

LINDANE
See ALDRIN, Method 5502, for Procedure

5502

C₆H₆Cl₆ MW: 290.82 CAS: 58-89-9 RTECS: GV4900000

METHOD: 5502, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA : 0.5 mg/m³ (skin)
NIOSH: 0.5 mg/m³ (skin)
ACGIH: 0.5 mg/m³ (skin)

PROPERTIES: solid; MP 112.5 °C; VP 0.0013 Pa
(9.4 x 10⁻⁶ mm Hg; 0.15 mg/m³) @ 20 °C

SYNONYMS: gamma-hexachlorocyclohexane; benzenehexachloride; gamma-BHC.

| SAMPLING | | MEASUREMENT | |
|---|--|---------------------------------------|---|
| SAMPLER: | FILTER AND BUBBLER (glass fiber + 15 mL isooctane) | TECHNIQUE: | GAS CHROMATOGRAPHY, ELECTROLYTIC CONDUCTIVITY DETECTOR |
| FLOW RATE: | 0.2 to 1 L/min | ANALYTE: | LINDANE |
| VOL-MIN: | 18 L @ 0.25 mg/m ³ | FILTER | |
| -MAX: | 240 L | EXTRACTION: | isooctane |
| SHIPMENT: | transfer bubbler solutions and filters in scintillation vials; pack carefully | INJECTION | |
| SAMPLE | | VOLUME: | 15 µL |
| STABILITY: | at least 1 week @ 25 °C | TEMPERATURE-FURNACE: | 750 to 770 °C |
| BLANKS: | 2 to 10 field blanks per set | -TRANSFER: | 225 °C |
| | | -VENT: | 205 to 260 °C |
| | | -COLUMN: | 160 to 190 °C |
| | | GASES-H₂ (furnace): | 150 to 160 mL/min |
| | | -N₂ (carrier): | 140 mL/min |
| | | COLUMN: | glass, 1.2 m x 3-mm OD; 5% SE-30 on 80/100 mesh acid-washed DMCS Chromosorb W or equivalent |
| ACCURACY | | CALIBRATION: | solution of analyte in isooctane |
| RANGE STUDIED: | 0.3 to 1.7 mg/m ³ [1]; | RANGE: | 5 to 135 µg per sample |
| BIAS: | - 0.44% | ESTIMATED LOD: | 3 µg per sample |
| OVERALL PRECISION (Ŝ_{r,T}): | 0.086 [1] | PRECISION (Ŝ_r): | 0.013 [1] |
| ACCURACY: | ± 16.9% | | |

APPLICABILITY: The working range is 0.05 to 1.5 mg/m³ for a 90-L air sample. Evaporation of isooctane from the bubbler necessitates refilling the bubbler frequently.

INTERFERENCES: None identified.

OTHER METHODS: This method combines and replaces S275 [2] and S290 [2]. Lindane has also been sampled with a filter-solid sorbent train [3].

REAGENTS:

1. Aldrin, reagent grade.*
2. Lindane, reagent grade.*
3. Isooctane, chromatographic grade.
NOTE: Needed for field use in sample collection.
4. Benzene, reagent grade.*
5. Calibration stock solution, 10 mg/mL. Dissolve 0.1 g accurately weighed Aldrin or Lindane in 1:5 (v:v) benzene:isooctane; dilute to 10 mL. Stable at least one week.
6. Hydrogen, prepurified.
7. Nitrogen, purified.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass fiber filter, organic binder-free (e.g., Gelman Type A/E), 37-mm, held without backup pad in a two-piece polystyrene cassette filter holder connected in series with a midjet bubbler containing 15 mL of the collection medium.
NOTE: a. Do not use filter holders made of Tenite.
b. Place glass tube, 5 cm x 6-mm ID, plugged with glass wool between the exit cover of the bubbler and the inlet of the personal sampling pump to avoid splashover or solvent condensation.
2. Personal sampling pump, 0.2 to 1 L/min, with flexible connecting tubing.
3. Vial, scintillation, with PTFE-lined cap, graduated at 15 mL.
4. Gas chromatograph with electrolytic conductivity detector, quartz reduction furnace, in-line vent upstream of furnace, integrator and column (page 5502-1).
5. Syringes, 5-, 10- and 25- μ L, for making standard solutions and GC injections.
6. Volumetric flasks.
7. Pipets, with pipet bulb.
8. Tweezers.

SPECIAL PRECAUTIONS: Benzene is a suspected human carcinogen; work with it only in a hood.

Aldrin and Lindane are toxic when absorbed through the skin [4].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Add 15 mL isooctane to the bubbler.
3. Sample at 0.5 L/min for 1 to 6 hrs (30 to 180 L).
NOTE: Check liquid level in the bubbler every 15 min. Maintain between 10 and 15 mL isooctane in the bubbler throughout the sampling period with a volume at the end of sampling of about 10 mL.
4. Remove the bubbler stem and tap it gently against the inside wall of the bubbler to transfer solvent from the stem to the bubbler.
5. Transfer contents of the bubbler to a vial. Rinse the bubbler with 2 mL isooctane. Add the rinse to the vial.
6. Transfer the glass fiber filter to the same vial using tweezers.
7. Cap the vial and pack carefully for shipment.

SAMPLE PREPARATION:

8. Adjust the volume of solution in the vial to 15 mL.

CALIBRATION AND QUALITY CONTROL:

9. Calibrate daily with at least five solutions covering the range 3 to 135 µg Aldrin or Lindane per sample.
 - a. Add calibration stock solution with a microliter syringe to 15 mL isooctane in a vial.
 - b. Analyze the working standards together with samples and blanks (steps 10 through 13).
 - c. Prepare calibration graph (peak area vs. µg Aldrin or Lindane). Analyze three additional quality control blind spikes and three analyst spikes to ensure that the calibration graph is in control.

MEASUREMENT:

10. Set gas chromatograph to manufacturer's specifications and to conditions given on page 5502-1.
11. Mix the contents of the scintillation vial thoroughly.
12. Inject 15 µL sample aliquot using solvent flush technique or autosampler.
NOTE: Open vent valve for 20 sec, beginning with time of injection, to prevent the solvent peak from entering the furnace.
13. Measure peak area.

CALCULATIONS:

14. Determine the mass, µg, of Aldrin or Lindane found in the sample (filter plus bubbler), W, and average media blank (filter plus bubbler), B, from the measured peak areas and the calibration graph.
15. Calculate the concentration, C (mg/m³), of Aldrin or Lindane in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Methods S275 (Aldrin) and S290 (Lindane) were issued on February 27, 1976, and March 26, 1976, respectively [3]. The substances used to generate test atmospheres at 25 °C and 760 mm Hg in dry air were Aldrite emulsifiable concentrate (64% Aldrin) and Ortho-Lindane Borer and Leaf Miner Spray [2]. Collection efficiencies and analytical method recoveries were 1.00 for both substances in the range 22 to 90 µg per sample. Sample filters extracted in isooctane immediately and stored one week at ambient conditions gave recoveries of 103% and 102%, respectively. Overall precision, \hat{S}_{τ} , was 0.092 for Aldrin and 0.086 for Lindane. No significant bias was found for either substance.

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S275 and S290, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
- [2] NIOSH Manual of Analytical Methods, V. 3, S275 and S290, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [3] Hill, R.H. and J.E. Arnold. Arch. Environ. Contam. Toxicol., **8**, 621-628 (1979).
- [4] Criteria for a Recommended Standard...Occupational Exposure During the Manufacture and Formulation of Pesticides, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-174 (1978).

METHOD REVISED BY: Gangadhar Choudhary, Ph.D., NIOSH/DPSE; S275 and S290 originally validated under NIOSH Contract CDC-99-74-45.