



STANDARD OPERATING PROCEDURES

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REV: 0.0
DATE: 09/16/94

DETERMINATION OF POLYCHLORINATED BIPHENYLS IN AIR

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SUPERCEDES: SOP #1815; Revision 0; 02/28/90; U.S. EPA Contract EP-W-09-031.



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1.0 SCOPE AND APPLICATION

This method was developed to determine the concentration of polychlorinated biphenyls (PCBs) in air. The method can be slightly modified for testing pesticides in air.

The method detection limit (MDL) was calculated by the rule given in "Federal Register, part VIII" namely three times the standard deviation of the lowest concentration in the linear range of the Gas Chromatograph (GC). In this case, five instead of seven replicates were used.

These are standard (i.e. typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

As NIOSH Method 5503 was used, the front and back Floristic beds of the tube were desorbed in 5 mL and 2 mL hexane, consecutively. Florisil should be desorbed for about 30 minutes with occasional agitation.

3.0 SAMPLE PRESERVATION AND STABILITY

Samples can be kept as collected in Floristic tubes for two months.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Chlorinated pesticides, such as DDT and DDE, may interfere with quantitation of PCBs. Sulfur-containing compounds in petroleum products might also interfere.

5.0 EQUIPMENT/APPARATUS

- Florisil tubes: 100/50 mg, 30/48 mesh by SKC Inc. or equivalent
- Glass vials, 4 mL and 10 mL, with PTFE-lined caps
- Gas chromatograph with electron capture detector (ECD), integrator, capillary column 0.5 mm ID (Supelco DB-608 or equivalent)
- Volumetric flasks, 10 mL and other convenient sizes
- Micro syringes, 10 μ L and other sizes as needed

6.0 REAGENTS

- Hexane, pesticides grade
- Argon/methane (5%) gas mixture



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- Standard stock solutions of PCBs in isooctane, commercially available

7.0 ANALYTICAL PROCEDURE

7.1 Sample Preparation

1. Transfer the front Florisil bed of the tube with the glass wool filter to a 10-mL vial. Add 5.0 mL hexane.
2. Transfer the back Florisil bed of the tube including the two urethane plugs to a 4-mL vial. Add 2.0 mL hexane.
3. Allow to stand for 30 minutes with occasional agitation.

7.2 Calibration and Quality Control

1. Set the gas chromatograph to manufacturer's recommendations and to the following conditions:

Column:	Supelco DB-608, 30 meter fused silica, 0.5 mm ID, 0.83 mm film thickness
Initial Temp.:	180°C
Initial Time:	2 minutes
Temp. Rate:	4°C/minute up to 240°C 1°C/minute up to 250°C
Final Temp.:	250°C
Final Time:	8 minutes
2. Calibrate the GC by running standard solutions over the range 250-5000 ng PCBs/mL, and prepare a calibration graph using the sum of areas or peak heights of selected peaks versus ng PCBs/mL.
3. Determine desorption efficiency (DE) at least once for each lot of Florisil tubes, use the same concentration levels of the calibration curve.
 - Remove and discard the back section of a blank tube.
 - Inject known amount of stock standard solution directly onto front section of the blank Florisil tube.
 - Cap the tube. Allow to stand overnight.
 - Desorb the front Florisil section in 5.0 mL hexane.
 - Prepare a graph for DE versus PCBs recovered.

7.3 Measurement

1. Inject sample aliquot manually using solvent flush technique or with an autosampler.
2. If peak area or height is above the linear range of the working standards, dilute with hexane, reanalyze and apply the appropriate dilution factor in calculations.



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3. Sum the peak areas or heights for five or more selected peaks.

8.0 CALCULATIONS

1. Calculate MDL according to the rule mentioned in Section 1.
2. Determine the mass, ng of PCBs (corrected for DE) found on the glass wood filter and Florisil front section (W_F), and the back Florisil section including the two urethane plugs (W_B).
3. Repeat the previous step for a blank Florisil tube to determine (B_F) and (B_B).

$$\text{Concentration (mg/m}^3\text{)} = \frac{W_F + W_B - B_F - B_B \times 10^3}{V_S}$$

4. Calculate the concentration, C, of PCBs in air volume sampled, (V_S) in liters.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

1. A laboratory blank shall be desorbed with every batch of samples.
2. Two laboratory spikes shall be analyzed with every 10 samples, or less.
3. The Relative Percent Difference (RPD) should fall below 10%. The percent recoveries of the laboratory spikes should be between 90-110%.
4. Performance evaluation sample shall be analyzed along with the calibration curve.

10.0 DATA VALIDATION

If data are not within the specified ranges of QA/QC, data shall be flagged questionable.

11.0 HEALTH AND SAFETY

Gloves, latex type, must be used when handling the organic solvents. All organic solvents must be handled under a fume hood with the recommended ventilation rate.

When working with potential hazardous materials, follow U.S. EPA, OSHA and corporate health and safety procedures.

12.0 REFERENCES

1. NIOSH Method 5503, dated August 15, 1987, revised by James E. Arnold, NIOSH/DPSE.
2. Code of Federal Regulations, Section 40, Method 608.