C_5Cl_6 MW: 272.77 CAS: 77-47-4 RTECS: GY1225000

METHOD: 2518, Issue 2 **EVALUATION: FULL** **Issue 1: 15 May 1985**
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OSHA : no PEL
NIOSH: 0.01 ppm; Group II Pesticide
ACGIH: 0.01 ppm
(1 ppm = 11.15 mg/m^3 @ NTP)

PROPERTIES: liquid; MP 10 °C; BP 239 °C;
d 1.714 g/mL @ 15.5 °C; VP 13 Pa
(0.1 mm Hg; 132 ppm) @ 25 °C

SYNONYMS: perchlorocyclopentadiene.

SAMPLING

SAMPLER: SOLID SORBENT TUBE
(2 tubes, Porapak T, 75 mg and 25 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 0.25 L @ 0.01 ppm
-MAX: 90 L

SHIPMENT: separate tubes, seal, ship @ 25 °C; store @ 0 °C in the dark

SAMPLE STABILITY: ≥7 days @ 25 °C; ≥28 days @ 0 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 13 to 873 µg/m^3 [1]
(3-L samples)

BIAS: - 0.3%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.082 [2]

ACCURACY: ± 16.4%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, ^{63}Ni ECD

ANALYTE: hexachloro-1,3-cyclopentadiene

DESORPTION: 1 mL hexane; ultrasonic bath 1 h

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 150 °C
-DETECTOR: 250 °C
-COLUMN: 135 °C

CARRIER GAS: 5% CH_4/95% Ar, 30 mL/min

COLUMN: glass, 2 m x 4-mm ID; 3% OV-1 on 100/120 Gas Chrom Q

CALIBRATION: hexachloro-1,3-cyclopentadiene in hexane

RANGE: 25 to 140 ng per sample

ESTIMATED LOD: 5 ng per sample

PRECISION ($\hat{S}_r$): 0.030 [1]

APPLICABILITY: The working range is 0.005 to 0.2 ppm (5 to 2400 mg/m^3) for a 5-L air sample. Area samples from a municipal wastewater treatment plant were analyzed by a variation of this method [2].

INTERFERENCES: None identified.

OTHER METHODS: This revises P&CAM 308 [3] which was evaluated with a reference method which involved sampling with impingers of hexane and gas chromatography with electron capture detection [1].

REAGENTS:

1. Porapak T (optional if commercial tubes are used), 80/100 mesh (Waters Associates, Inc.), cleaned as follows:
   a. Soxhlet extract 4 h with 80/20 acetone/methanol (v/v).
   b. Soxhlet extract 4 h with hexane.
   c. Dry in vacuo 12 h at 50 to 75 °C.
2. Hexane, spectral quality.*
3. Hexachloro-1,3-cyclopentadiene, 98% or purer.
4. Acetone, spectral quality.*
5. Methanol, spectral quality.*
6. Stock solution, 20 mg/mL. Dissolve 2.00 g hexachloro-1,3-cyclopentadiene in hexane to make 100 mL solution. Store at 0 °C, protect from light and discard after 60 days.
7. Calibration stock solution, 10 µg/mL. Dilute 50 µL stock solution with hexane to make 100 mL solution. Store at 0 °C, protect from light and discard after 14 days.
8. Mixture of 5% methane and 95% argon.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: two glass tubes connected in series with 1.8-cm piece of plastic tubing; each tube, 5 cm long, 6-mm OD, 4-mm ID, contains one section of 80/100 mesh Porapak T held in place with two plugs of silylated glass wool (front tube = 75 mg; back tube = 25 mg) with plastic caps. Pressure drop <2.3 kPa (<17 mm Hg) at flow rate of 0.2 L/min. Samplers are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. PTFE tape.
4. Gas chromatograph, ^{63}Ni ECD, integrator and column (page 2518-1).
5. Volumetric flasks, 10- and 100-mL.
6. Vials, 1-mL, with PTFE-lined caps.
7. Syringes, 10-µL, readable to 0.1 µL.
8. Syringes, 100-µL, readable to 1 µL.
9. Syringes, 1-mL, readable to 10 µL.
10. Pipet, 1-mL.
11. Ultrasonic bath.

SPECIAL PRECAUTIONS: Hexane, acetone and methanol are flammable. Hexachloro-1,3-cyclopentadiene is highly toxic and can be absorbed through the skin. Wear gloves, avoid inhalation of vapors and protect solvents from sparks and flames.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size of 0.25 to 90 L. Do not sample for longer than 8 h.
4. Separate the two tubes of the sampler. Cap the ends of each tube with PTFE tape and plastic caps. Pack securely for shipment.

SAMPLE PREPARATION:

NOTE: If the tubes have been refrigerated, allow the tubes to stand at room temperature before removal of the caps and tape to prevent condensation of water on the sorbent.

5. Place the front and back sorbent sections with their respective glass wool plugs, in separate vials.
6. Add 1.0 mL hexane to each vial. Attach cap to each vial.
7. Allow to stand 60 min in an ultrasonic bath.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 5 to 140 ng hexachloro-1,3-cyclopentadiene per sample.

a. Add known amounts (5 to 140 µL) of calibration stock 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 (peak area or height vs. ng hexachloro-1,3-cyclopentadiene).

9. Determine desorption efficiency (DE) at least once for each lot of Porapak T used for sampling in the range 25 to 12,500 ng hexachloro-1,3-cyclopentadiene per sample. Prepare three tubes at each of five levels plus three media blanks.
   a. Prepare five DE standard solutions in the range 5 to 2500 µg/mL by diluting aliquots of 20 mg/mL stock solution with hexane.
   b. Inject 5 µL of a DE standard solution directly into a media blank front sorbent section with a microliter syringe.
   c. Cap the tube. Allow to stand 12 hrs at room temperature.
   d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
   e. Prepare a graph of DE vs. ng hexachloro-1,3-cyclopentadiene 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 2518-1. Prior to analyzing samples, condition the column by making three 5-µL injections of a 1-µg/mL solution of hexachloro-1,3-cyclopentadiene in hexane. Inject sample aliquot manually using solvent flush technique or with autosampler. t_r = 7 min for these conditions.
    NOTE: If peak area is above the useful range of the working standards, dilute with hexane, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area or height.

CALCULATIONS:

13. Determine the mass, ng (corrected for DE) of hexachloro-1,3-cyclopentadiene found in the sample front (W_f) and back (W_b) sorbent tubes, and in the average media blank front (B_f) and back (B_b) sorbent tubes.
    NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C, of hexachloro-1,3-cyclopentadiene in the air volume sampled, V (L):

$$C \frac{(W_f + W_b - B_f - B_b)}{V}, \mu g/m^3.$$

EVALUATION OF METHOD:

A variation of this method employed single 100- or 50-mg sections of Porapak T per sampler. This variation was tested with controlled atmospheres and verified with impingers containing hexane and with gas chromatographic analysis for 13.1 to 873 µg/m^3 [1]. Overall precision ($\hat{S}_{rT}$) was 0.082 (18 samples, pooled) for 2.9-L samples; the temperature of each atmosphere was 25 to 28 °C, and the relative humidity was at least 90%. Mean recovery for three concentration levels was 99.7%. The average concentration found at each level was not significantly different from the corresponding reference concentration at 95% confidence level. Breakthrough (1 mg/m^3, 0.2 L/min, 27 °C, RH 90%) = 55 L for a 50-mg section of Porapak T. Values of DE from 50 mg of Porapak T with 1 mL of hexane were 1.01, 0.99, and 1.01 for 24.5, 352, and 3670 ng per sample, respectively; $\bar{S}_r$ = 0.030 (18 samples, pooled). Hexachloro-1,3-cyclopentadiene (about 30 ng per sample) was stable for 28 days inside each tube which contained 100- and 50-mg sections of Porapak T (samples were stored at room temperature for the first seven days and at 0 °C for the next 21 days). However, hexachloro-1,3-cyclopentadiene did

migrate from front sections of sorbent to backup sections. Area samples from a municipal wastewater treatment plant were collected and analyzed by a variation of this method [2]. Differences in the method included sampling with 150 mg of 80/100 Chromosorb 102 and desorption with petroleum ether. Concentrations of hexachloro-1,3-cyclopentadiene ranged from 0.03 to 39 µg/m ^{3}.

REFERENCES:

[1] Dillon, H. K. Research Report for Hexachlorocyclopentadiene, Southern Research Institute, Birmingham, AL, NIOSH Contract 210-78-0012 (1980).
[2] Elia, V. J., C. S. Clark, V. A. Majeti, P. S. Gartside, T. MacDonald, N. Richdale, C. R. Meyer, G. L. Van Meer and K. Hunninen. Environ. Res., 32, 360-371 (1983).
[3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 5, P&CAM 308, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).

METHOD REVISED BY:

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