$C_{12}H_8OCl_6$ MW: 380.93 CAS: 72-20-8 RTECS: IO1575000

METHOD: 5519, Issue 2 **EVALUATION: FULL** **Issue 1: 15 May 1989**
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OSHA : 0.1 mg/m^3 (skin)
NIOSH: 0.1 mg/m^3 (skin); Group I Pesticide
ACGIH: 0.1 mg/m^3 (skin)

PROPERTIES: crystals; MP 200 °C; decomposes @ 245 °C; VP 0.27 x 10^{-7} kPa (2×10^{-7} mm Hg) @ 25 °C

SYNONYMS: Mendrin; Nendrin; Hexadrin

SAMPLING

SAMPLER: FILTER + SOLID SORBENT TUBE (0.8 µm cellulose ester membrane + Chromosorb 102, 100/50 mg)

FLOW RATE: 0.5 to 1 L/min

VOL-MIN: 12 L @ 0.1 mg/m^3
-MAX: 400 L

SHIPMENT: routine

SAMPLE STABILITY: at least 1 week @ 25 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.06 to 0.31 mg/m^3 [1,2] (120-L samples)

BIAS: - 0.7%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.071 [2]

ACCURACY: ± 13.9%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, ^{63}Ni ECD

ANALYTE: endrin

EXTRACTION: 5 mL toluene, stand 15 min

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTOR: 175 °C
-DETECTOR: 280 °C
-COLUMN: 160 °C

CARRIER GAS: 95% argon/5% methane @ 60 mL/min

COLUMN: 2 m x 4-mm ID glass, packed with 3% OV-1 on 100/120 Chromosorb Q

CALIBRATION: standard solutions of endrin in toluene

RANGE: 1.2 to 36 µg per sample [2]

ESTIMATED LOD: 0.02 µg per sample [1]

PRECISION ($\bar{S}_r$): 0.016 @ 1.2 to 24.5 µg per sample [1]

APPLICABILITY: The working range is 0.01 to 0.33 mg/m^3 for a 120-L air sample. Smaller concentrations can be determined if desorption efficiency is satisfactory. The use of a capillary column, e.g., DB-1, may improve resolution and sensitiv ity.

INTERFERENCES: None identified.

OTHER METHODS: This is a modification of the method of Hill and Arnold [3] and it replaces NIOSH method S284 [2].

REAGENTS:

1. Toluene, ACS reagent grade or better.
2. Hexane, ACS reagent grade or better.
3. Xylene, ACS reagent grade or better.
4. Endrin.*
5. Calibration stock solution, 3 mg/mL. Dissolve 30 mg endrin in 1 mL xylene and dilute to 10 mL with hexane.
6. 95% Argon/5% methane mixture, purified.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 37-mm, 0.8-µm pore size MCEF filter supported by a stainless steel screen in cassette filter holder followed by a 7 cm x 8-mm OD x 6-mm ID, glass tube, flame-sealed with plastic caps, containing 20/40 mesh Chromosorb 102 (front = 100 mg; back = 50 mg), separated by 3-mm silanized glass wool plug. Pressure drop across the tube at 1 L/min must not exceed 3.4 kPa. Tubes commercially available (SKC #226-49-20-102 or equivalent).
2. Personal sampling pump, 0.5 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, electron capture detector, integrator and column (page 5519-1).
4. Vials, glass, scintillation, 20-mL, PTFE-lined caps.
5. Syringe, 10-µL, readable to 0.1-µL.
6. Volumetric flasks, 10-mL.
7. File, triangular.
8. Pipets, 5- and 10-mL.
9. Tweezers.

SPECIAL PRECAUTIONS: Endrin is absorbed through the skin. Gloves and safety glasses must be used to avoid direct contact with this compound. Handle all chemicals and organic solvents in the laboratory hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the tube immediately before sampling. Connect the Chromosorb tube to the cassette with a short piece of tubing. Connect the tube to the pump with backup section nearest the sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.5 and 1 L/min for a total sample size of 12 to 400 L.
4. Remove the cassette from the sampling train, carefully remove the filter from the cassette with tweezers and place it in a clean vial. Reassemble the cassette and plug the inlet and outlet.
5. Score the Chromosorb tube in front of the front section and break the tube at score line. Transfer the front (larger) section of sorbent and the glass wool plug to the scintillation vial containing the filter.
6. Cap the ends of the sampling tube containing the backup section and ship along with the sample vial.
7. In a separate package, ship bulk sample of the suspected material in a vial.

SAMPLE PREPARATION:

8. Transfer the backup sorbent section and associated glass wool plugs to a clean vial.
9. Add 5.0 mL toluene to each vial (filter + front sorbent section in one vial and back sorbent section in another vial). Cap each vial. Allow to stand, with occasional swirling, for 15 min.

10. Wash the filter cassette parts as follows: Pipet 10.0 mL hexane into a clean vial and mark the level. Remove the hexane and allow to dry. Place the bottom part-cassette on the open, marked vial. Invert the top part-cassette and place it on the bottom part-cassette. Hold the metal screen over the set-up with clean tweezers. Rinse the screen with 10.0 mL hexane, allowing the rinse to drain from top cassette through bottom cassette to the marked vial. Remove any remaining rinse from the cassette parts with clean disposable pipet and transfer to the marked vial. Dilute with hexane to the marked levels. Cap immediately.
NOTE: Analysis should be done within 24 hours of desorption.

CALIBRATION AND QUALITY CONTROL:

11. Calibrate daily with at least six working standards covering the range of samples.
    a. Add known amounts of calibration stock solution to toluene in 10-mL volumetric flasks and dilute to the mark. Use serial dilution as needed to obtain endrin concentrations in the range 0.004 to 7 µg/mL.
    b. Analyze (steps 14 through 16) the working standards and blank in duplicate.
    c. Prepare a calibration graph (peak area vs. µg endrin); analyze two additional check standards for each ten sample injections.
12. Determine the analytical method recovery at least once for each lot of filter and Chromosorb 102 used. Prepare four samplers at each of five levels plus three media blanks.
    a. Place a MCEF filter and 100 mg of Chromosorb 102 in a vial.
    b. Inject calibration stock solution onto the combined filter and Chromosorb 102 in the container with a microliter syringe. Include blank samples.
    c. Cap the vial and let stand overnight.
    d. Analyze in duplicate; prepare graph of recovery vs. µg endrin.
13. Check recovery at two levels for each sample set in duplicate. Repeat recovery graph determination if checks do not agree to within 5% of recovery graph.

MEASUREMENT:

14. Set gas chromatograph to conditions given on page 5519-1.
15. Inject 5 µL sample aliquot using solvent flush technique. Make duplicate injections of sample and standards.
    NOTE: Under these conditions, t_r for endrin is ca. 5 min.
16. Measure peak areas.

CALCULATIONS:

17. Determine the mass, µg (corrected for recovery) of endrin found on the sampler (filter plus front section of Chromosorb 102), W_1, back of section, W_2, and the rinse from screen and cassette, F, and in the average media blank (filter plus front tube section, B_1, and back tube section, B_2.
    NOTE: If $W_2 > W_1 + F/10$, report breakthrough and possible sample loss.
18. Calculate the concentration of endrin, C (mg/m^3), in the air volume sampled, V (L):

$$C = \frac{(W_1 + W_2 + F - B_1 - B_2)}{V}, \ mg/m^3.$$

EVALUATION OF METHOD:

Method S284 was validated on July 8, 1979 [1,2,4]. The substance used to dynamically generate test atmospheres at 25 °C and 760 mm Hg was: 1.6 EC, Velsicol Chemical Corporation. Collection efficiencies were close to 1.00 in the range 28 to 33 µg per sample. The analytical recoveries from

sorbent tubes (range: 1.2 to 6.1 µg per sample) and from the filter sampler (range: 6.1 to 24.5 µg per sample) were 99% with a combined precision of 0.016. No significant breakthrough was observed after 240 min of sampling an atmosphere containing 0.257 mg/m ^{3} endrin at a flow rate of approximately 1 L/min. Samples stored one week at room temperature and extracted with toluene gave recoveries of 96 to 100%. Overall precision ($\hat{S}_{rT}$) was 0.07. No significant bias was found.

REFERENCES:

[1] NIOSH Backup Data Report S284 (July 8, 1979).
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 6, S284, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-125 (1980).
[3] Hill, R. H. and J. E. Arnold., "A Personal Air Sampler for Pesticides", Arch. Environ. Contam. Toxicol., 8, 621-628 (1979).
[4] NIOSH Research Report-Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

METHOD REVISED BY:

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