

CHLORINATED DIPHENYL OXIDE

5025

$C_{12}H_4Cl_6O$

MW: 376.88

CAS: 55720-99-5

RTECS: KO4200000

METHOD: 5025, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 August 1987

Issue 2: 15 August 1994

OSHA : 0.5 mg/m³
 NIOSH: 0.5 mg/m³
 ACGIH: 0.5 mg/m³

PROPERTIES: Table 1

SYNONYMS: chlorinated diphenyl ether; hexachlorodiphenyl oxide

APPLICABILITY: The working range is 0.05 to 1.5 mg/m³ for a 90-L air sample. The method has been evaluated only for the hexachloro derivative. The sampler may not be adequate for the monochloro or dichloro derivatives, which have higher vap or pressures.

INTERFERENCES: None identified.

OTHER METHODS: This revises Method S119 [2].

REAGENTS:

1. Isooctane, chromatographic quality.
2. Calibration stock solution, 2 µg/µL chlorinated diphenyl oxide* in isooctane. Prepare in duplicate.
3. Nitrogen, purified.
4. Hydrogen, prepurified.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: cellulose ester membrane filter, 0.8-µm pore size, 37-mm diameter, supported by cellulose backup pad in two-piece polystyrene filter holder.
2. Personal sampling pump, 0.5 to 1.5 L/min, with flexible connecting tubing.
3. Gas chromatograph, electrolytic conductivity detector, effluent vent, integrator and column (page 5025-1).
4. Vials, 20-mL, PTFE-lined caps, for shipping filters.
5. Syringes, 25-µL, readable to 0.1 µL.
6. Volumetric flasks, 10-mL.
7. Pipet, 10-mL.
8. Tweezers.

SPECIAL PRECAUTIONS: Prolonged skin contact with chlorinated diphenyl oxide can cause chloracne; acute and chronic exposure can cause liver damage [3,4].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 0.5 and 1.5 L/min for a total sample size of 8 to 200 L.
3. Within 1 h of sampling, using tweezers, carefully transfer the filter and backup pad to a vial.

SAMPLE PREPARATION:

4. Pipet 10.0 mL isooctane into each vial containing a sample or blank filter and backup pad. Seal and gently swirl the vial to wet the filter and backup pad. Allow to stand 2 h.

CALIBRATION AND QUALITY CONTROL:

5. Calibrate daily with at least six working standards.
 - a. Add known amounts of calibration stock solution to isooctane in 10-mL volumetric flasks and dilute to the mark to obtain concentrations in the range 0.4 to 15 µg chlorinated diphenyl oxide/mL.
 - b. Analyze with samples and blanks (steps 8 and 9).
 - c. Prepare calibration graph (sum of areas of selected peaks vs. µg chlorinated diphenyl oxide).
6. Determine recovery (R) at least once for each batch of filters used for sampling in the calibration range. Prepare three filters at each of five levels plus three media blanks.
 - a. Deposit a known amount of calibration stock solution onto the filter. Allow filters to air dry.
 - b. Store samples overnight in vials.
 - c. Prepare (step 4) and analyze with working standards (steps 8 and 9).
 - d. Graph R vs. µg chlorinated diphenyl oxide.
7. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and R graph are in control.

MEASUREMENT:

8. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5025-1. Inject sample aliquot manually using solvent flush technique or with autosampler. Open the effluent vent to keep the solvent from passing into the detector. Close the effluent vent after the solvent peak has eluted.
NOTE: Under conditions given, the solvent elutes in about 20 sec. The large quantity of solvent injected may cause malfunction of the conductivity cell, unless it is vented.
9. Sum the areas of selected peaks.
NOTE: Use a bulk sample if necessary to identify the appropriate chromatographic peaks.

CALCULATIONS:

10. Determine the mass, μg (corrected for R) of chlorinated diphenyl oxide found in the sample (W) and in the average media blank (B) from the calibration graph.
11. Calculate concentration, C, of chlorinated diphenyl oxide in the air volume sampled, V (L):

$$C = \frac{W - B}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S119 was evaluated over the range 0.1 to 1 mg/m^3 hexachlorodiphenyl oxide at 22 °C and 767 mm Hg using 90-L samples [1]. Overall precision, \bar{S}_{rT} , was 0.070; no reference method was used. The test atmospheres were generated from solutions (0.25 to 0.5% w/v) of chlorinated diphenyl oxide (Chem Samples) in toluene, using a fluid aspirator, cyclone and an impactor. Sampling with two filters in series was conducted in an atmosphere containing 1 mg/m^3 chlorinated diphenyl oxide. Chlorinated diphenyl oxide was found only on the front filters (with LOD = 0.002 mg/m^3). Filters enriched with 100 μg chlorinated diphenyl oxide were analyzed after passing 100 L of air through them. The resulting recovery of the analyte was 99.75% with $\bar{S}_r = 0.017\%$. The recovery of chlorinated diphenyl oxide from enriched filters through which no air was drawn was 1.012 in the range 23 to 90 μg per sample. Storage stability of the sample was not tested.

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S119, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as Stock No. PB 274-248 from NTIS, Springfield, VA 22161.
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S119, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as Stock #PB83-154609 from NTIS, Springfield, VA 22161.
- [4] Occupational Diseases, A Guide to Their Recognition, revised ed., 255-256, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-181 (1978).
- [5] Patty, F. A. Industrial Hygiene and Toxicology, 2nd ed, Vol. 2, 1706-1707, Interscience, New York (1963).

METHOD REVISED BY:

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Table 1. Properties of chlorinated diphenyl oxide [3,5].

<u>Compound</u>	<u>BP, °C (8 mm Hg)</u>	<u>d, g/mL 25 °C</u>	<u>Vapor Pressure @ 25 °C</u>			
			<u>Pa</u>	<u>mm Hg</u>	<u>mg/m³</u>	<u>1 ppm in mg/m³ @ NTP</u>
Monochloro-	153	1.19	0.93	0.007	77	8.37
Dichloro-	168.2	1.32	0.08	0.0006	8	9.78
Hexachloro-	230 to 260	1.57 @ 20 °C	<0.008	<0.00006	<1	15.41