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## **METHOD 508A**

### **I. SCOPE AND APPLICATION:**

This procedure may be use for screening finished drinking water, raw source water, or drinking water in any treatment stage for polychlorinated biphenyls (PCBs) This procedure is applicable to samples containing PCBs as single congeners or as complex mixtures such as weathered, intact, or mixtures of commercial Aroclors. The procedure is incapable of identifying the parent PCBs because the original PCBs are chemically converted to a common product, decachlorobiphenyl (DCB). This method has only been evaluated using Aroclors and 2-chlorobiphenyl as a source of PCBs.

<b><u>Analyte</u></b>	<b><u>Chemical Abstract Services Registry Numbers (CASRN)</u></b>
Aroclor 1016 (PCB-1016)	12674-11-2
Aroclor 1221 (PCB-1221)	11104-28-2
Aroclor 1232 (PCB-1232)	11141-16-5
Aroclor 1242 (PCB-1242)	53469-21-9
Aroclor 1248 (PCB-1248)	12672-29-6
Aroclor 1254 (PCB-1254)	11097-69-1
Aroclor 1260 (PCB-1260)	11096-82-5
2-Chlorobiphenyl	

### **II. REAGENTS: none**

### **III. MATERIALS:**

- 1-liter amber borosilicate sample bottle fitted with screw caps lined with TFE-fluorocarbon. Collect samples in duplicate.
- Latex gloves
- Protective eyewear
- Paper towels
- Kim wipes
- Pliers

### **IV. PROCEDURE:**

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.
2. Open the tap and allow the system to flush for about 10 minutes. This should be adequate to allow the water temperature to stabilize and get a representative sample.
3. Adjust the water flow to about 1000 ml/minute or slow enough that no air bubbles purge the sample when collecting from the flowing stream.
4. Remove the cap from the 1-liter container. Do not rinse the container as it has already been acid rinsed.
5. To fill, tip the bottle to about a 45° angle into the stream of water. Ensure the stream is sufficiently slow so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to within approximately ½ inch of the mouth.
6. Remove the bottle from the flow and recap. Collect samples in duplicate.

### **V. SAMPLE TRANSPORT:**

After obtaining the water samples, attach the completed chain of custody seal around the plastic cap of each 1-liter bottle. Place the sample bottle into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature until analysis. Always use chopped, grated, or dry ice when chilling the semi-volatile samples for transportation. Never use “blue ice” as the semi-volatile samples may never chill adequately. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory.

## VI. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

## VII. SUMMARY OF METHOD:

METHOD 508A: The sample volume is measured and spiked with a surrogate (and any target compounds for quality control purposes) then transferred into a 2-liter separatory funnel containing 50g of NaCl. The extraction process begins by adding 60 ml of methylene chloride to the sample bottle, which is capped and mixed to rinse the inner walls of the bottle, and then transferred to a separatory funnel. The sample is extracted by shaking the separatory funnel for two minutes with periodic venting to release excess pressure. The organic layer is allowed to separate from the water phase. The organic layer is removed and placed in an erlenmeyer flask. The process of adding methylene chloride to the water sample, extraction, and separation of the organic layer is performed two more times. After each extraction, the methylene chloride extract is combined in the erlenmeyer flask.

The entire contents of the erlenmeyer flask is concentrated to approximately 10-20 ml using a Kuderna-Danish (K-D) concentrator (this is an evaporative process) and passed through a drying tube containing about 10 cm of pre-rinsed anhydrous sodium sulfate to remove water and collected in an evaporative flask. The drying column is rinsed with 20-30 ml of methylene chloride to remove any target compounds adhering to the drying tube and collected in the evaporative flask containing the “dried” extract which is then concentrated (evaporated) to a volume of 1.0 ml.

The “dried” extract is transferred to a screw cap test tube and the KD ampule is rinsed three times using about 250 $\mu$ l of chloroform for each rinse. The extract is concentrated to a volume of 0.1 ml by directing a stream of nitrogen gas at about 100 ml/min into the test tube while warming the base of the test tube in a 50°C water bath. An additional 2 ml of chloroform is added to the extract and again concentrated to 0.1 ml. The process of adding chloroform and concentrating the extract is known as a “solvent exchange”. The original extraction solvent, which contains the target compounds, is being exchanged so that the target compounds will be present in a chloroform solvent matrix.

The “perchlorination” process begins when 100 mg of iron powder (acting as a catalyst) is added to the 0.1 ml of extract. Using a disposable pipette, 25 drops of SbCl<sub>5</sub> (antimony pentachloride) is added to the extract and heated to 205°C for about 40 minutes. After cooling, 0.5 ml of 1:1 hydrochloric acid is added to the perchlorinated extract along with 2 ml of hexane, the test tube is capped and shaken for several minutes. After the two phases separate, the top layer is removed and placed in a 5 ml volumetric flask and re-extracted two additional times: first with 2 ml of hexane and then with 1 ml of hexane, adding the extracts to the 5 ml volumetric flask. The flask is then brought to volume using hexane.

## **VII. SUMMARY OF METHOD (continued):**

In a 15 ml screw cap test tube, 4 ml of 0.1 N NaHCO<sub>3</sub> (sodium bicarbonate) is added as well as the 5 ml of extract and shaken for a minute. After the two phases separate, remove the top layer into a second 15 ml test tube and add 4 ml of reagent water. Cap and shake the test tube for another minute. Remove the top layer and transfer to a hermetically sealed container for analysis.

A 2µl aliquot of the purified extract is injected into a gas chromatograph (GC) for separation that is equipped with an electron capture detector (ECD) for analysis and measurement. The GC is calibrated using DCB as the standard.