents other than hexane must be removed from the extract.

12.4.3. Adjust extract volume to <10 mL.

12.4.4. Rinse the ABSG column with ~60 mL hexane, discard the eluate.

12.4.5. Transfer the extract to the column with 2-4 small portions of hexane, begin collecting the eluate.

12.4.6. When the extract reaches the sodium sulfate, add 100 mL of hexane.

12.4.7. Concentrate the eluate appropriately.

12.5. Acid Alumina (AA) - Coplanar PCBs only.

12.5.1. Prepare column as depicted in Figure 2

12.5.2. Rinse column with ~30 mL DCM and then ~30 mL of hexane, discard the eluate.

12.5.3. Adjust extract volume to ~5mL.

12.5.4. Transfer the extract to the column with 2-4 small portions of hexane. Discard the eluate.

12.5.5. Elute the column with ~10 mL hexane. Discard the eluate.

12.5.6. Elute column with ~50 mL 20% DCM:Hexane, collect the eluate.

12.5.7. Concentrate the eluate to 2-3 mL.

13. ADJUST TO FINAL VOLUME

13.1. Coplanar PCBs (Solid)

13.1.1. Using hexane, quantitatively transfer the concentrated eluate to a conical vial that contains the Recovery Standard (RS) and 40 μL of tetradecane.

13.1.2. Using nitrogen blow down, concentrate to the tetradecane.

13.1.3. Rinse the walls of the conical vial with hexane, re-blow down to the tetradecane.

13.1.4. Using a 50-100 μL Wiretrol, transfer the tetradecane to an insert in a crimp top autoinjector vial and then cap.

13.2. Coplanar PCBs (Aqueous and Tissue)

13.2.1. Using hexane, quantitatively transfer the concentrated eluate to a conical vial that contains the Recovery Standard (RS) and 10 μL of tetradecane.

13.2.2. Using nitrogen blow down, concentrate to the tetradecane.

13.2.3. Rinse the walls of the conical vial with hexane, re-blow down to the tetradecane.

13.2.4. Using a 50-100 μL Wiretrol, transfer the tetradecane to an insert in a crimp top autoinjector vial and then cap.

13.3. Total PCBs

13.3.1. Use hexane to quantitatively transfer concentrated eluate to conical test tube or conical vial.

13.3.2. Using nitrogen blow down, concentrate to 250 μL.

13.3.3. Aqueous

- Add 20 μL C_9 to a conical vial and transfer sample to conical with hexane, blow down to ~50 – 100 μL. Add ~40 μL C_9 and

RS. Blow down to 20 μL.

13.3.4. All other matrices

- Add 100 μL of C_9 and appropriate RS. Using nitrogen blow down, slowly concentrate to C_9. Using 50-100 μL wiretrol, transfer the concentrate to an amber crimp top autoinjector vial, with an insert, and then cap.

14. QUALITY CONTROL

14.1. Method Blank (MB): Method Blank is a matrix preparation that is free from native analyte that has been prepared and analyzed using the same procedures followed for the rest of the sample batch. HPLC water is used for aqueous samples, sand is used for solid and fish samples.

14.1.1. A MB is run with every analytical batch or 20 samples, whichever is less, per matrix type.

14.1.2. Analyze the method or solvent blank immediately after analysis of the OPR to demonstrate freedom from contamination.

14.1.3. If the amount found is greater than the minimum level or one-third the regulatory compliance limit, whichever is greater; or if any potentially interfering compound is found in the blank at or above the minimum level for each congener, the data must be evaluated to determine whether the batch shall be re-extracted or the data is qualified appropriately.

14.2. Ongoing Precision and Recovery (OPR): An ongoing precision and recovery sample is prepared by adding a known quantity of native standard to an interferent free matrix and used to assess method performance (precision and recovery).

14.2.1. Spike 10 μL of compound spiking solution into the sample.

14.2.2. An OPR is analyzed with every analytical batch or 20 samples (whichever is less) per matrix type.

14.2.3. For each native and labeled compound, compare the concentration with the limits for ongoing accuracy in Table 7. If all compounds do not meet acceptance criteria, then re-extract and analyze the batch.

14.3. Matrix Spike (MS/MSD): A matrix spike sample is prepared by adding a known quantity of native standard to a sample matrix prior to extraction.

14.3.1. Matrix Spikes are performed by client request or to fulfill state agency requirements.

Note: Projects performed pursuant to the guidelines established by the DOD QSM shall contain an associated Matrix Spike per preparatory batch. A Matrix Spike Duplicate or Laboratory Duplicate shall also be analyzed per preparatory batch for these projects.

14.3.2. Spike 10 μL of compound spiking solution into the sample.

14.3.3. The relative percent difference between MS/MSD samples should be ≤20%.

14.4. Duplicate Samples: Duplicate samples are two separate aliquots taken from the same source. Duplicate samples are analyzed independently to assess laboratory precision. Duplicate samples are performed by client request.

14.4.1. If the relative percent difference from duplicate sample analyses is greater than 25%, then both duplicate samples will be reanalyzed.

14.4.2. Criteria for qualitative and quantitative determinations of chromatographic peaks must be met.

15. HRMS ANALYSIS

15.1. Establish the necessary operating conditions. The GC conditions may be optimized for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, blanks, OPR aliquots and samples. The following GC operating conditions are for guidance and adjustments may be required.

Injector temperature:	270°C
Interface temperature:	290°C
Initial temperature:	130°C
Initial time:	1.0 minutes
Temperature program:	130 - 200°C at 5°C/min
	200°C for 16 minutes
	200 - 280°C at 8°C/min
	280°C for 11 minutes
	280 - 300°C at 13°C/min
	300°C for 4.5 minutes

15.2. GC Column

15.2.1. The retention time for decachlorobiphenyl must be greater than 55 minutes, for a ZB-1 column and SPB-Octyl column or equivalent.

15.2.2. The ZB -1 column must resolve the following congeners:

- 28, 31 with a valley height less than 50%
- 66, 70, 74, 80 with a valley height less than 40%

- 123, 118 with a valley height less than 40%
- 156, 157 with a valley height less than 40%

15.2.3. The SPB-Octyl column must resolve the following congeners:

- 34, 23 with a valley height less than 40%
- 187, 182 with a valley height less than 40%

15.2.4. If a project/client requires separation of any co-eluting PCB congeners, then the extract may be analyzed on an alternate GC column.

15.3. Instrument Tuning

15.3.1. Inject the reference compound perfluorokerosene (PFK) performed at the beginning and at the end of each shift. PFK provides the required lock masses and is used for tuning the mass spectrometer.

15.3.2. Using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 at or close to m/z 304.9824. For each descriptor, monitor and record the resolution and exact m/z of three to five reference peaks covering the range of the descriptor.

15.3.3. The deviation between exact m/z and the theoretical m/z for each exact m/z monitored must be less than 5 ppm.

15.3.4. An appropriate lock mass will be monitored for each descriptor and shall not vary by more than $\pm$ 20% throughout the respective retention time window.

15.3.5. For the measurement of PCBs, the exact m/z's to be monitored in each descriptor are listed in Table 6.

15.3.6. Because of the extensive mass range covered in each function, it may not be possible to maintain 10,000 resolution throughout the mass range. Therefore, resolution must be 8,000 throughout the mass range and must be 10,000 in the center of the mass range for each function.

15.4. Initial Calibration

15.4.1. Under the same conditions, inject 1-2 μL of each of the six calibration solutions containing all PCB isomers. Calibration standard solutions are presented in Table 2. For consistency, all samples and standards will have the same injection volume.

15.4.2. Forty-four internal standards and six recovery standards are used to improve quantitation.

15.4.3. The signal to noise ratio (s/n) must exceed 10:1 for all ions monitored (except Di-CBs are at or above 2.5:1).

15.4.4. The ion abundance ratio measurements must be within the theoretical ratio limits (See Table 3).

15.4.5. Calibration by Isotope Dilution: Isotope dilution calibration is used for the native PCBs for which labeled compounds are available.

15.4.6. If the relative response for any compound is less than 20% RSD over the 6-point calibration range, an averaged relative response is used for that compound.

15.4.7. All initial instrument calibrations are verified with a standard solution from a second manufacturer or lot.

15.4.8. The in-house limits of 60-140% have been developed as the acceptance criteria based upon second source standard history.

- Natives for which a labeled compound is not available will use internal standard calibration. The average Relative Response for that congener will be used and %RSD must be < 30%.

15.5. Set up the analytical run following this sequential injection pattern: Window Defining Mix (CS3), OPR, Solvent Blank, Method Blank, Samples, Solvent Blank, and CS3.

15.6. Continuing Calibration

15.6.1. Inject a mid-range standard from the initial calibration curve (CS3). The following criteria must be met:

1.) Calculate the concentration of each native compound either by isotope dilution or internal standard technique. Each compound must be within the verification limits established in Table 7.

2.) The ion ratios must be within the theoretical ratio limits (Table 3).

3.) The signal to noise ratio (s/n) must exceed 10:1 for all ions monitored.

4.) The absolute retention times of the internal standards shall be within ±15 seconds of the retention times obtained during calibration.

5.) The relative retention times of the peak for a native and labeled PCB should be within 0.5% of the retention time windows established from the initial calibration curve.

15.6.2. If the above criteria are not met for any compound, correct the problem and repeat the calibration verification, or recalibrate and document return to control.

15.7. Qualitative Determination

15.7.1. A chromatographic peak is identified as a PCB or labeled compound when all of the following criteria are met:

1.) The signals for the two exact m/z's being monitored (Table 8 of Method 1668) must be present and must maximize within ±2 seconds of one another.

2.) The signal-to-noise ratio (S/N) of each of the two exact m/z's must be ≥2.5:1 for a sample extract, and ≥10:1 for a calibration standard.

3.) The ratio of the integrated areas of the two exact m/z's must be within the limits established in Table 3.

- Optional reporting: If the ion abundance ratio is outside the limits established in Table 3, the chromatographic peak will be quantified as an estimated maximum possible concentration or EMPC.

4.) The relative retention times of the peak for a native and labeled PCB should be within 0.5% of the retention time windows established from the initial calibration curve.

- If the above mentioned criteria are not met, the peak is not identified as a positive.

15.8. Quantitative Determination

15.8.1. Any numerical values that are calculated below the quantitation limit are reported as non-detects unless requested otherwise by client. Coplanar PCBs are reported as non-detects below the detection limit and may be qualified with a "J" flag if detected below the quantitation limit.

15.8.2. For peaks which meet the criteria listed above, quantitate the PCB peaks from the response relative to the appropriate internal standard.

15.8.3. Any peaks representing the other congeners are quantitated using an average of the response factors from all of the labeled PCBs

isomers at the same level of chlorination.

15.8.4. Recovery of each internal standard must be within limits in Table 7 for samples.

15.8.5. It the above-mentioned criteria are not met but the qualitative criteria is met, then the data are qualified appropriately.

- For DRBC samples, analyze on SPB-Octyl column.

16. CALCULATIONS

16.1. The concentrations for PCB compounds are calculated by using the formula:

$$C_X = \frac{(A_X)(Q_{IS})}{(A_{IS})(W)(RRF)}$$

Where:

C_X = *Concentration of unlabeled PCB congeners (or group of coeluting isomers within an homologous series),*
A_X = *Sum of the integrated ion abundances of the quantitation ions for unlabeled PCBs*
A_{IS} = *Sum of the integrated ion abundances of the quantitation ion for the labeled internal standards,*
Q_{IS} = *Quantity, in pg, of the internal standard added to the sample before extraction,*
W = *Weight of the sample (solid, dry weight or liquid)*
DW = *Sample wt.$\times$ %solids/100*
RRF = *Calculated relative response factor for the analyte.*

16.2. The detection limits can be calculated using the following formula:

$$DL = \frac{(2.5)(H_N)(Q_{IS})}{(H_{IS})(W)(RRF)}$$

Where:

DL = *Sample specific estimated detection limit,*
H_N = *Noise height (peak to peak),*
H_{IS} = *Peak height of the internal standard,*
Q_{is} = *Quantity, in pg, of the internal standard added to the sample before extraction,*
W = *Weight of the sample (solid or liquid), and*
RRF = *Calculated relative response factor for the analyte.*

16.3. The reporting limits can be calculated using the following formula:

$$RL = \frac{(\text{Extract Conc. of Low point of curve})(\text{Final volume})}{(\text{Weight of sample})}(\text{Split})$$

16.4. The Relative Response factor can be calculated using the following formula:

$$RRF = \frac{\left(A^1_N + A^2_N\right)(C_{IS})}{\left(A^1_{IS} + A^2_{IS}\right)(C_N)}$$

Where:

$A1_N$, $A2_N$	=	*Areas of the primary and secondary m/z's for the native compound*
$A1_{IS}$, $A2_{IS}$	=	*Areas of the primary and secondary m/z's for the labeled compound.*
C_{IS}	=	*Concentration of the labeled compound in the calibration standard.*
C_N	=	*Concentration of the native compound in the calibration standard*

16.5. Estimated Maximum Possible Concentration (EMPC):

$$EMPC = \frac{(A_X)\ (Q_{IS})}{(A_{IS})\ (W)\ (RF_N)}$$

Where:

A_X	=	*Sum of the area of the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio*
A_{IS}	=	*Sum of the integrated ion abundances of the quantitation ions for the labeled internal standards*
Q_{IS}	=	*Quantity, in pg, of the internal standard added to the sample before extraction*
W	=	*Weight of the sample or volume of aqueous sample*
RF_N	=	*Calculated mean relative response factor for the analyte*

17. POLLUTION PREVENTION

17.1. The solvent evaporation techniques used in this method are amenable to solvent recovery, and it is recommended that the laboratory recover solvents wherever feasible.

17.2. Standards should be prepared in volumes consistent with laboratory use to minimize disposal of standards.

18. WASTE MANAGEMENT

18.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. Safety officer should be contacted if additional information is required.

18.2. The laboratory waste management is in compliance with all federal, state, and local regulations to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations.

19. METHOD PERFORMANCE

19.1. This SOP is based on the following method: EPA Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS, United States Office of Water, EPA No. EPA 821-R-00-002, Environmental Protection Agency (4303), December 1999

20. REFERENCES

20.1. Alford-Stevens, A., Bellar, T. A., Eichelberger, J. W., and W. L. Budde, 1984. Method 680 -- Determination of Pesticides and PCBs in Water and Soil/Sediment by HRGC/LRMS, U. S. EPA. Cincinnati, OH.

20.2. Draft Method 1668. Measurement of Toxic PCB Congeners By Isotope Dilution HRGC/HRMS, Prepared by Analytical Methods Staff, Engineering and Analysis Division (4303), Office of Science and Technology, Office of Water, U. S. Environmental Protection Agency, Washington, DC, March, 1997.

20.3. EPA Region 10 SOP For the Validation of Method 1668 Toxic, Dioxin-Like, PCB Data. Revision 1.0, December 8, 1995.

20.4. Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS. Prepared under the direction of William A. Telliard, Engineering and Analysis Division, Office of Water, U.S. Environmental Protection Agency, Washington D.C., December 1999

20.5. Vista Analytical Laboratory SOP 9, Manual Integrations

20.6. SOP 10 - Instrument Maintenance Logbooks and Schedule

20.7. Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Engineering and Analysis Division (4303T), Washington D.C., April 2010.

Figure 1

Acid Base Silica Gel (ABSG)

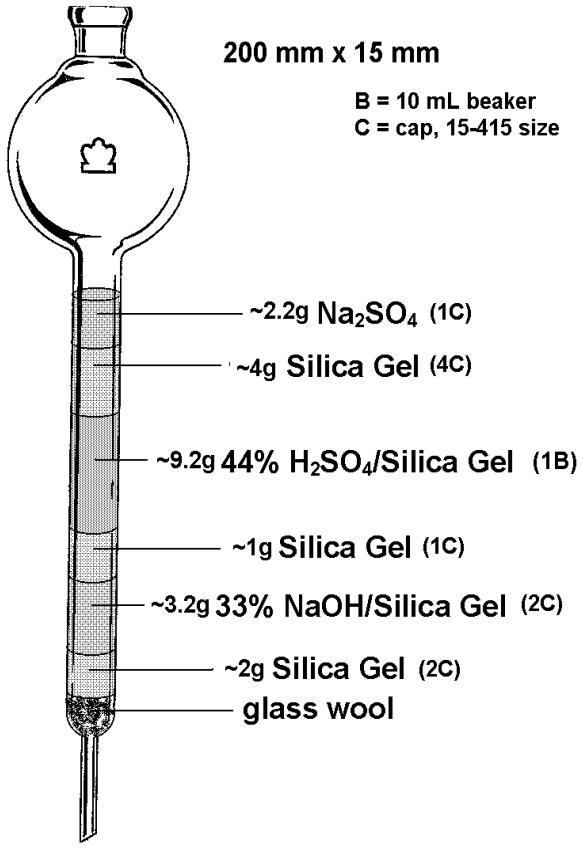

Figure 2

Acid Alumina (AA) Coplanar PCBs only

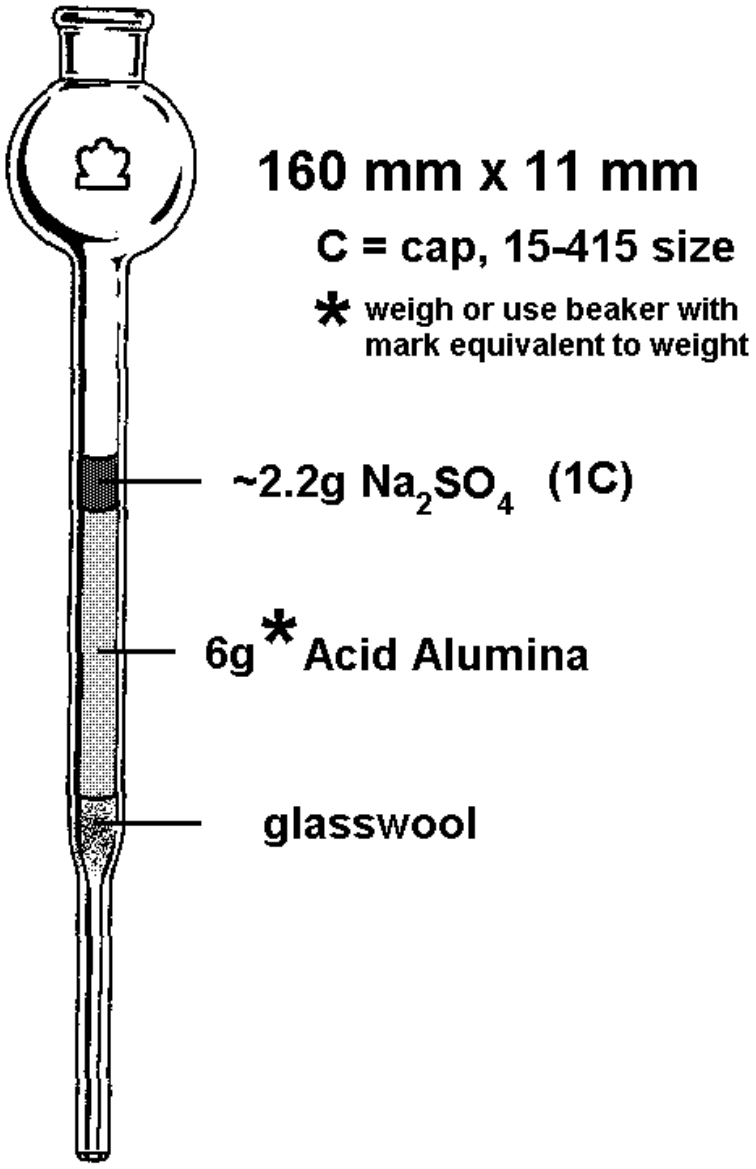

Table 1

PCB Target Compounds and Quantitation Limits

Coplanar PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
3,3',4,4'-Tetra-CB	77	5.0	2.5	0.5	0.25	50.0
3,4,4',5-Tetra-CB	81	5.0	2.5	0.5	0.25	50.0
3,3',4,4',5-Penta-CB	126	5.0	2.5	0.5	0.25	50.0
3,3',4,4',5,5'-Hexa-CB	169	5.0	2.5	0.5	0.25	50.0
Toxically Significant Mono-Ortho Substituted PCBs						
2,3,3',4,4'-Penta-CB	105	5.0	2.5	0.5	0.25	50.0
2,3,4,4',5-Penta-CB	114	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5-Penta-CB	118	5.0	2.5	0.5	0.25	50.0
2',3,4,4',5-Penta-CB	123	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5-Hexa-CB	156	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5'-Hexa-CB	157	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5,5'-Hexa-CB	167	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5,5'-Hepta-CB	189	5.0	2.5	0.5	0.25	50.0
Other Environmentally Significant PCBs (209)						
2-Mono-CB	1	5.0	2.5	0.5	0.25	50.0
3-Mono-CB	2	5.0	2.5	0.5	0.25	50.0
4-Mono-CB	3	5.0	2.5	0.5	0.25	50.0
2,2'-Di-CB 2,6-Di-CB	4/10	20.0	10.0	2.0	1.00	200.0
2,3'-Di-CB	6	10.0	5.0	1.0	0.50	100.0
2,3-Di-CB 2,4'-Di-CB	5/8	20.0	10.0	2.0	1.00	200.0
2,4-Di-CB 2,5-Di-CB	7/9	20.0	10.0	2.0	1.00	200.0
3,3'-Di-CB	11	10.0	5.0	1.0	0.50	100.0
3,4-Di-CB 3,4'-Di-CB	12/13	20.0	10.0	2.0	1.00	200.0
3,5-Di-CB	14	10.0	5.0	1.0	0.50	100.0
4,4'-Di-CB	15	10.0	5.0	1.0	0.50	100.0
2,2',3-Tri-CB 2,2',3-Tri-CB	16/32	10.0	5.0	1.0	0.50	100.0
2,2',4-Tri-CB	17	5.0	2.5	0.5	0.25	50.0
2,2',5-Tri-CB	18	5.0	2.5	0.5	0.25	50.0
2,2',6-Tri-CB	19	5.0	2.5	0.5	0.25	50.0
2,3,3'-Tri-CB 2,3,4-Tri-CB 2',3,4-Tri-CB	20/21/33	15.0	7.5	1.5	0.75	150.0
2,3,4'-Tri-CB	22	5.0	2.5	0.5	0.25	50.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,3,5-Tri-CB	23	5.0	2.5	0.5	0.25	50.0
2,3,6-Tri-CB 2,3',6-Tri-CB	24/27	10.0	5.0	1.0	0.50	100.0
2,3',4-Tri-CB	25	5.0	2.5	0.5	0.25	50.0
2,3',5-Tri-CB	26	5.0	2.5	0.5	0.25	50.0
2,4,4'-Tri-CB	28	5.0	2.5	0.5	0.25	50.0
2,4,5-Tri-CB	29	5.0	2.5	0.5	0.25	50.0
2,4,6-Tri-CB	30	5.0	2.5	0.5	0.25	50.0
2,4',5-Tri-CB	31	5.0	2.5	0.5	0.25	50.0
2,3',5'-Tri-CB	34	5.0	2.5	0.5	0.25	50.0
3,3',4-Tri-CB	35	5.0	2.5	0.5	0.25	50.0
3,3',5-Tri-CB	36	5.0	2.5	0.5	0.25.	50.0
3,4,4'-Tri-CB	37	5.0	2.5	0.5	0.25	50.0
3,4,5-Tri-CB	38	5.0	2.5	0.5	0.25	50.0
3,4',5-Tri-CB	39	5.0	2.5	0.5	0.25	50.0
2,2',3,3'-Tetra-CB	40	5.0	2.5	0.5	0.25	50.0
2,2',3,4-Tetra-CB 2,3,4',6-Tetra-CB 2,3',4',6-Tetra-CB 2,3',5,5'-Tetra-CB	41/64/71/72	20.0	10.0	2.0	1.00	200.0
2,2',3,4'-Tetra-CB 2,3,3',6-Tetra-CB	42/59	10.0	5.0	1.0	0.50	100.0
2,2',3,5-Tetra-CB 2,2',4,5'-Tetra-CB	43/49	10.0	5.0	1.0	0.50	100.0
2,2',3,5'-Tetra-CB	44	5.0	2.5	0.5	0.25	50.0
2,2',3,6-Tetra-CB	45	5.0	2.5	0.5	0.25	50.0
2,2',3,6'-Tetra-CB	46	5.0	2.5	0.5	0.25	50.0
2,2',4,4'-Tetra-CB	47	5.0	2.5	0.5	0.25	50.0
2,2',4,5-Tetra-CB 2,4,4',6-Tetra-CB	48/75	10.0	5.0	1.0	0.50	100.0
2,2',4,6-Tetra-CB	50	5.0	2.5	0.5	0.25	50.0
2,2',4,6'-Tetra-CB	51	5.0	2.5	0.5	0.25	50.0
2,2',5,5'-Tetra-CB 2,3',4,6-Tetra-CB	52/69	10.0	5.0	1.0	0.50	100.0
2,2',5,6'-Tetra-CB	53	5.0	2.5	0.5	0.25	50.0
2,2',6,6'-Tetra-CB	54	5.0	2.5	0.5	0.25	50.0
2,3,3',4-Tetra-CB	55	5.0	2.5	0.5	0.25	50.0
2,3,3',4'-Tetra-CB 2,3,4,4'-Tetra-CB	56/60	10.0	5.0	1.0	0.50	100.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,3,3',5-Tetra-CB	57	5.0	2.5	0.5	0.25	50.0
2,3,3',5'-Tetra-CB	58	5.0	2.5	0.5	0.25	50.0
2,3,4,5-Tetra-CB 2,3',4',5-Tetra-CB	61/70	10.0	5.0	1.0	0.50	100.0
2,3,4,6-Tetra-CB	62	5.0	2.5	0.5	0.25	50.0
2,3,4',5-Tetra-CB	63	5.0	2.5	0.5	0.25	50.0
2,3,5,6-Tetra-CB	65	5.0	2.5	0.5	0.25	50.0
2,3',4,5-Tetra-CB	67	5.0	2.5	0.5	0.25	50.0
2,3',4,5'-Tetra-CB	68	5.0	2.5	0.5	0.25	50.0
2,3',4',5-Tetra-CB	70	5.0	2.5	0.5	0.25	50.0
2,3',5',6-Tetra-CB	73	5.0	2.5	0.5	0.25	50.0
2,4,4',5-Tetra-CB	74	5.0	2.5	0.5	0.25	50.0
2',3,4,5-Tetra-CB 2,3',4,4'-Tetra-CB	76/66	10.0	5.0	1.0	0.50	100.0
3,3',4,4'-Tetra-CB	77	5.0	2.5	0.5	0.25	50.0
3,3',4,5-Tetra-CB	78	5.0	2.5	0.5	0.25	50.0
3,3',4,5'-Tetra-CB	79	5.0	2.5	0.5	0.25	50.0
3,3',5,5'-Tetra-CB	80	5.0	2.5	0.5	0.25	50.0
3,4,4',5-Tetra-CB	81	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4-Penta-CB	82	5.0	2.5	0.5	0.25	50.0
2,2',3,3',5-Penta-CB	83	5.0	2.5	0.5	0.25	50.0
2,2',3,3',6-Penta-CB 2,2',3,5,5'-Penta-CB	84/95	10.0	5.0	1.0	0.50	100.0
2,2',3,4,4'-Penta-CB 2,3,4,5,6-Penta-CB	85/116	10.0	5.0	1.0	0.50	100.0
2,2',3,4,5-Penta-CB	86	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5'-Penta-CB 2,3,4',5,6-Penta-CB 2',3,4,5,6'-Penta-CB	87/117/125	15.0	7.5	1.5	0.75	150.0
2,2',3,4,6-Penta-CB 2,2',3,4',6-Penta-CB	88/91	10.0	5.0	1.0	0.50	100.0
2,2',3,4,6'-Penta-CB	89	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5-Penta-CB 2,2',4,5,5'-Penta-CB	90/101	10.0	5.0	1.0	0.50	100.0
2,2',3,5,6-Penta-CB	93	5.0	2.5	0.5	0.25	50.0
2,2',3,5,6'-Penta-CB	94	5.0	2.5	0.5	0.25	50.0
2,2',3,5',6-Penta-CB 2,2',3',4,6-Penta-CB 2,2',4,5,6'-Penta-CB	95/98/102	15.0	7.5	1.5	0.75	150.0
2,2',3,6,6'-Penta-CB	96	5.0	2.5	0.5	0.25	50.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,2',3,4',5-Penta-CB	97	5.0	2.5	0.5	0.25	50.0
2,2',4,4',5-Penta-CB	99	5.0	2.5	0.5	0.25	50.0
2,2',4,4',6-Penta-CB	100	5.0	2.5	0.5	0.25	50.0
2,2',4,5',6-Penta-CB	103	5.0	2.5	0.5	0.25	50.0
2,2',4,4,6'-Penta-CB	104	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4'-Penta-CB	105	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	10.0	5.0	1.0	0.50	100.0
2,3,3',4',5-Penta-CB 2,3,3',4,6-Penta-CB	107/109	10.0	5.0	1.0	0.50	100.0
2,3,3',4,5'-Penta-CB 2,3,3',5,6-Penta-CB	108/112	10.0	5.0	1.0	0.50	100.0
2,3,3',4',6-Penta-CB	110	5.0	2.5	0.5	0.25	50.0
2,3,3',5,5'-Penta-CB 2,3,4,4',6-Penta-CB	111/115	10.0	5.0	1.0	0.50	100.0
2,3,3',5',6-Penta-CB	113	5.0	2.5	0.5	0.25	50.0
2,3,4,4',5-Penta-CB	114	5.0	2.5	0.5	0.25	50.0
2,3',4,4',6-Penta-CB	119	5.0	2.5	0.5	0.25	50.0
2,3',4,5,5'-Penta-CB	120	5.0	2.5	0.5	0.25	50.0
2,3',4,5',6-Penta-CB	121	5.0	2.5	0.5	0.25	50.0
2,3,3',4',5'-Penta-CB	122	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5'-Penta-CB	123	5.0	2.5	0.5	0.25	50.0
2,3',4',5,5'-Penta-CB	124	5.0	2.5	0.5	0.25	50.0
3,3'4,4',5-Penta-CB	126	5.0	2.5	0.5	0.25	50.0
3,3',4,5,5'-Penta-CB	127	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4'-Hexa-CB 2,3,3',4',5,5'-Hexa-CB	128/162	10.0	5.0	1.0	0.50	100.0
2,2',3,3',4,5-Hexa-CB	129	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5'-Hexa-CB	130	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,6-Hexa-CB	131	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,6'-Hexa-CB 2,3,3',4,5',6-Hexa-CB	132/161	10.0	5.0	1.0	0.50	100.0
2,2',3,3',5,5'-Hexa-CB 2,2',3,4,5,6-Hexa-CB	133/142	10.0	5.0	1.0	0.50	100.0
2,2',3,3',5,6-Hexa-CB 2,2',3,4,5,6'-Hexa-CB	134/143	10.0	5.0	1.0	0.50	100.0
2,2',3,3',5,6'-Hexa-CB	135	5.0	2.5	0.5	0.25	50.0
2,2',3,3',6,6'-Hexa-CB	136	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',5-Hexa-CB	137	5.0	2.5	0.5	0.25	50.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,2',3,4,4',5'-Hexa-CB 2,3,3',4',5,6-Hexa-CB 2,3,3',4',5',6-Hexa-CB	138/163/164	15.0	7.5	1.5	0.75	150.0
2,2',3,4,4',6-Hexa-CB 2,2',3,4',5',6-Hexa-CB	139/149	10.0	5.0	1.0	0.50	100.0
2,2',3,4,4',6'-Hexa-CB	140	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5,5'-Hexa-CB	141	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5',6-Hexa-CB	144	5.0	2.5	0.5	0.25	50.0
2,2',3,4,6,6'-Hexa-CB	145	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5,5'-Hexa-CB 2,3,3',5,5',6-Hexa-CB	146/165	10.0	5.0	1.0	0.50	100.0
2,2',3,4',5,6-Hexa-CB	147	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5,6'-Hexa-CB	148	5.0	2.5	0.5	0.25	50.0
2,2',3,4',6,6'-Hexa-CB	150	5.0	2.5	0.5	0.25	50.0
2,2',3,5,5',6-Hexa-CB	151	5.0	2.5	0.5	0.25	50.0
2,2',3,5,6,6'-Hexa-CB	152	5.0	2.5	0.5	.0.25	50.0
2,2',4,4',5,5'-Hexa-CB	153	5.0	2.5	0.5	0.25	50.0
2,2',4,4',5,6'-Hexa-CB	154	5.0	2.5	0.5	0.25	50.0
2,2',4,4',6,6'-Hexa-CB	155	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5-Hexa-CB	156	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5'-Hexa-CB	157	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',6-Hexa-CB 2,3,3',4,5,6-Hexa-CB	158/160	10.0	5.0	1.0	0.50	100.0
2,3,3',4,5,5'-Hexa-CB	159	5.0	2.5	0.5	0.25	50.0
2,3,4,4',5,6-Hexa-CB	166	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5,5'-Hexa-CB	167	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5',6-Hexa-CB	168	5.0	2.5	0.5	0.25	50.0
3,3',4,4',5,5'-Hexa-CB	169	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5-Hepta-CB	170	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',6-Hepta-CB	171	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,5'-Hepta-CB	172	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,6-Hepta-CB	173	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,6'-Hepta-CB	174	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5',6-Hepta-CB	175	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,6,6'-Hepta-CB	176	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4',5,6-Hepta-CB	177	5.0	2.5	0.5	0.25	50.0
2,2',3,3',5,5',6-Hepta-CB	178	5.0	2.5	0.5	0.25	50.0
2,2',3,3',5,6,6'-Hepta-CB	179	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',5,5'-Hepta-CB	180	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',5,6-Hepta-CB	181	5.0	2.5	0.5	0.25	50.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,2',3,4,4',5,6'-Hepta-CB 2,2',3,4',5,5',6-Hepta-CB	182/187	10.0	5.0	1.0	0.50	100.0
2,2',3,4,4',5',6-Hepta-CB	183	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',6,6'-Hepta-CB	184	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5,5',6-Hepta-CB	185	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5,6,6-'Hepta-CB	186	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5,6,6'-Hepta-CB	188	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5,5'-Hepta-CB	189	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5,6-Hepta-CB	190	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5',6-Hepta-CB	191	5.0	2.5	0.5	0.25	50.0
2,3,3',4,5,5',6-Hepta-CB	192	5.0	2.5	0.5	0.25	50.0
2,3,3',4',5,5',6-Hepta-CB	193	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,5'-Octa-CB	194	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,6-Octa-CB	195	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,6'-OctaCB 2,2',3,4,4',5,5',6-OctaCB	196/203	10.0	5.0	1.0	0.50	100.0
2,2',3,3',4,4',6,6'-Octa-CB	197	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,5',6-Octa-CB	198	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,5',6'-Octa-CB	199	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,6,6'-Octa-CB	200	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5',6,6'-Octa-CB	201	5.0	2.5	0.5	0.25	50.0
2,2',3,3'5,5',6,6'-Octa-CB	202	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',5,6,6'-Octa-CB	204	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5,5',6-Octa-CB	205	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,5',6-Nona-CB	206	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,6,6'-Nona-CB	207	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,5',6,6'-Nona-CB	208	5.0	2.5	0.5	0.25	50.0

Table 1

PCB Target Compounds and Quantitation Limits

Deca-CB	209	5.0	2.5	0.5	0.25	50.0
Total PCB Homologues						
Total PCB Homologues		**Water (pg/L)**	**Solid (pg/g)**	**Fish/Tissue (pg/g)**		
Monochlorobiphenyl		5.0	2.5	0.5		
Dichlorobiphenyl		10.0	5.0	1.0		
Trichlorobiphenyl		5.0	2.5	0.5		
Tetrachlorobiphenyl		5.0	2.5	0.5		
Pentachlorobiphenyl		5.0	2.5	0.5		
Hexachlorobiphenyl		5.0	2.5	0.5		
Heptachlorobiphenyl		5.0	2.5	0.5		
Octachlorobiphenyl		5.0	2.5	0.5		
Nonachlorobiphenyl		5.0	2.5	0.5		
Decachlorobiphenyl		5.0	2.5	0.5		

- Quantitation limits listed are based upon 1 liter of aqueous sample, 10 grams dry weight solid, 10 grams fish/tissue sample and 20 grams Sample size for Serum [Serum (pg/g-Lipids) assumes 0.5% Lipid content]. Quantitation limits for co-eluting congeners are based on their sum.
- Final Volumes for all matrices are 20 μL except Solid is 100 μL.
- *California SIP samples have a reporting limit of 20 pg/L for all congeners.*
- For DOD clients the lowest standard of the calibration establishes the QL.

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. Environmentally Significant PCBs (209)	**Congener No.**	**CS0**	**CS1**	**CS2**	**CS3***	**CS4**	**CS5**
2-Mono-CB	1	0.25	1.0	2.5	50	400	750
3-Mono-CB	2	0.25	1.0	2.5	50	400	750
4-Mono-CB	3	0.25	1.0	2.5	50	400	750
2,2'-Di-CB 2,6-Di-CB	4/10	1.0	4.0	10	200	1600	3000
2,3'-Di-CB	6	0.5	2.0	5.0	100	800	1500
2,3-Di-CB 2,4'-Di-CB	5/8	1.0	4.0	10	200	1600	3000
2,4-Di-CB 2,5-Di-CB	7/9	1.0	4.0	10	200	1600	3000
3,3'-Di-CB	11	0.5	2.0	5.0	100	800	1500
3,4-Di-CB 3,4'-Di-CB	12/13	1.0	4.0	10	200	1600	3000
3,5-Di-CB	14	0.5	2.0	5.0	100	800	1500
4,4'-Di-CB	15	0.5	2.0	5.0	100	800	1500
2,2',3-Tri-CB 2,4',6-Tri-CB	16/32	0.5	2.0	5.0	100	800	1500
2,2',4-Tri-CB	17	0.25	1.0	2.5	50	400	750
2,2',5-Tri-CB	18	0.25	1.0	2.5	50	400	750
2,2',6-Tri-CB	19	0.25	1.0	2.5	50	400	750
2,3,3'-Tri-CB 2,3,4-Tri-CB 2',3,4-Tri-CB	20/21/33	0.75	3.0	7.5	150	1200	2250
2,3,4'-Tri-CB	22	0.25	1.0	2.5	50	400	750
2,3,5-Tri-CB	23	0.25	1.0	2.5	50	400	750
2,3,6-Tri-CB 2,3',6-Tri-CB	24/27	0.5	2.0	5.0	100	800	1500
2,3',4-Tri-CB	25	0.25	1.0	2.5	50	400	750
2,3',5-Tri-CB	26	0.25	1.0	2.5	50	400	750
2,4,4'-Tri-CB	28	0.25	1.0	2.5	50	400	750
2,4,5-Tri-CB	29	0.25	1.0	2.5	50	400	750
2,4,6-Tri-CB	30	0.25	1.0	2.5	50	400	750
2,4',5-Tri-CB	31	0.25	1.0	2.5	50	400	750
2,3',5'-Tri-CB	34	0.25	1.0	2.5	50	400	750
3,3',4-Tri-CB	35	0.25	1.0	2.5	50	400	750
3,3',5-Tri-CB	36	0.25	1.0	2.5	50	400	750
3,4,4'-Tri-CB	37	0.25	1.0	2.5	50	400	750
3,4,5-Tri-CB	38	0.25	1.0	2.5	50	400	750
3,4',5-Tri-CB	39	0.25	1.0	2.5	50	400	750
2,2',3,3'-Tetra-CB	40	0.25	1.0	2.5	50	400	750

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. Environmentally Significant PCBs (209)	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2,2',3,4-Tetra-CB 2,3,4',6-Tetra-CB 2,3',4',6-Tetra-CB 2,3',5,5'-Tetra-CB	41/64/71/72	1.0	4.0	10	200	1600	3000
2,3',5,5'-Tetra-CB 2,2',3,4'-Tetra-CB	42/59	0.50	2.0	5.0	100	800	1500
2,3,3',6-Tetra-CB 2,2',3,5-Tetra-CB	43/49	0.50	2.0	5.0	100	800	1500
2,2',4,5'-Tetra-CB	44	0.25	1.0	2.5	50	400	750
2,2',3,5'-Tetra-CB	45	0.25	1.0	2.5	50	400	750
2,2',3,6-Tetra-CB	46	0.25	1.0	2.5	50	400	750
2,2',3,6'-Tetra-CB	47	0.25	1.0	2.5	50	400	750
2,2',4,4'-Tetra-CB 2,2',4,5-Tetra-CB	48/75	0.50	2.0	5.0	100	800	1500
2,4,4',6-Tetra-CB	50	0.25	1.0	2.5	50	400	750
2,2',4,6-Tetra-CB	51	0.25	1.0	2.5	50	400	750
2,2',4,6'-Tetra-CB 2,2',5,5'-Tetra-CB	52/69	0.50	2.0	5.0	100	800	1500
2,3',4,6-Tetra-CB	53	0.25	1.0	2.5	50	400	750
2,2',5,6'-Tetra-CB	54	0.25	1.0	2.5	50	400	750
2,2',6,6'-Tetra-CB	55	0.25	1.0	2.5	50	400	750
2,3,3',4-Tetra-CB 2,3,3',4'-Tetra-CB	56/60	0.50	2.0	5.0	100	800	1500
2,3,4,4'-Tetra-CB	57	0.25	1.0	2.5	50	400	750
2,3,3',5-Tetra-CB	58	0.25	1.0	2.5	50	400	750
2,3,3',5'-Tetra-CB 2,3,4,5-Tetra-CB	61/70	0.50	2.0	5.0	100	800	1500
2,3',4',5-Tetra-CB	62	0.25	1.0	2.5	50	400	750
2,3,4,6-Tetra-CB	63	0.25	1.0	2.5	50	400	750
2,3,4',5-Tetra-CB	65	0.25	1.0	2.5	50	400	750
2,3,5,6-Tetra-CB	67	0.25	1.0	2.5	50	400	750
2,3',4,5-Tetra-CB	68	0.25	1.0	2.5	50	400	750
2,3',4,5'-Tetra-CB	70	0.25	1.0	2.5	50	400	750
2,3',4',5-Tetra-CB	73	0.25	1.0	2.5	50	400	750
2,3',5',6-Tetra-CB	74	0.25	1.0	2.5	50	400	750
2,4,4',5-Tetra-CB 2',3,4,5-Tetra-CB	76/66	0.50	2.0	5.0	100	800	1500
2,3',4,4'-Tetra-CB	77	0.25	1.0	2.5	50	400	750
3,3',4,4'-Tetra-CB	78	0.25	1.0	2.5	50	400	750
3,3',4,5-Tetra-CB	79	0.25	1.0	2.5	50	400	750
3,3',4,5'-Tetra-CB	80	0.25	1.0	2.5	50	400	750

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. Environmentally Significant PCBs (209)	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
3,3',5,5'-Tetra-CB	81	0.25	1.0	2.5	50	400	750
3,4,4',5-Tetra-CB	82	0.25	1.0	2.5	50	400	750
2,2',3,3',4-Penta-CB	83	0.25	1.0	2.5	50	400	750
2,2',3,3',5-Penta-CB 2,2',3,3',6-Penta-CB	84/92	0.50	2.0	5.0	100	800	1500
2,2',3,5,5'-Penta-CB 2,2',3,4,4'-Penta-CB	85/116	0.50	2.0	5.0	100	800	1500
2,3,4,5,6-Penta-CB	86	0.25	1.0	2.5	50	400	750
2,2',3,4,5-Penta-CB 2,2',3,4,5'-Penta-CB 2,3,4',5,6-Penta-CB	87/117/125	0.75	3.0	7.5	150	1200	2250
2',3,4,5,6'-Penta-CB 2,2',3,4,6-Penta-CB	88/91	0.50	2.0	5.0	100	800	1500
2,2',3,4',6-Penta-CB	89	0.25	1.0	2.5	50	400	750
2,2',3,4,6'-Penta-CB 2,2',3,4',5-Penta-CB	90/101	0.50	2.0	5.0	100	800	1500
2,2',4,5,5'-Penta-CB	93	0.25	1.0	2.5	50	400	750
2,2',3,5,6-Penta-CB	94	0.25	1.0	2.5	50	400	750
2,2',3,5,6'-Penta-CB 2,2',3,5',6-Penta-CB 2,2',3',4,6-Penta-CB	95/98/102	0.75	3.0	7.5	150	1200	2250
2,2',4,5,6'-Penta-CB	96	0.25	1.0	2.5	50	400	750
2,2',3,6,6'-Penta-CB	97	0.25	1.0	2.5	50	400	750
2,2',3,4',5-Penta-CB	99	0.25	1.0	2.5	50	400	750
2,2',4,4',5-Penta-CB	100	0.25	1.0	2.5	50	400	750
2,2',4,4',6-Penta-CB	103	0.25	1.0	2.5	50	400	750
2,2',4,5',6-Penta-CB	104	0.25	1.0	2.5	50	400	750
2,2',4,4,6'-Penta-CB	105	0.25	1.0	2.5	50	400	750
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	0.50	2.0	5.0	100	800	1500
2,3',4,4',5-Penta-CB 2,3,3',4',5-Penta-CB	107/109	0.50	2.0	5.0	100	800	1500
2,3,3',4,6-Penta-CB 2,3,3',4,5'-Penta-CB	108/112	0.50	2.0	5.0	100	800	1500
2,3,3',5,6-Penta-CB	110	0.25	1.0	2.5	50	400	750
2,3,3',4',6-Penta-CB 2,3,3',5,5'-Penta-CB	111/115	0.50	2.0	5.0	100	800	1500
2,3,4,4',6-Penta-CB	113	0.25	1.0	2.5	50	400	750
2,3,3',5',6-Penta-CB	114	0.25	1.0	2.5	50	400	750
2,3,4,4',5-Penta-CB	119	0.25	1.0	2.5	50	400	750
2,3',4,4',6-Penta-CB	120	0.25	1.0	2.5	50	400	750

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. Environmentally Significant PCBs (209)	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2,3',4,5,5'-Penta-CB	121	0.25	1.0	2.5	50	400	750
2,3',4,5',6-Penta-CB	122	0.25	1.0	2.5	50	400	750
2,3,3',4',5'-Penta-CB	123	0.25	1.0	2.5	50	400	750
2,3',4,4',5'-Penta-CB	124	0.25	1.0	2.5	50	400	750
2,3',4',5,5'-Penta-CB	126	0.25	1.0	2.5	50	400	750
3,3'4,4',5-Penta-CB	127	0.25	1.0	2.5	50	400	750
3,3',4,5,5'-Penta-CB 2,2',3,3',4,4'-Hexa-CB	128/162	0.50	2.0	5.0	100	800	1500
2,3,3',4',5,5'-Hexa-CB	129	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5-Hexa-CB	130	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5'-Hexa-CB	131	0.25	1.0	2.5	50	400	750
2,2',3,3',4,6-Hexa-CB 2,2',3,3',4,6'-Hexa-CB	133/142	0.50	2.0	5.0	100	800	1500
2,3,3',4,5',6-Hexa-CB 2,2',3,3',5,5'-Hexa-CB	132/161	0.50	2.0	5.0	100	800	1500
2,2',3,4,5,6-Hexa-CB 2,2',3,3',5,6-Hexa-CB	134/143	0.50	2.0	5.0	100	800	1500
2,2',3,3',5,6'-Hexa-CB	135	0.25	1.0	2.5	50	400	750
2,2',3,3',5,6'-Hexa-CB	136	0.25	1.0	2.5	50	400	750
2,2',3,3',6,6'-Hexa-CB	137	0.25	1.0	2.5	50	400	750
2,2',3,4,4',5-Hexa-CB 2,2',3,4,4',5'-Hexa-CB 2,3,3',4',5,6-Hexa-CB	138/163/164	0.75	3.0	7.5	150	1200	2250
2,3,3',4',5',6-Hexa-CB 2,2',3,4,4',6-Hexa-CB	139/149	0.50	2.0	5.0	100	800	1500
2,2',3,4',5',6-Hexa-CB	140	0.25	1.0	2.5	50	400	750
2,2',3,4,4',6'-Hexa-CB	141	0.25	1.0	2.5	50	400	750
2,2',3,4,5,5'-Hexa-CB	144	0.25	1.0	2.5	50	400	750
2,2',3,4,5',6-Hexa-CB	145	0.25	1.0	2.5	50	400	750
2,2',3,4,6,6'-Hexa-CB 2,2',3,4',5,5'-Hexa-CB	146/165	0.50	2.0	5.0	100	800	1500
2,3,3',5,5',6-Hexa-CB	147	0.25	1.0	2.5	50	400	750
2,2',3,4',5,6-Hexa-CB	148	0.25	1.0	2.5	50	400	750
2,2',3,4',5,6'-Hexa-CB	150	0.25	1.0	2.5	50	400	750
2,2',3,4',6,6'-Hexa-CB	151	0.25	1.0	2.5	50	400	750
2,2',3,5,5',6-Hexa-CB	152	0.25	1.0	2.5	50	400	750
2,2',3,5,6,6'-Hexa-CB	153	0.25	1.0	2.5	50	400	750
2,2',4,4',5,5'-Hexa-CB	154	0.25	1.0	2.5	50	400	750
2,2',4,4',5,6'-Hexa-CB	155	0.25	1.0	2.5	50	400	750
2,2',4,4',6,6'-Hexa-CB	156	0.25	1.0	2.5	50	400	750

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. Environmentally Significant PCBs (209)	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2,3,3',4,4',5-Hexa-CB	157	0.25	1.0	2.5	50	400	750
2,3,3',4,4',5'-Hexa-CB 2,3,3',5,5',6-Hexa-CB	158/160	0.50	2.0	5.0	100	800	1500
2,3,3',4,5,6-Hexa-CB	159	0.25	1.0	2.5	50	400	750
2,3,3',4,5,5'-Hexa-CB	166	0.25	1.0	2.5	50	400	750
2,3,4,4',5,6-Hexa-CB	167	0.25	1.0	2.5	50	400	750
2,3',4,4',5,5'-Hexa-CB	168	0.25	1.0	2.5	50	400	750
2,3',4,4',5',6-Hexa-CB	169	0.25	1.0	2.5	50	400	750
3,3',4,4',5,5'-Hexa-CB	170	0.25	1.0	2.5	50	400	750
2,2',3,3',4,4',5-Hepta-CB	171	0.25	1.0	2.5	50	400	750
2,2',3,3',4,4',6-Hepta-CB	174	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5,5'-Hepta-CB	172	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5,6-Hepta-CB	173	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5,6'-Hepta-CB	174	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5',6-Hepta-CB	175	0.25	1.0	2.5	50	400	750
2,2',3,3',4,6,6'-Hepta-CB	176	0.25	1.0	2.5	50	400	750
2,2',3,3',4',5,6-Hepta-CB	177	0.25	1.0	2.5	50	400	750
2,2',3,3',5,5',6-Hepta-CB	178	0.25	1.0	2.5	50	400	750
2,2',3,3',5,6,6'-Hepta-CB	179	0.25	1.0	2.5	50	400	750
2,2',3,4,4',5,5'-Hepta-CB	180	0.25	1.0	2.5	50	400	750
2,2',3,4,4',5,6-Hepta-CB	181	0.25	1.0	2.5	50	400	750
2,2',3,4,4',5,6'-Hepta-CB 2,2',3,4',5,5',6-Hepta-CB	182/187	0.50	2.0	5.0	100	800	1500
2,2',3,4,4',5',6-Hepta-CB	183	0.25	1.0	2.5	50	400	750
2,2',3,4,4',6,6'-Hepta-CB	184	0.25	1.0	2.5	50	400	750
2,2',3,4,5,5',6-Hepta-CB	185	0.25	1.0	2.5	50	400	750
2,2',3,4,5,6,6'Hepta-CB	186	0.25	1.0	2.5	50	400	750
2,2',3,4',5,6,6'-Hepta-CB	188	0.25	1.0	2.5	50	400	750
2,3,3',4,4',5,5'-Hepta-CB	189	0.25	1.0	2.5	50	400	750
2,3,3',4,4',5,6-Hepta-CB	190	0.25	1.0	2.5	50	400	750
2,3,3',4,4',5',6-Hepta-CB	191	0.25	1.0	2.5	50	400	750
2,3,3',4,5,5',6-Hepta-CB	192	0.25	1.0	2.5	50	400	750
2,3,3',4',5,5',6-Hepta-CB	193	0.25	1.0	2.5	50	400	750
2,2',3,3',4,4',5,5'-Octa-CB	194	0.25	1.0	2.5	50	400	750
2,2',3,3',4,4',5,6-Octa-CB	195	0.25	1.0	2.5	50	400	750
2,2',3,3',4,4',5,6'-Octa-CB 2,2',3,4,4',5,5',6-Octa-CB	196/203	0.50	2.0	5.0	100	800	1500
2,2',3,3',4,4',6,6'-Octa-CB	197	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5,5',6-Octa-CB	198	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5,5',6'-Octa-CB	199	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5,6,6'-Octa-CB	200	0.25	1.0	2.5	50	400	750

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. Environmentally Significant PCBs (209)	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2,2',3,3',4,5',6,6'-Octa-CB	201	0.25	1.0	2.5	50	400	750
2,2',3,3'5,5',6,6'-Octa-CB	202	0.25	1.0	2.5	50	400	750
2,2',3,4,4',5,6,6'-Octa-CB	204	0.25	1.0	2.5	50	400	750
2,3,3',4,4',5,5',6-Octa-CB	205	0.25	1.0	2.5	50	400	750
2,2',3,3',4,4',5,5',6-Nona-CB	206	0.25	1.0	2.5	50	400	750
2,2',3,3',4,4',5,6,6'-Nona-CB	207	0.25	1.0	2.5	50	400	750
2,2',3,3',4,5,5',6,6'-Nona-CB	208	0.25	1.0	2.5	50	400	750
Deca-CB	209	0.25	1.0	2.5	50	400	750

*Calibration Verification Solution

Table 2

Concentration of labeled PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2b. Internal Standards	Congener	CS1	CS2	CS3*	CS4	CS5
^{13}C-2-Mono-CB	1	100	100	100	100	100
^{13}C-4-Mono-CB	3	100	100	100	100	100
^{13}C-2,2'-Di-CB	4	100	100	100	100	100
^{13}C-2,5-Di-CB	9	100	100	100	100	100
^{13}C-3,3'-Di-CB	11	100	100	100	100	100
^{13}C-2,2',6-Tri-CB	19	100	100	100	100	100
^{13}C-2,4,4'-Tri-CB	28	100	100	100	100	100
^{13}C-2,4',6-Tri-CB	32	100	100	100	100	100
^{13}C-3,4,4'-Tri-CB	37	100	100	100	100	100
^{13}C-2,2',4,4'-Tetra-CB	47	100	100	100	100	100
^{13}C-2,2',4,6'-Tetra-CB	52	100	100	100	100	100
^{13}C-2,2',6,6'-Tetra-CB	54	100	100	100	100	100
^{13}C-2,3',4',5-Tetra-CB	70	100	100	100	100	100
^{13}C-3,3',4,4'-Tetra-CB	77	100	100	100	100	100
^{13}C -3,3',5,5'-Tetra-CB	80	100	100	100	100	100
^{13}C-3,4,4',5'-Tetra-CB	81	100	100	100	100	100
^{13}C-2,2',3,5',6-Penta-CB	95	100	100	100	100	100
^{13}C-2,2',3,4',5'-Penta-CB	97	100	100	100	100	100
^{13}C-2,2',4,5,5'-Penta-CB	101	100	100	100	100	100
^{13}C-2,2',4,4,6'-Penta-CB	104	100	100	100	100	100
^{13}C-2,3,3',4,4'-Penta-CB	105	100	100	100	100	100
^{13}C-2,3,4,4',5-Penta-CB	114	100	100	100	100	100

Table 2

Concentration of labeled PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2b. Internal Standards	Congener	CS1	CS2	CS3*	CS4	CS5
^{13}C-2,3',4,4',5-Penta-CB	118	100	100	100	100	100
^{13}C-2',3,4,4',5-Penta-CB	123	100	100	100	100	100
^{13}C-3,3',4,4',5-Penta-CB	126	100	100	100	100	100
^{13}C-3,3',4,5,5'-Penta-CB	127	100	100	100	100	100
^{13}C-2,2',3,4,4',5'-Hexa-CB	138	100	100	100	100	100
^{13}C-2,2',3,4,5,5'-Hexa-CB	141	100	100	100	100	100
^{13}C-2,2',4,4',5,5'-Hexa-CB	153	100	100	100	100	100
^{13}C-2,2',4,4',6,6'-Hexa-CB	155	100	100	100	100	100
^{13}C-2,3,3',4,4',5-Hexa-CB	156	100	100	100	100	100
^{13}C-2,3,3',4,4',5'-Hexa-CB	157	100	100	100	100	100
^{13}C-2,3,3',4,5,5'-Hexa-CB	159	100	100	100	100	100
^{13}C-2,3',4,4',5,5'-Hexa-CB	167	100	100	100	100	100
^{13}C-3,3',4,4',5,5'-Hexa-CB	169	100	100	100	100	100
^{13}C-2,2',3,3',4,4',5-Hepta-CB	170	100	100	100	100	100
^{13}C-2,2',3,4,4',5,5'-Hepta-CB	180	100	100	100	100	100
^{13}C-2,2',3,4',5,6,6'-Hepta-CB	188	100	100	100	100	100
^{13}C-2,3,3',4,4',5,5'-Hepta-CB	189	100	100	100	100	100
^{13}C-2,2',3,3',4,4',5,5'-Octa-CB	194	100	100	100	100	100
^{13}C-2,2',3,3',5,5',6,6'-Octa-CB	202	100	100	100	100	100
^{13}C-2,2',3,3',4,4',5,5',6-Nona-CB	206	100	100	100	100	100
^{13}C-2,2',3,3',4,5,5',6,6'-Nona-CB	208	100	100	100	100	100
^{13}C-Deca-CB	209	100	100	100	100	100
2c. Recovery Standards						
^{13}C-4,4'-Di-CB	15	100	100	100	100	100
^{13}C-2,4',5-Tri-CB	31	100	100	100	100	100
^{13}C-2,3,4,4'-Tetra-CB	60	100	100	100	100	100
^{13}C-2,3,3',5,5'-Penta-CB	111	100	100	100	100	100
^{13}C-2,2',3,4,4,',5'-Hexa-CB	128	100	100	100	100	100
^{13}C-2,3,3',4,4',5,5',6-Octa-CB	205	100	100	100	100	100
2d. Cleanup Recovery Standards						
^{13}C-2,2',5,5'-Tetra-CB	79	100	100	100	100	100
^{13}C-2,2,3,3,5,5',6-Hepta-CB	178	100	100	100	100	100

Table 3

Theoretical Ion Abundance Ratios and QC Limits

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits[1]	
			Lower	Upper
1	M/M+2	3.13	2.66	3.60
2	M/M+2	1.56	1.33	1.79
3	M/M+2	1.04	0.88	1.20
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
7	M+2/M+4	1.05	0.89	1.21
7[1]	M/M+2	0.45	0.38	0.52
8	M+2/M+4	0.89	0.76	1.02
9	M+4/M+6	1.34	1.14	1.54
9[2]	M+2/M+4	0.77	0.65	0.89
10	M+4/M+6	1.16	0.99	1.33

(1) Used for 13C-Hepa-CB
(2) Used for 13C-Nona-CB

Table 4

Quantitation Limits for the Determination of Coplanar/Mono-Ortho PCBs Only

Coplanar/Mono-Ortho PCB Target Compounds				
Target Compound	Congener Number	QL Water (pg/L)	QL Solid (pg/g)	QL Fish/Tissue (pg/g)
3,3',4,4'-Tetra-CB	77	5.0	1.25	0.5
3,4,4',5-Tetra-CB	81	5.0	1.25	0.5
2,3,3',4,4'-Penta-CB	105	5.0	1.25	0.5
2,3,4,4',5-Penta-CB	114	5.0	1.25	0.5
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	10	2.50	1.0
2',3,4,4',5-Penta-CB	123	5.0	1.25	0.5
3,3',4,4',5-Penta-CB	126	5.0	1.25	0.5
2,3,3',4,4',5-Hexa-CB	156	5.0	1.25	0.5
2,3,3',4,4',5'-Hexa-CB	157	5.0	1.25	0.5
2,3',4,4',5,5'-Hexa-CB	167	5.0	1.25	0.5
3,3',4,4',5,5'-Hexa-CB	169	5.0	1.25	0.5
2,3,3',4,4',5,5'-Hepta-CB	189	5.0	1.25	0.5

- Quantitation limits are based on 1 liter aqueous sample, 10 grams dry weight solid, and 10 grams fish/tissue sample. Final volume of 20µl for water and tissue and 50µl for solid matrices.

Table 5

Concentration of Coplanar/Mono-Ortho PCBs in Calibration Solutions

		Solution Concentration (pg/μL)					
Coplanar/Mono-Ortho PCB Congeners	**Congener No.**	**CS0**	**CS1**	**CS2**	**CS3***	**CS4**	**CS5**
3,3',4,4'-Tetra-CB	77	0.25	2.5	10	50	200	2000
3,4,4',5-Tetra-CB	81	0.25	2.5	10	50	200	2000
2,3,3',4,4'-Penta-CB	105	0.25	2.5	10	50	200	2000
2,3,4,4',5-Penta-CB	114	0.25	2.5	10	50	200	2000
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	0.5	5.0	20	100	400	4000
2',3,4,4',5-Penta-CB	123	0.25	2.5	10	50	200	2000
3,3',4,4',5-Penta-CB	126	0.25	2.5	10	50	200	2000
2,3,3',4,4',5-Hexa-CB	156	0.25	2.5	10	50	200	2000
2,3,3',4,4',5'-Hexa-CB	157	0.25	2.5	10	50	200	2000
2,3',4,4',5,5'-Hexa-CB	167	0.25	2.5	10	50	200	2000
3,3',4,4',5,5'-Hexa-CB	169	0.25	2.5	10	50	200	2000
2,3,3',4,4',5,5'-Hepta-CB	189	0.25	2.5	10	50	200	2000
Internal Standards							
^{13}C-3,3',4,4'-Tetra-CB	77	100	100	100	100	100	100
^{13}C-3,4,4',5'-Tetra-CB	81	100	100	100	100	100	100
^{13}C-2,3,3',4,4'-Penta-CB	105	100	100	100	100	100	100
^{13}C-2,3,4,4',5-Penta-CB	114	100	100	100	100	100	100
^{13}C-2,3',4,4',5-Penta-CB	118	100	100	100	100	100	100
^{13}C-2',3,4,4',5-Penta-CB	123	100	100	100	100	100	100
^{13}C-3,3',4,4',5-Penta-CB	126	100	100	100	100	100	100
^{13}C-2,3,3',4,4',5-Hexa-CB	156	100	100	100	100	100	100
^{13}C-2,3,3',4,4',5'-Hexa-CB	157	100	100	100	100	100	100
^{13}C-2,3,3',4,4',5'-Hexa-CB	167	100	100	100	100	100	100
^{13}C-3,3',4,4',5,5'-Hexa-CB	169	100	100	100	100	100	100
^{13}C-2,3,3',4,4',5,5'-Hepta-CB	189	100	100	100	100	100	100
Recovery Standards							
^{13}C-2,3,3',4'-Tetra-CB	60	100	100	100	100	100	100
^{13}C-2,2',4,5,5'-Penta-CB	101	100	100	100	100	100	100
^{13}C-2,2',3,4,4',5'-Hexa-CB	138	100	100	100	100	100	100

* Calibration Verification Solution

Table 6

Exact Masses Monitored for PCBs

Compound	Native PCBs	Internal Standard PCBs
Mono-CB	188.0393, 190.0363	200.0795, 202.0766
Di-CB	222.0003, 223.9974	234.0406, 236.0376
Tri-CB	255.9613, 257.9584	268.0016, 269.9986
Tetra-CB	289.9224, 291.9194	301.9626, 303.9597
Penta-CB	325.8804, 327.8775	337.9207, 339.9177
Hexa-CB	359.8415, 361.8385	371.8817, 373.8788
Hepta-CB	393.8025, 395.7995	403.8457, 405.8428
Octa-CB	427.7635, 429.7606	439.8038, 441.8008
Nona-CB	463.7216, 465.7186	473.7648, 475.7619
Deca-CB	497.6826, 499.6797	509.7229, 511.7199

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	**Congener No.**	**VER (%)**	**OPR (%)**	**IS Samples(%)**	**VER (%)**	**OPR (%)**	**IS Samples(%)**
2-Mono-CB	1	70-130	50-150	NA	75-125	60-135	NA
3-Mono-CB	2	70-130	50-150	NA	75-125	60-135	NA
4-Mono-CB	3	70-130	50-150	NA	75-125	60-135	NA
2,2'-Di-CB 2,6-Di-CB	4/10	70-130	50-150	NA	75-125	60-135	NA
2,3-Di-CB 2,4'-Di-CB6	5/8	70-130	50-150	NA	75-125	60-135	NA
2,3'-Di-CB	6	70-130	50-150	NA	75-125	60-135	NA
2,4-Di-CB 2,5-Di-CB	7/9	70-130	50-150	NA	75-125	60-135	NA
3,3'-Di-CB	11	70-130	50-150	NA	75-125	60-135	NA
3,4-Di-CB 3,4'-Di-CB	12/13	70-130	50-150	NA	75-125	60-135	NA
3,5-Di-CB	14	70-130	50-150	NA	75-125	60-135	NA
4,4'-D-iCB	15	70-130	50-150	NA	75-125	60-135	NA
2,2',3-Tri-CB 2,4',6-Tri-CB	16/32	70-130	50-150	NA	75-125	60-135	NA
2,2',4-Tri-CB	17	70-130	50-150	NA	75-125	60-135	NA
2,2',5-Tri-CB	18	70-130	50-150	NA	75-125	60-135	NA
2,2',6-Tri-CB	19	70-130	50-150	NA	75-125	60-135	NA
2,3,3'-Tri-CB 2,3,4-Tri-CB 2',3,4-Tri-CB	20/21/33	70-130	50-150	NA	75-125	60-135	NA
2,3,4'-Tri-CB	22	70-130	50-150	NA	75-125	60-135	NA
2,3,5-Tri-CB	23	70-130	50-150	NA	75-125	60-135	NA
2,3,6-Tri-CB 2,3',6-Tri-CB	24/27	70-130	50-150	NA	75-125	60-135	NA
2,3',4-Tri-CB	25	70-130	50-150	NA	75-125	60-135	NA
2,3',5-Tri-CB	26	70-130	50-150	NA	75-125	60-135	NA
2,4,4'-Tri-CB	28	70-130	50-150	NA	75-125	60-135	NA
2,4,5-Tri-CB	29	70-130	50-150	NA	75-125	60-135	NA
2,4,6-Tri-CB	30	70-130	50-150	NA	75-125	60-135	NA
2,4',5-Tri-CB	31	70-130	50-150	NA	75-125	60-135	NA
2,3',5'-Tri-CB	34	70-130	50-150	NA	75-125	60-135	NA
3,3',4-Tri-CB	35	70-130	50-150	NA	75-125	60-135	NA
3,3',5-Tri-CB	36	70-130	50-150	NA	75-125	60-135	NA
3,4,4'-Tri-CB	37	70-130	50-150	NA	75-125	60-135	NA
3,4,5-Tri-CB	38	70-130	50-150	NA	75-125	60-135	NA
3,4',5-Tri-CB	39	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	Congener No.	VER (%)	OPR (%)	IS Samples(%)	VER (%)	OPR (%)	IS Samples(%)
2,2’,3,3’-Tetra-CB	40	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4-Tetra-CB 2,3,4',6-Tetra-CB 2,3',4',6-Tetra-CB 2,3',5,5'-Tetra-CB	41/64/71/72	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4'-Tetra-CB 2,3,3',6-Tetra-CB	42/59	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5-Tetra-CB 2,2',4,5'-Tetra-CB	43/49	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,5’-Tetra-CB	44	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,6-Tetra-CB‘	45	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,6’-Tetra-CB	46	70-130	50-150	NA	75-125	60-135	NA
2,2’,4,4’-Tetra-CB	47	70-130	50-150	NA	75-125	60-135	NA
2,2',4,5-Tetra-CB 2,4,4',6-Tetra-CB	48/75	70-130	50-150	NA	75-125	60-135	NA
2,2’,4,6-Tetra-CB	50	70-130	50-150	NA	75-125	60-135	NA
2,2’,4,6’-Tetra-CB	51	70-130	50-150	NA	75-125	60-135	NA
2,2',5,5'-Tetra-CB 2,3',4,6-Tetra-CB	52/69	70-130	50-150	NA	75-125	60-135	NA
2,2’,5,6’-Tetra-CB	53	70-130	50-150	NA	75-125	60-135	NA
2,2’,6,6’-Tetra-CB	54	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4-Tetra-CB	55	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4'-Tetra-CB 2,3,4,4'-Tetra-CB	56/60	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,5-Tetra-CB	57	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,5’-Tetra-CB	58	70-130	50-150	NA	75-125	60-135	NA
2,3,4,5-Tetra-CB 2,3',4',5-Tetra-CB	61/70	70-130	50-150	NA	75-125	60-135	NA
2,3,4,6-Tetra-CB	62	70-130	50-150	NA	75-125	60-135	NA
2,3,4’,5-Tetra-CB	63	70-130	50-150	NA	75-125	60-135	NA
2,3,5,6-Tetra-CB	65	70-130	50-150	NA	75-125	60-135	NA
2,3’,4,5-Tetra-CB	67	70-130	50-150	NA	75-125	60-135	NA
2,3’,4,5’-Tetra-CB	68	70-130	50-150	NA	75-125	60-135	NA
2,3’,4’,5-Tetra-CB	70	70-130	50-150	NA	75-125	60-135	NA
2,3’,5’,6-Tetra-CB	73	70-130	50-150	NA	75-125	60-135	NA
2,4,4’,5-Tetra-CB	74	70-130	50-150	NA	75-125	60-135	NA
2',3,4,5-Tetra-CB 2,3',4,4'-Tetra-CB	76/66	70-130	50-150	NA	75-125	60-135	NA
3,3’,4,5-Tetra-CB	77	70-130	50-150	NA	75-125	60-135	NA
3,3’,4,5’-Tetra-CB	78	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	Congener No.	VER (%)	OPR (%)	IS Samples(%)	VER (%)	OPR (%)	IS Samples(%)
3,3',5,5'-Tetra-CB	79	70-130	50-150	NA	75-125	60-135	NA
3,4,4',5-Tetra-CB	80	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4-Penta-CB	81	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5-Penta-CB	82	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5-Penta-CB	83	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',6-Penta-CB 2,2',3,5,5'-Penta-CB	84/92	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4'-Penta-CB 2,3,4,5,6-Penta-CB	85/116	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5-Penta-CB	86	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5'-Penta-CB 2,3,4',5,6-Penta-CB 2',3,4,5,6'-Penta-CB	87/117/125	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,6-Penta-CB 2,2',3,4',6-Penta-CB	88/91	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,6'-Penta-CB	89	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5-Penta-CB 2,2',4,5,5'-Penta-CB	90/101	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5,6-Penta-CB	93	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5,6'-Penta-CB	94	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5',6-Penta-CB 2,2',3',4,6-Penta-CB 2,2',4,5,6'-Penta-CB	95/98/102	70-130	50-150	NA	75-125	60-135	NA
2,2',3,6,6'-Penta-CB	96	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5-Penta-CB	97	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',5-Penta-CB	99	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',6-Penta-CB	100	70-130	50-150	NA	75-125	60-135	NA
2,2',4,5',6-Penta-CB	103	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4,6'-Penta-CB	104	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4'-Penta-CB	105	70-130	50-150	NA	75-125	60-135	NA
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4',5-Penta-CB 2,3,3',4,6-Penta-CB	107/109	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,5'-Penta-CB 2,3,3',5,6-Penta-CB	108/112	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4',6-Penta-CB	110	70-130	50-150	NA	75-125	60-135	NA
2,3,3',5,5'-Penta-CB 2,3,4,4',6-Penta-CB	111/115	70-130	50-150	NA	75-125	60-135	NA
2,3,3',5',6-Penta-CB	113	70-130	50-150	NA	75-125	60-135	NA
2,3,4,4',5-Penta-CB	114	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	Congener No.	VER (%)	OPR (%)	IS Samples(%)	VER (%)	OPR (%)	IS Samples(%)
2,3',4,4',6-Penta-CB	119	70-130	50-150	NA	75-125	60-135	NA
2,3',4,5,5'-Penta-CB	120	70-130	50-150	NA	75-125	60-135	NA
2,3',4,5',6-Penta-CB	121	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4',5'-Penta-CB	122	70-130	50-150	NA	75-125	60-135	NA
2,3',4,4',5'-Penta-CB	123	70-130	50-150	NA	75-125	60-135	NA
2,3',4',5,5'-Penta-CB	124	70-130	50-150	NA	75-125	60-135	NA
3,3'4,4',5-Penta-CB	126	70-130	50-150	NA	75-125	60-135	NA
3,3',4,5,5'-Penta-CB	127	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4'-Hexa-CB 2,3,3',4',5,5'-Hexa-CB	128/162	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5-Hexa-CB	129	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5'-Hexa-CB	130	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,6-Hexa-CB	131	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,6'-Hexa-CB 2,3,3',4,5',6-Hexa-CB	132/161	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,5'-Hexa-CB 2,2',3,4,5,6-Hexa-CB	133/142	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,6-Hexa-CB 2,2',3,4,5,6'-Hexa-CB	134/143	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,6'-Hexa-CB	135	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',6,6'-Hexa-CB	136	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5-Hexa-CB	137	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5'-Hexa-CB 2,3,3',4',5,6-Hexa-CB 2,3,3',4',5',6-Hexa-CB	138/163/164	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',6-Hexa-CB 2,2',3,4',5',6-Hexa-CB	139/149	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',6'-Hexa-CB	140	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5,5'-Hexa-CB	141	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5',6-Hexa-CB	144	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,6,6'-Hexa-CB	145	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5,5'-Hexa-CB 2,3,3',5,5',6-Hexa-CB	146/165	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5,6-Hexa-CB	147	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5,6'-Hexa-CB	148	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',6,6'-Hexa-CB	150	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5,5',6-Hexa-CB	151	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5,6,6'-Hexa-CB	152	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',5,5'-Hexa-CB	153	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',5,6'-Hexa-CB	154	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	Congener No.	VER (%)	OPR (%)	IS Samples(%)	VER (%)	OPR (%)	IS Samples(%)
2,2',4,4',6,6'-Hexa-CB	155	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',5-Hexa-CB	156	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',5'-Hexa-CB	157	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',6-Hexa-CB 2,3,3',4,5,6-Hexa-CB	158/160	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,5,5'-Hexa-CB	159	70-130	50-150	NA	75-125	60-135	NA
2,3,4,4',5,6-Hexa-CB	166	70-130	50-150	NA	75-125	60-135	NA
2,3',4,4',5,5'-Hexa-CB	167	70-130	50-150	NA	75-125	60-135	NA
2,3',4,4',5',6-Hexa-CB	168	70-130	50-150	NA	75-125	60-135	NA
3,3',4,4',5,5'-Hexa-CB	169	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5-Hepta-CB	170	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',6-Hepta-CB	171	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5,5'-Hepta-CB	172	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5,6-Hepta-CB	173	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5,6'-Hepta-CB	174	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5',6-Hepta-CB	175	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3'4,6,6'-Hepta-CB	176	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4',5,6-Hepta-CB	177	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,5',6-Hepta-CB	178	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,6,6'-Hepta-CB	179	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5,5'-Hepta-CB	180	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5,6-Hepta-CB	181	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5,6'-Hepta-CB 2,2',3,4',5,5',6-Hepta-CB	182/187	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5',6-Hepta-CB	183	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',6,6'-Hepta-CB	184	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5,5',6-Hepta-CB	185	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5,6,6'-Hepta-CB	186	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5,6,6'-Hepta-CB	188	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',5,5'-Hepta-CB	189	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',5,6-Hepta-CB	190	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',5',6-Hepta-CB	191	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,5,5',6-Hepta-CB	192	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4',5,5',6-Hepta-CB	193	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5,5'-OctaCB	194	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5,6-OctaCB	195	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5,6'-OctaCB 2,2',3,4,4',5,5',6-OctaCB	196/203	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',6,6'-OctaCB	197	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5,5',6-OctaCB	198	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5,5',6'-OctaCB	199	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	**Congener No.**	**VER (%)**	**OPR (%)**	**IS Samples(%)**	**VER (%)**	**OPR (%)**	**IS Samples(%)**
2,2',3,3',4,5,6,6'-OctaCB	200	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5',6,6'-OctaCB	201	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,5',6,6'-OctaCB	202	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5,6,6'-OctaCB	204	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',5,5',6-OctaCB	205	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5,5',6-NonaCB	206	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5,6,6'-NonaCB	207	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5,5',6,6'-NonaCB	208	70-130	50-150	NA	75-125	60-135	NA
Deca-CB	209	70-130	50-150	NA	75-125	60-135	NA
Internal Standards							
^{13}C-2-MonoCB	1	50-150	15-140	15-150	50-145	15-145	5-145
^{13}C-4-MonoCB	3	50-150	15-140	15-150	50-145	15-145	5-145
^{13}C-2,2'-DiCB	4	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,5-DiCB	9	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-3,3'-DiCB	11	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,2',6-Tri-CB	19	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,4,4'-Tri-CB	28	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,4',6-Tri-CB	32	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-3,4,4'-Tri-CB	37	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,2',4,4'-Tetra-CB	47	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,2',5,5'-Tetra-CB	52	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,2',6,6'-Tetra-CB	54	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,3',4',5-Tetra-CB	70	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,4'-Tetra-CB	77	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,4,4',5-Tetra-CB	80	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,4'-Tetra-CB	81	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,5',6-Penta-CB	95	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4',5-Penta-CB	97	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',4,5,5'-Penta-CB	101	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',4,6,6'-Penta-CB	104	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,3',4,4'-Penta-CB	105	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,4,4',5-Penta-CB	114	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3',4,4',5-Penta-CB	118	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2',3,4,4',5-Penta-CB	123	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,4',5-Penta-CB	126	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,5,5'-Penta-CB	127	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4,4',5'-Hexa-CB	138	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4,5,5'-Hexa-CB	141	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',4,4',5,5'-Hexa-CB	153	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',4,4',6,6'-Hexa-CB	155	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,3',4,4',5-Hexa-CB	156	50-150	30-140	25-150	50-145	40-145	10-145

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	**Congener No.**	**VER (%)**	**OPR (%)**	**IS Samples(%)**	**VER (%)**	**OPR (%)**	**IS Samples(%)**
^{13}C-2,3,3',4,4',5'-Hexa-CB	157	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,3',4,5,5'-Hexa-CB	159	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3',4,4',5,5'-Hexa-CB	167	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,4',5,5'-Hexa-CB	169	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',4,4',5-Hepta-CB	170	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4,4',5,5'-Hepta-CB	180	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4',5,6,6'-Hepta-CB	188	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,3',4,4',5,5'-Hepta-CB	189	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',4,4',5,5'-OctaCB	194	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',5,5',6,6'-OctaCB	202	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',4,4',5,5',6-NonaCB	206	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',4,5,5',6,6'-NonaCB	208	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-Deca-CB	209	50-150	30-140	25-150	50-145	40-145	10-145
Cleanup Recovery Standards							
^{13}C-3,3',4,5'-Tetra-CB[1]	79	60-130	40-125	30-135	75-125	40-145	10-145
^{13}C-2,2'3,3'5,5'6-Hepta-CB[1]	178	60-130	40-125	30-135	75-125	40-145	10-145

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-1	^{13}C-PCB-1	^{13}C-PCB-15
PCB-2	^{13}C-PCB-3	^{13}C-PCB-15
PCB-3	^{13}C-PCB-3	^{13}C-PCB-15
PCB-4/10	^{13}C-PCB-4	^{13}C-PCB-15
PCB-7/9	^{13}C-PCB-9	^{13}C-PCB-15
PCB-6	^{13}C-PCB-9	^{13}C-PCB-15
PCB-5/8	^{13}C-PCB-9	^{13}C-PCB-15
PCB-14	^{13}C-PCB-11	^{13}C-PCB-15
PCB-11	^{13}C-PCB-11	^{13}C-PCB-15
PCB-12/13	^{13}C-PCB-11	^{13}C-PCB-15
PCB-15	^{13}C-PCB-11	^{13}C-PCB-15
PCB-19	^{13}C-PCB-19	^{13}C-PCB-15
PCB-30	^{13}C-PCB-19	^{13}C-PCB-15
PCB-18	^{13}C-PCB-32	^{13}C-PCB-15
PCB-17	^{13}C-PCB-32	^{13}C-PCB-15
PCB-24/27	^{13}C-PCB-32	^{13}C-PCB-15
PCB-16/32	^{13}C-PCB-32	^{13}C-PCB-15
PCB-34	^{13}C-PCB-28	^{13}C-PCB-31
PCB-23	^{13}C-PCB-28	^{13}C-PCB-31
PCB-29	^{13}C-PCB-28	^{13}C-PCB-31
PCB-26	^{13}C-PCB-28	^{13}C-PCB-31
PCB-25	^{13}C-PCB-28	^{13}C-PCB-31
PCB-31	^{13}C-PCB-28	^{13}C-PCB-31
PCB-28	^{13}C-PCB-28	^{13}C-PCB-31
PCB-20/21/33	^{13}C-PCB-28	^{13}C-PCB-31
PCB-22	^{13}C-PCB-28	^{13}C-PCB-31
PCB-36	^{13}C-PCB-37	^{13}C-PCB-31
PCB-39	^{13}C-PCB-37	^{13}C-PCB-31
PCB-38	^{13}C-PCB-37	^{13}C-PCB-31
PCB-35	^{13}C-PCB-37	^{13}C-PCB-31
PCB-37	^{13}C-PCB-37	^{13}C-PCB-31
PCB-54	^{13}C-PCB-54	^{13}C-PCB-60
PCB-50	^{13}C-PCB-54	^{13}C-PCB-60
PCB-53	^{13}C-PCB-52	^{13}C-PCB-60
PCB-51	^{13}C-PCB-52	^{13}C-PCB-60
PCB-45	^{13}C-PCB-52	^{13}C-PCB-60
PCB-46	^{13}C-PCB-52	^{13}C-PCB-60
PCB-52/69	^{13}C-PCB-52	^{13}C-PCB-60
PCB-73	^{13}C-PCB-52	^{13}C-PCB-60
PCB-43/49	^{13}C-PCB-52	^{13}C-PCB-60

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-47	^{13}C-PCB-47	^{13}C-PCB-60
PCB-48/75	^{13}C-PCB-47	^{13}C-PCB-60
PCB-65	^{13}C-PCB-47	^{13}C-PCB-60
PCB-62	^{13}C-PCB-47	^{13}C-PCB-60
PCB-44	^{13}C-PCB-47	^{13}C-PCB-60
PCB-42/59	^{13}C-PCB-47	^{13}C-PCB-60
PCB-41/64/71/72	^{13}C-PCB-47	^{13}C-PCB-60
PCB-68	^{13}C-PCB-47	^{13}C-PCB-60
PCB-40	^{13}C-PCB-47	^{13}C-PCB-60
PCB-57	^{13}C-PCB-70	^{13}C-PCB-60
PCB-67	^{13}C-PCB-70	^{13}C-PCB-60
PCB-58	^{13}C-PCB-70	^{13}C-PCB-60
PCB-63	^{13}C-PCB-70	^{13}C-PCB-60
PCB-74	^{13}C-PCB-70	^{13}C-PCB-60
PCB-61/70	^{13}C-PCB-70	^{13}C-PCB-60
PCB-76/66	^{13}C-PCB-70	^{13}C-PCB-60
PCB-80	^{13}C-PCB-80	^{13}C-PCB-60
PCB-55	^{13}C-PCB-80	^{13}C-PCB-60
PCB-56/60	^{13}C-PCB-80	^{13}C-PCB-60
PCB-79	^{13}C-PCB-80	^{13}C-PCB-60
PCB-78	^{13}C-PCB-81	^{13}C-PCB-60
PCB-81	^{13}C-PCB-81	^{13}C-PCB-60
PCB-77	^{13}C-PCB-77	^{13}C-PCB-60
PCB-104	^{13}C-PCB-104	^{13}C-PCB-111
PCB-96	^{13}C-PCB-104	^{13}C-PCB-111
PCB-103	^{13}C-PCB-104	^{13}C-PCB-111
PCB-100	^{13}C-PCB-104	^{13}C-PCB-111
PCB-94	^{13}C-PCB-95	^{13}C-PCB-111
PCB-95/98/102	^{13}C-PCB-95	^{13}C-PCB-111
PCB-93	^{13}C-PCB-95	^{13}C-PCB-111
PCB-88/91	^{13}C-PCB-95	^{13}C-PCB-111
PCB-121	^{13}C-PCB-95	^{13}C-PCB-111
PCB-84/92	^{13}C-PCB-101	^{13}C-PCB-111
PCB-89	^{13}C-PCB-101	^{13}C-PCB-111
PCB-90/101	^{13}C-PCB-101	^{13}C-PCB-111
PCB-113	^{13}C-PCB-101	^{13}C-PCB-111
PCB-99	^{13}C-PCB-101	^{13}C-PCB-111
PCB-119	^{13}C-PCB-97	^{13}C-PCB-111
PCB-108/112	^{13}C-PCB-97	^{13}C-PCB-111
PCB-83	^{13}C-PCB-97	^{13}C-PCB-111
PCB-97	^{13}C-PCB-97	^{13}C-PCB-111

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-86	^{13}C-PCB-97	^{13}C-PCB-111
PCB-87/117/125	^{13}C-PCB-97	^{13}C-PCB-111
PCB-111/115	^{13}C-PCB-97	^{13}C-PCB-111
PCB-85/116	^{13}C-PCB-97	^{13}C-PCB-111
PCB-120	^{13}C-PCB-97	^{13}C-PCB-111
PCB-110	^{13}C-PCB-97	^{13}C-PCB-111
PCB-82	^{13}C-PCB-123	^{13}C-PCB-111
PCB-124	^{13}C-PCB-123	^{13}C-PCB-111
PCB-107/109	^{13}C-PCB-123	^{13}C-PCB-111
PCB-123	^{13}C-PCB-123	^{13}C-PCB-111
PCB-106/118	^{13}C-PCB-118	^{13}C-PCB-111
PCB-114	^{13}C-PCB-114	^{13}C-PCB-128
PCB-122	^{13}C-PCB-114	^{13}C-PCB-128
PCB-105	^{13}C-PCB-105	^{13}C-PCB-128
PCB-127	^{13}C-PCB-127	^{13}C-PCB-128
PCB-126	^{13}C-PCB-126	^{13}C-PCB-128
PCB-155	^{13}C-PCB-155	^{13}C-PCB-111
PCB-150	^{13}C-PCB-155	^{13}C-PCB-111
PCB-152	^{13}C-PCB-155	^{13}C-PCB-111
PCB-145	^{13}C-PCB-155	^{13}C-PCB-111
PCB-136	^{13}C-PCB-155	^{13}C-PCB-111
PCB-148	^{13}C-PCB-155	^{13}C-PCB-111
PCB-154	^{13}C-PCB-155	^{13}C-PCB-111
PCB-151	^{13}C-PCB-155	^{13}C-PCB-111
PCB-135	^{13}C-PCB-155	^{13}C-PCB-111
PCB-144	^{13}C-PCB-155	^{13}C-PCB-111
PCB-147	^{13}C-PCB-155	^{13}C-PCB-111
PCB-139/149	^{13}C-PCB-155	^{13}C-PCB-111
PCB-140	^{13}C-PCB-155	^{13}C-PCB-111
PCB-134/143	^{13}C-PCB-153	^{13}C-PCB-128
PCB-133/142	^{13}C-PCB-153	^{13}C-PCB-128
PCB-131	^{13}C-PCB-153	^{13}C-PCB-128
PCB-146/165	^{13}C-PCB-153	^{13}C-PCB-128
PCB-132/161	^{13}C-PCB-153	^{13}C-PCB-128
PCB-153	^{13}C-PCB-153	^{13}C-PCB-128
PCB-168	^{13}C-PCB-153	^{13}C-PCB-128
PCB-141	^{13}C-PCB-141	^{13}C-PCB-128
PCB-137	^{13}C-PCB-141	^{13}C-PCB-128
PCB-130	^{13}C-PCB-141	^{13}C-PCB-128
PCB-138/163/164	^{13}C-PCB-138	^{13}C-PCB-128
PCB-158/160	^{13}C-PCB-138	^{13}C-PCB-128

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-129	^{13}C-PCB-138	^{13}C-PCB-128
PCB-166	^{13}C-PCB-159	^{13}C-PCB-128
PCB-159	^{13}C-PCB-159	^{13}C-PCB-128
PCB-128/162	^{13}C-PCB-159	^{13}C-PCB-128
PCB-167	^{13}C-PCB-167	^{13}C-PCB-128
PCB-156	^{13}C-PCB-156	^{13}C-PCB-128
PCB-157	^{13}C-PCB-157	^{13}C-PCB-128
PCB-169	^{13}C-PCB-169	^{13}C-PCB-128
PCB-188	^{13}C-PCB-188	^{13}C-PCB-128
PCB-184	^{13}C-PCB-188	^{13}C-PCB-128
PCB-179	^{13}C-PCB-188	^{13}C-PCB-128
PCB-176	^{13}C-PCB-188	^{13}C-PCB-128
PCB-186	^{13}C-PCB-188	^{13}C-PCB-128
PCB-178	^{13}C-PCB-188	^{13}C-PCB-128
PCB-175	^{13}C-PCB-188	^{13}C-PCB-128
PCB-182/187	^{13}C-PCB-188	^{13}C-PCB-128
PCB-183	^{13}C-PCB-188	^{13}C-PCB-128
PCB-185	^{13}C-PCB-180	^{13}C-PCB-128
PCB-174	^{13}C-PCB-180	^{13}C-PCB-128
PCB-181	^{13}C-PCB-180	^{13}C-PCB-128
PCB-177	^{13}C-PCB-180	^{13}C-PCB-128
PCB-171	^{13}C-PCB-180	^{13}C-PCB-128
PCB-173	^{13}C-PCB-180	^{13}C-PCB-128
PCB-172	^{13}C-PCB-180	^{13}C-PCB-128
PCB-192	^{13}C-PCB-180	^{13}C-PCB-128
PCB-180	^{13}C-PCB-180	^{13}C-PCB-128
PCB-193	^{13}C-PCB-180	^{13}C-PCB-128
PCB-191	^{13}C-PCB-180	^{13}C-PCB-128
PCB-170	^{13}C-PCB-170	^{13}C-PCB-128
PCB-190	^{13}C-PCB-170	^{13}C-PCB-128
PCB-189	^{13}C-PCB-189	^{13}C-PCB-128
PCB-202	^{13}C-PCB-202	^{13}C-PCB-128
PCB-201	^{13}C-PCB-202	^{13}C-PCB-128
PCB-204	^{13}C-PCB-202	^{13}C-PCB-128
PCB-197	^{13}C-PCB-202	^{13}C-PCB-128
PCB-200	^{13}C-PCB-202	^{13}C-PCB-128
PCB-198	^{13}C-PCB-202	^{13}C-PCB-128
PCB-199	^{13}C-PCB-202	^{13}C-PCB-128
PCB-196/203	^{13}C-PCB-202	^{13}C-PCB-128
PCB-195	^{13}C-PCB-194	^{13}C-PCB-205
PCB-194	^{13}C-PCB-194	^{13}C-PCB-205

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-205	^{13}C-PCB-194	^{13}C-PCB-205
PCB-208	^{13}C-PCB-208	^{13}C-PCB-205
PCB-207	^{13}C-PCB-208	^{13}C-PCB-205
PCB-206	^{13}C-PCB-206	^{13}C-PCB-205
PCB-209	^{13}C-PCB-209	^{13}C-PCB-205
^{13}C-PCB-15 (RS)		
^{13}C-PCB-31 (RS)		
^{13}C-PCB-60 (RS)		
^{13}C-PCB-111 (RS)		
^{13}C-PCB-128 (RS)		
^{13}C-PCB-205 (RS)		
	^{13}C-PCB-79 (CRS)	^{13}C-PCB-60
	^{13}C-PCB-178 (CRS)	^{13}C-PCB-128
	^{13}C-PCB-79 (PS)	^{13}C-PCB-81
	^{13}C-PCB-178 (PS)	^{13}C-PCB-180

GLOSSARY

Symbols

°C	Celsius
µL	microliter
µm	micrometer
<	less than
>	greater than
%	percent

Alphabetical Abbreviations

cm	centimeter
g	gram
L	liter
M	molecular ion
m	meter
mg	milligram
ml	milliliter
mm	millimeter
m/z	mass-to-charge ration
N	normal
pg	picogram
ppb	part-per-billion
ppm	part-per-million
ppq	part-per-quadrillion
ppt	part-per-trillion
v/v	volume per unit volume
w/v	weight per unit volume

Definitions and Acronyms

Analyte – a PCB tested for by this method. The analytes are listed in Table 1.

Calibration Standard – a solution prepared from a secondary standard and/or stock solutions used to calibrate the response of the instrument.

Calibration Verification Standard – the mid-point calibration standard (CS-3) that is used to verify calibration.

CB – chlorinated biphenyl congener.

CS-1, CS-2, CS-3, CS-4, CS-5 – See calibration standards and Table 2.

HRMS – High resolution mass spectrometry.

Internal Standard – an internal standard is a labeled PCB which is added to all field samples, blanks and other quality control samples before extraction. It is also included in the calibration solutions. Internal standards are used to measure the concentration of the analyte and surrogate compounds.

IPR – initial precision and recovery, four aliquots of a reference matrix spiked with the analytes of interest and labeled compounds and analyzed to establish the ability of the laboratory to generated acceptable precision and recovery.

Isotope Dilution – a means of determining a naturally occurring (native) compound by reference to the same compound in which one or more atoms has been isotopically enriched.

Matrix Spike (MS) – a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte is concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on the methods recovery efficiency.

Method Blank (MB) – an aliquot of reagent water that is treated exactly as a sample including

exposure to all glassware, equipment, solvents, regents, internal standards, and surrogates that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

Minimum Level - The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all Method-specified sample weights, volumes, and cleanup procedures have been employed.

OPR – ongoing precision and recovery standard, a method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

PCB – polychlorinated biphenyl

PFK – perfluorokerosene, a mixture of compounds used to calibrate the exact m/z scale in the HRMS.

Practical Quantitation Limit (PQL) – The PQL is a limit for each compound at or below which data must not be reported.

Reagent Water – water demonstrated to be free from the analytes of interest and potentially interfering substances at the method detection limit for the analyte.

Recovery Standard – A recovery standard is a labeled compound, which is added to the extracts of all samples, blanks, and QC samples before analysis. It is also included in the calibration solutions. The response of the internal standards relative to the recovery standard is used to estimate the recovery of the internal standards. The internal standard recovery is an indicator of the overall performance of the analysis.

Relative Response Factor – The relative response factor is the response of the mass spectrometer to a known amount of an analyte or labeled compound relative to a known amount of an internal standard or another labeled compound.

Signal-to-Noise Ratio (S/N) – the height of the signal as measured from the mean (average) of the noise to the peak maximum divided by the width of the noise.

Stock Solution – a solution containing an analyte that is prepared using a reference material traceable to a source that will attest to the purity and authenticity of the reference material.

SOP 26, Revision 9 / SOP 31, Revision 10	Amendment
POLYCHLORINATED DIBENZO DIOXIN/FURANS BY USEPA METHOD 1613B PREPARATION AND ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBS) BY METHOD 1668A/C	
Management: Matthew Moier	
Quality Assurance: RW Harrison	
Effective Date: January 14, 2013	

Description of Amendment
Requirements for extraction and analysis of samples from the State of Wisconsin: All samples from the State of Wisconsin shall be analyzed only on instruments with a valid LOD study for that parameter, matrix, and method, i.e., DoD instruments. All samples from the State of Wisconsin shall be extracted with a measurement of precision, such as an MS/MSD, LCSD, or sample duplicate, per batch and precision assessed. All reportable values for samples from the State of Wisconsin shall be within the range of calibration. Concentrations shall not be "E" qualified.