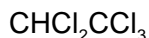


PENTACHLOROETHANE

2517



MW: 202.30

CAS: 76-01-7

RTECS: KI6300000

METHOD: 2517, Issue 2

EVALUATION: FULL

Issue 1: 15 May 1985

Issue 2: 15 August 1994

OSHA : no standard
 NIOSH: handle with caution
 ACGIH: no standard
 (1 ppm = 8.27 mg/m³ @ NTP)

PROPERTIES: liquid; d 1.671 g/mL @ 25 °C; BP 162 °C;
 VP 450 Pa (3.4 mm Hg; 4500 ppm) @ 20 °C

SYNONYMS: Pentalin; ethane pentachloride.

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (Porapak R, 70 mg/35 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, ⁶³ Ni ECD
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	pentachloroethane
VOL-MIN:	1 L @ 0.1 mg/m ³	DESORPTION:	2 mL hexane; 30 min ultrasonic
-MAX:	10 L	TEMPERATURE-INJECTION:	240 °C
SHIPMENT:	routine	-DETECTOR:	250 °C
SAMPLE STABILITY:	stable ≥ 7 days @ 25 °C; ≥ 21 days @ 0 °C in the dark	-COLUMN:	70 °C
BLANKS:	2 to 10 field blanks per set	INJECTION VOLUME:	5 µL
		CARRIER GAS:	5% methane, 95% argon; 30 mL/min
		DETECTOR PURGE GAS:	5% methane, 95% argon if required; 80 mL/min
		COLUMN:	nickel, 2-mm ID x 2 m, packed with 3% OV-17 on 100/120 mesh Chromosorb WHP
		CALIBRATION:	standard solutions of pentachloroethane in hexane
		RANGE:	0.04 to 40 µg per sample
		ESTIMATED LOD:	0.004 µg per sample [2]
		PRECISION (S_p):	0.019 [1]
ACCURACY			
RANGE STUDIED:	0.014 to 5.37 mg/m ³ [1] (3-L samples)		
BIAS:	not significant [1]		
OVERALL PRECISION (S_{r,T}):	0.075 [1]		
ACCURACY:	± 15%		

APPLICABILITY: The working range is 0.01 to 20 mg/m³ (0.001 to 2.4 ppm) for a 5-L air sample.

INTERFERENCES: 1,2,2,3-Tetrachloropropane interferes with the analyte GC peak but is not likely to be present in most samples. Large concentrations of 1,1,2,2-tetrachloroethane may obscure the analyte GC peak.

OTHER METHODS: This revises P&CAM 335 [2].

REAGENTS:

1. Hexane, distilled in glass.*
2. Pentachloroethane, 96% pure.*
3. Mixture of 5% methane, 95% argon.
4. Calibration stock solution, 2 mg/mL. Dissolve 0.2 g pentachloroethane in hexane to make 100 mL solution. Stable indefinitely at room temperature.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: borosilicate tubes, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of 50/80 mesh Porapak R (front = 70 mg; back = 35 mg) separated by a urethane foam plug. A glass wool plug held in place with a metal spring precedes the front section and a urethane foam plug follows the back section. Pressure drop across the tube at 0.2 L/min airflow must be less than 1.2 kPa (5 inches H₂O). Tubes are commercially available, e.g., SKC, Inc. ST226-59-04.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, ⁶³Ni ECD, integrator, and column (page 2517-1).
4. Vials, 2-mL, with PTFE-lined crimp caps.
5. Microliter syringes, 10- μ L and convenient sizes for making dilutions.
6. Ultrasonic bath.
7. Pipets, 2-mL.
8. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Hexane is flammable (flash point = -21 °C) and toxic; all work with it must be done in a hood.

Pentachloroethane causes CNS effects, with possible liver and kidney effects [3]. Technical grade pentachloroethane (containing 4.2% hexachloroethane, a known carcinogen in mice) was carcinogenic for B6C3F₁ mice, causing hepatocellular carcinomas in males and females, and adenomas in females [4].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. 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 1 to 10 L.
4. Cap the samplers with PTFE tape and plastic caps. Pack securely for shipment.

SAMPLE PREPARATION:

5. Refrigerate the samples upon receipt.
6. Place the front and back sorbent sections of the sampler tube in separate vials. Add the glass wool plug to the front sorbent section and the foam plugs to the back sorbent section.
7. Add 2.0 mL hexane to each vial. Attach crimp cap to each vial.
8. Allow to stand 30 min in an ultrasonic bath.

CALIBRATION AND QUALITY CONTROL:

9. Calibrate daily with at least six working standards over the range 0.004 to 40 µg pentachloroethane per sample (0.002 to 20 µg/mL).
 - a. Add known amounts of calibration stock solution, or serial dilutions thereof, to hexane in 10-mL volumetric flasks and dilute to the mark.
NOTE: The working standards are stable indefinitely at room temperature if kept in airtight containers.
 - b. Analyze together with samples and blanks (steps 12 and 13).
 - c. Prepare calibration graph (peak area or height vs. µg pentachloroethane).
10. Determine desorption efficiency (DE) at least once for each lot of Porapak R used for sampling in the calibration range (step 9). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount (1 to 20 µL) of calibration stock solution, or serial dilutions thereof, directly onto front sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 6 through 8) and analyze together with working standards (steps 12 and 13).
 - e. Prepare a graph of DE vs. µg pentachloroethane recovered.
11. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

12. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 2517-1. Inject sample aliquot manually using solvent flush technique or with autosampler. $t_r = 3.5$ min at these conditions.
NOTE: If peak area is above the linear range of the working standards, dilute with hexane, reanalyze, and apply the appropriate dilution factor in calculations.
13. Measure peak area or height.

CALCULATIONS:

14. Determine the mass, µg (corrected for DE) of pentachloroethane found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
15. Calculate concentration, C, of pentachloroethane in the air volume sampled, V (L):

$$C = \frac{W_f + W_b - B_f - B_b}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

P&CAM 335 was issued on August 29, 1980, and validated over the range 0.014 to 5.4 mg/m³ with samples from dynamically generated test atmospheres [1]. A gas chromatograph with gas sampling loop was used to monitor pentachloroethane concentration of the atmospheres. To study sample stability, four sets of six 3-L samples of 0.014 mg/m³ pentachloroethane at 80% relative humidity were obtained. The samples were stored at room temperature for the first 7 days, and at 0 °C for the remainder of the period. These sets were analyzed at day 1, 7, 14, and 28. The mean recoveries were, respectively, 104, 105, 101.4, and 97.0%. The respective relative standard deviations were 7.4, 6.2, 9.0, and 16.7%. The breakthrough volume for the 70-mg bed of Porapak R was 14 L, when an atmosphere at 40 °C containing 126 mg/m³ pentachloroethane and >80% relative humidity was sampled at 0.2 L/min.

REFERENCES:

- [1] Development of Air Sampling and Analytical Methods for Toxic Chlorinated Organic Compounds - Research Report for Pentachloroethane, Contract No. 210-78-0012, Southern Research Institute for the Department of Health and Human Services, NIOSH (August, 1980).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 6, P&CAM 335, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-125 (1980).
- [3] NIOSH Current Intelligence Bulletin 27. Chloroethanes: Review of Toxicity, U.S. Dept. of Health, Education & Welfare (NIOSH), Publ. 78-181 (1978).
- [4] Carcinogenesis Bioassay of Pentachloroethane (CAS #76-01-7) in F344/N Rats and B6C3F₁ Mice (Gavage Study), Technical Report Series #232, NTP, U.S. Department of Health and Human Services, Publ. (NIOSH) 83-1788 (April, 1983) and the National Toxicology Program, Box 12233, Research Triangle Park, NC 27709.

METHOD REVISED BY:

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