

POLYCHLOROBIPHENYLS

5503

mixture: $C_{12}H_{10-x}Cl_x$
[where x = 1 to 10]

MW: ca. 258 (42% Cl ; $C_{12}H_7Cl_3$);
ca. 326 (54% Cl ; $C_{12}H_5Cl_5$)

CAS: Table 1

RTECS: Table 1

METHOD: 5503, Issue 2

EVALUATION: PARTIAL

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OSHA : 1 mg/m³ (42% Cl);
0.5 mg/m³ (54% Cl)

NIOSH: 0.001 mg/m³/10 h (carcinogen)

ACGIH: 1 mg/m³ (42% Cl) (skin)
0.5 mg/m³ (54% Cl) (skin)

PROPERTIES: 42% Cl: BP 325 to 366 °C; MP -19 °C;
d 1.38 g/mL @ 25 °C;

VP 0.01 Pa (8 x 10⁻⁵ mm Hg;
1 mg/m³) @ 20 °C

54% Cl: BP 365 to 390 °C; MP 10 °C;
d 1.54 g/mL @ 25 °C; VP
0.0004 Pa (3 x 10⁻⁶ mm Hg;
0.05 mg/m³) @ 20 °C

SYNONYMS: PCB; 1,1'-biphenyl chloro; chlorodiphenyl, 42% Cl (Aroclor 1242); and 54% Cl (Aroclor 1254)

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER + SOLID SORBENT (13-mm glass fiber + Florisil, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, ECD (⁶³ Ni)
FLOW RATE:	0.05 to 0.2 L/min or less	ANALYTE:	polychlorobiphenyls
VOL-MIN:	1 L @ 0.5 mg/m ³	DESORPTION:	filter + front section, 5 mL hexane; back section, 2 mL hexane
-MAX:	50 L	INJECTION	
SHIPMENT:	transfer filters to glass vials after sampling	VOLUME:	4-μL with 1-μL backflush
SAMPLE		TEMPERATURE-INJECTION:	250 to 300 °C
STABILITY:	unknown for filters; 2 months for Florisil tubes [1]	-DETECTOR:	300 to 325 °C
BLANKS:	2 to 10 field blanks per set	-COLUMN:	180 °C
ACCURACY		CARRIER GAS:	N ₂ , 40 mL/min
RANGE STUDIED:	not studied	COLUMN:	glass, 1.8 m x 2-mm ID, 1.5% OV-17/1.95% QF-1 on 80/100 mesh Chromosorb WHP
BIAS:	none identified	CALIBRATION:	standard PCB mixture in hexane
OVERALL PRECISION (\hat{S}_{RT}):	not evaluated	RANGE:	0.4 to 4 μg per sample [2]
ACCURACY:	not determined	ESTIMATED LOD:	0.03 μg per sample [2]
		PRECISION (\hat{S}_j):	0.044 [1]

APPLICABILITY: The working range is 0.01 to 10 mg/m³ for a 40-L air sample [1]. With modifications, surface wipe samples may be analyzed [3,4].

INTERFERENCES: Chlorinated pesticides, such as DDT and DDE, may interfere with quantification of PCB. Sulfur-containing compounds in petroleum products also interfere [5].

OTHER METHODS: This method revises methods S120 [6] and P&CAM 244 [1]. Methods S121 [7] and P&CAM 253 [8] for PCB have not been revised.

REAGENTS:

1. Hexane, pesticide quality.
2. Florisil, 30/48 mesh sieved from 30/60 mesh. After sieving, dry at 105 °C for 45 min. Mix the cooled Florisil with 3% (w/w) distilled water.
3. Nitrogen, purified.
4. Stock standard solution of the PCB in methanol or isooctane (commercially available).*

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 13-mm glass fiber filter without binders in a Swinnex cassette (Cat. No. SX 0001300, Millipore Corp.) followed by a glass tube, 7 cm long, 6-mm OD, 4-mm ID containing two sections of 30/48 mesh deactivated Florisil. The front section is preceded by glass wool and contains 100 mg and the backup section contains 50 mg; urethane foam between sections and behind the backup section. (SKC 226-39, Supelco ORBO-60, or equivalent) Join the cassette and Florisil tube with PVC tubing, 3/8" L x 9/32" OD x 5/32" ID, on the outlet of the cassette and with another piece of PVC tubing, 3/4" L x 5/16" OD x 3/16" ID, complete the union.
2. Personal sampling pump, 0.05 to 0.2 L/min, with flexible connecting tubing.
3. Tweezers.
4. Vials, glass, 4- and 7-mL, with aluminum or PTFE-lined caps
5. Gas chromatograph, electron capture detection (⁶³Ni), integrator and column (page 5503-1).
6. Volumetric flasks, 10-mL and other convenient sizes for preparing standards.
7. Syringe, 10-μL.

SPECIAL PRECAUTIONS: Avoid prolonged or repeated contact of skin with PCB and prolonged or repeated breathing of the vapor [9-11].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the Florisil tube immediately before sampling. Connect Florisil tube to Swinnex cassette and attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.05 and 0.2 L/min for a total sample size of 1 to 50 L.
NOTE: At low PCB concentrations, the sampler was found to be efficient when operated at flow rates up to 1 L/min, for 24 hours [4]. Under these conditions, the limit of detection was 0.02 μg/m³.
4. Transfer the glass fiber filters to 7-mL vials. Cap the Florisil tubes with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the glass wool and 100-mg Florisil bed in the same 7-mL vial in which the filter was stored. Add 5.0 mL hexane.
6. In a 4-mL vial, place the 50-mg Florisil bed including the two urethane plugs. Add 2.0 mL hexane.
7. Allow to stand 20 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 10 to 500 ng/mL PCB.
 - a. Add known amounts of stock standard solution to hexane in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (sum of areas of selected peaks vs. ng PCB per sample).
9. Determine desorption efficiency (DE) at least once for each lot of glass fiber filters and Florisil used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank Florisil tube.
 - b. Inject known amounts of stock standard solution directly onto front sorbent section and onto a media blank filter with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. µg PCB recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5503-1. Inject sample aliquot manually using solvent flush technique or with autosampler.

NOTE 1: Where individual identification of PCB is needed, a procedure using a capillary column may be used [12].

NOTE 2: If peak area is above the linear range of the working standards, dilute with hexane, reanalyze and apply the appropriate dilution factor in calculations.
12. Sum the areas for five or more selected peaks.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE) of PCB found on the glass fiber filter (W) and in the Florisil front (W_f) and back (W_b) sorbent sections, and in the average media blank filter (B) and front (B_f) and back (B_b) sorbent sections.

NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C, of PCB in the air volume sampled, V (L):

$$C = \frac{(W + W_f + W_b - B - B_f - B_b)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

This method uses 13-mm glass fiber filters which have not been evaluated for collecting PCB. In Method S120, however, Aroclor 1242 was completely recovered from 37-mm glass fiber filters using 15 mL isooctane [8,13,14]. With 5 mL of hexane, Aroclor 1016 was also completely recovered from 100-mg Florisil beds after one-day storage [1]. Thus, with no adsorption effect likely on glass fiber filters for PCB, 5 mL hexane should be adequate to completely extract PCB from combined filters and front sorbent sections. Sample stability on glass fiber filters has not been investigated. Breakthrough volume was >48 L for the Florisil tube at 75% RH in an atmosphere containing 10 µg/m³ Aroclor 1016 [1].

REFERENCES:

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METHOD REVISED BY:

James E. Arnold, NIOSH/DPSE; S120 originally validated under NIOSH Contract 210-76-0123.

Table 1. General Information.

<u>Compound</u>	<u>CAS</u>	<u>RTECS</u>
Polychlorinated Biphenyls	1336-36-3	TQ1350000
Chlorobiphenyl	27323-18-8	DV2063000
Aroclor 1016 (41% Cl)	12674-11-2	TQ1351000
Aroclor 1242 (42% Cl)	53469-21-9	TQ1356000
Aroclor 1254 (54% Cl)	11097-69-1	TQ1360000

Table 2. Composition of some Aroclors [15].

<u>Major Components</u>	<u>Aroclor 1016</u>	<u>Aroclor 1242</u>	<u>Aroclor 1254</u>
Biphenyl	0.1%	<0.1%	<0.1%
Monochlorobiphenyls	1	1	<0.1
Dichlorobiphenyls	20	16	0.5
Trichlorobiphenyls	57	49	1
Tetrachlorobiphenyls	21	25	21
Pentachlorobiphenyls	1	8	48
Hexachlorobiphenyls	<0.1	1	23
Heptachlorobiphenyls	none detected	<0.1	6
Octachlorobiphenyls	none detected	none detected	none detected