

1, 2-ジクロロプロパン分析法

構造式 CH3CHClCH2Cl

分子量: 112.99

CASNo: 78-87-5

許容濃度等: ACGIH 10ppm (TWA)

物性等

融点 -100.4℃

沸点 96.4℃

蒸気圧 53.3mmHg (25℃)

分配係数 log Pw = 1.98

別名 塩化プロピレン、二塩化プロピレン

サンプリング

分析

NIOSH 分析法 [別添参照]

精度

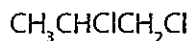
適用;

妨害;

参考文献; NIOSH method No.1013

PROPYLENE DICHLORIDE

1013



MW: 112.99

CAS: 78-87-5

RTECS: TX9625000

METHOD: 1013, Issue 2

EVALUATION: FULL

Issue 1: 15 May 1985

Issue 2: 15 August 1994

OSHA: 75 ppm
NIOSH: lowest feasible; carcinogen; Group I pesticide
ACGIH: 75 ppm; STEL 110 ppm; carcinogen
(1 ppm = 4.62 mg/m³ @ NTP)

PROPERTIES: liquid; d 1.159 g/mL @ 25 °C; BP 95 °C; MP -70 °C; VP 5.3 kPa (40 mm Hg; 5.2% v/v) @ 20 °C; explosive range 3.4 to 14.5% v/v in air

SYNONYMS: 1,2-dichloropropane

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (petroleum charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, ELECTROLYTIC CONDUCTIVITY DETECTOR (HALL)
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	propylene dichloride
VOL-MIN:	0.1 L @ 75 ppm	DESORPTION:	1 mL 15% (v/v) acetone/cyclohexane; ultrasonic bath, 30 min
-MAX:	3.5 L	INJECTION VOLUME:	5 µL
SAMPLE STABILITY:	at least 26 days @ 25 °C in the dark [1]	TEMPERATURE-INJECTION:	150 °C
BLANKS:	2 to 10 field blanks per set	-COLUMN:	50 °C
ACCURACY		HALL DETECTOR FURNACE:	840 °C
RANGE STUDIED:	0.12 to 128 mg/m ³ [1] (3-L samples)	-ELECTROLYTE:	0.9 mL/min
BIAS:	3%	-HYDROGEN:	50 mL/min
OVERALL PRECISION ($\bar{S}_r$):	0.064 [1]	CARRIER GAS:	N ₂ , 25 mL/min
ACCURACY:	±15.5%	COLUMN:	nickel, 3 m × 2-mm ID, 3% Carbowax 1500 on 60/80 mesh Chromosorb WHP
		CALIBRATION:	solutions in acetone/cyclohexane
		RANGE:	0.5 to 400 µg per sample [2]
		ESTIMATED LOD:	0.1 µg per sample [2]
		PRECISION ($\bar{S}_r$):	0.031 @ 0.5 to 520 µg per sample [1]

APPLICABILITY: The working range is 0.05 to 130 ppm (0.25 to 600 mg/m³) for a 2-L air sample at about 90% RH.

INTERFERENCES: Propylene dichloride cannot be resolved from 1,2-dichloroethane with a Carbowax packed column. They were resolved with a Carbowax capillary column.

OTHER METHODS: This revises P&CAM 321 [2].

REAGENTS:

1. Acetone, distilled in glass.*
2. Cyclohexane, distilled in glass.*
3. Eluent: 15% (v/v) acetone in cyclohexane.
4. Propylene dichloride, reagent grade.*
5. Nitrogen, high purity, for GC carrier gas.
6. Hydrogen, prepurified, for Hall detector.
7. Electrolyte solution for Hall detector:
2-propanol and distilled, deionized water, 50% (v/v).
8. Calibration stock solution, 34.7 mg/mL. Dilute 3.00 mL (3.47 g) 1,2-dichloropropane to 100 mL with eluent. Stable indefinitely at room temperature in airtight container.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of activated petroleum-based charcoal (front = 100 mg; back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 0.2 L/min airflow must be less than 3.4 kPa. Tubes are commercially available (SKC No. 226-65 or equivalent).
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. PTFE tape.
4. Gas chromatograph with Hall electrolytic conductivity detector and column (page 1013-1).
5. Vials, 1-mL, with PTFE-lined silicone rubber septa and serum caps.[†]
6. Syringes, 10-mL and other convenient sizes for making dilutions.
7. Pipets, 1-mL and other convenient sizes for making dilutions.[†]
8. Volumetric flasks, 10- and 100-mL.[†]
9. Ultrasonic bath.

[†]Clean thoroughly and rinse with, in order, 50% (v/v) HNO₃, distilled water, acetone, and cyclohexane.

SPECIAL PRECAUTIONS: Propylene dichloride vapor is a narcotic and an eye irritant [3]. Acetone and cyclohexane are extremely flammable. Work with these in a fume hood.

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 0.1 to 3.5 L.
4. Cap the samplers with PTFE tape. Pack securely for shipment. Store samplers in the dark.
5. Refrigerate the samplers in an airtight container upon receipt.

SAMPLE PREPARATION:

6. Bring the samplers to room temperature before uncapping to prevent moisture condensation on the sorbent.
7. Place the front and back sorbent sections of the sampler tube in separate vials. Place the glass wool plug with the front sorbent section and the foam plugs with the back sorbent section.

8. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial immediately.
9. Allow to stand 30 min in an ultrasonic bath.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate daily with at least six working standards over the range 0.1 to 400 µg propylene dichloride per sample.
 - a. Add known amounts of calibration stock solution, or a serial dilution thereof, to eluent in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 13 and 14).
 - c. Prepare calibration graph (peak height vs. µg propylene dichloride).
11. Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 10). 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 a serial dilution thereof, directly onto front sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 6 through 9) and analyze together with working standards (steps 13 and 14).
 - e. Prepare a graph of DE vs. µg propylene dichloride recovered.
12. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

13. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1013-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
NOTE: If peak height is above the linear range of the working standards, dilute with eluent, reanalyze, and apply the appropriate dilution factor in calculations.
14. Measure peak height.

CALCULATIONS:

15. Determine the mass, µg (corrected for DE), of propylene dichloride 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.
NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
16. Calculate concentration, C, of propylene dichloride 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:

Method P&CAM 321 was issued on June 15, 1980 [2] and was validated over the range 0.37 to 384 µg propylene dichloride per charcoal tube [1]. The $\bar{S}_r$ for replicate measurements from generated atmospheres was 0.064 for 17 samples. Recoveries ranged from 97% to 103%. The pooled relative standard deviation for the analysis of spiked samplers was 0.031. The breakthrough volume was found to be about 20 L with sampling rates of 0.07 to 0.17 L/min at 1,2-dichloropropane concentrations of 62 to 85 mg/m³ at RH >90% and ca. 6 L at concentrations of 738 to 865 mg/m³. Desorption efficiency averaged 0.95 in the range 0.5 to 520 µg 1,2-dichloropropane per sampler.

REFERENCES:

- [1] Research Report for 1,2-Dichloropropane, NIOSH Contract No. 210-78-0012, Southern Research Institute, Birmingham, AL (NIOSH, unpublished, 1979).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 6, P&CAM 321, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-125 (1980).
- [3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, Propylene dichloride, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.

METHOD REVISED BY:

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