

PROPYLENE OXIDE

1612

C_3H_6O

MW: 58.08

CAS: 75-56-9

RTECS: TZ2975000

METHOD: 1612, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 May 1985

Issue 2: 15 August 1994

OSHA : 100 ppm

NIOSH: lowest feasible (carcinogen);
Group I Pesticide

ACGIH: 20 ppm
(1 ppm = 2.37 mg/m³ @ NTP)

PROPERTIES: liquid; d 0.8287 g/mL @ 20 °C;
BP 34 °C; MP -104 °C;
VP 59 kPa (442 mm Hg; 58% v/v)
@ 20 °C; explosive range 2.1 to
37% (v/v) in air

SYNONYMS: 1,2-epoxypropane; methyloxirane

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	propylene oxide
VOL-MIN:	0.5 L @ 100 ppm	DESORPTION:	1 mL CS ₂ ; stand 30 min
-MAX:	5 L	INJECTION VOLUME:	5 µL
SHIPMENT:	refrigerated	TEMPERATURE-INJECTION:	190 °C
SAMPLE STABILITY:	not determined	-DETECTOR:	255 °C
FIELD BLANKS:	2 to 10 field blanks per set	-COLUMN:	40 °C, 6 min; 50 °C/min to 250 °C
ACCURACY		CARRIER GAS:	N ₂ , 1 mL/min; makeup 29 mL/min
RANGE STUDIED:	121 to 482 mg/m ³ [1] (5-L samples)	COLUMN:	30 m x 0.32-mm fused silica capillary column, 1.0-µm DB-5 [2]
BIAS:	- 1.8%	CALIBRATION:	propylene oxide in CS ₂
OVERALL PRECISION (\hat{S}_{rT}):	0.085 [1]	RANGE:	0.03 to 3 mg per sample [2]
ACCURACY:	± 11.9%	ESTIMATED LOD:	0.01 mg per sample
		PRECISION (\hat{S}_j):	0.029 @ 0.6 to 2.4 mg per sample [1]

APPLICABILITY: The working range is 8 to 295 ppm (20 to 700 mg/m³) for a 5-L air sample [1].

INTERFERENCES: None found.

OTHER METHODS: This revises method S75 [3].

REAGENTS:

1. Propylene oxide (PrO), reagent grade.*
2. Carbon disulfide (CS₂), chromatographic quality.*
3. Hexane, reagent grade.*
4. Calibration stock solution, 10 mg/mL, in carbon disulfide.
5. Desorption efficiency (DE) stock solution, 150 mg/mL, in hexane.
6. Nitrogen, purified.
7. Hydrogen, purified.
8. Air, compressed, filtered.

* 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 (600 °C) coconut shell 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 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Shipping container, with bagged refrigerant.
4. Gas chromatograph, FID, integrator, and column (page 1612-1).
5. Vials, 2-mL, PTFE-lined caps.
6. Syringes, 10- μ L, readable to 0.1 μ L, and other convenient sizes for preparing standards.
7. Volumetric flasks, 10-mL.
8. Pipet, 1-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and a dangerous fire and explosion hazard (flash point = -30 °C). Propylene oxide is a suspect carcinogen and is extremely flammable (flash point = -37 °C) [4]. Hexane is extremely flammable. Work with these only in a 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 0.5 to 5 L.
4. Cap the samplers and pack securely for shipment in an insulated container with bagged refrigerant. Refrigerate upon receipt.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL CS₂ to each vial. Attach cap to each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 0.01 to 3 mg propylene oxide (PrO) per sample.
 - a. Add known amounts of PrO or calibration stock solution to CS₂ 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 vs. mg PrO).
9. Determine desorption efficiency (DE) at least once for each lot of charcoal used for sampling in the calibration range (step 8). 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 DE stock solution directly onto front sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. mg PrO 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 1612-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
NOTE: If peak area is above the linear range of the working standards, dilute with CS₂, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of PrO 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.
14. Calculate concentration, C, of PrO in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - W_f - W_b) \cdot 10^3}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S75 was issued on February 14, 1975 [3], and validated over the range 121 to 482 mg/m³ at 24 °C and 766 mm Hg using a 5-L sample [1]. Overall precision, $\hat{S}_{r,T}$, was 0.085, with an average recovery of 94.4% at the OSHA standard level. Desorption efficiencies averaged 0.88, 0.95, and 0.96 at 0.6, 1.2, and 2.4 mg propylene oxide per sample, respectively. Breakthrough (5% on back section) occurred at 45 min when sampling an atmosphere of propylene oxide containing 484 mg/m³ in dry air at 0.185 L/min. At this time, the front section contained 4 mg propylene oxide. Breakthrough in humid air and storage stability were not determined.

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S75, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.
- [2] DataChem, Inc. NIOSH Sequences #6599, 6722, 6723 (unpublished, 1989).
- [3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S75, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [4] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, 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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