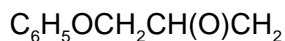


PHENYL GLYCIDYL ETHER

1619



MW: 150.18

CAS: 122-60-1

RTECS: TZ3675000

METHOD: 1619, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 August 1994

OSHA : 10 ppm  
 NIOSH: C 1 ppm/15 min; carcinogen  
 ACGIH: 1 ppm  
 (1 ppm = 6.14 mg/m<sup>3</sup> @ NTP)

PROPERTIES: liquid; d 1.109 g/mL @ 20 °C;  
 BP 245 °C; VP 1 Pa (0.01 mm Hg,  
 13 ppm) @ 25 °C

SYNONYMS: glycidyl phenyl ether; 1,2-epoxy-3-phenoxypropane; phenyl 2,3-epoxypropyl ether; (phenoxyethyl)oxirane; PGE

SAMPLING		MEASUREMENT	
<b>SAMPLER:</b>	SOLID SORBENT TUBE (coconut shell charcoal; 100 mg/50 mg)	<b>TECHNIQUE:</b>	GAS CHROMATOGRAPHY, FID
<b>FLOW RATE:</b>	0.01 to 1 L/min	<b>ANALYTE:</b>	phenyl glycidyl ether
<b>VOL-MIN:</b>	80 L @ 1 ppm	<b>DESORPTION:</b>	0.5 mL CS <sub>2</sub> , 30 min
<b>-MAX:</b>	150 L	<b>TEMPERATURE-INJECTION:</b>	230 °C
<b>SHIPMENT:</b>	routine (refrigerate at lab)	<b>-DETECTOR:</b>	265 °C
<b>SAMPLE STABILITY:</b>	not determined	<b>-COLUMN:</b>	90 °C
<b>BLANKS:</b>	2 to 10 field blanks per set	<b>INJECTION VOLUME:</b>	5 µL
<b>ACCURACY</b>		<b>CARRIER GAS:</b>	N <sub>2</sub> , 50 mL/min
<b>RANGE STUDIED:</b>	31 to 121 mg/m <sup>3</sup> [1] (50-L samples)	<b>COLUMN:</b>	stainless steel, 3.2-mm ID x 3 m, packed with 10% FFAP on 80/100 mesh Chromosorb W-AW DMCS
<b>BIAS:</b>	- 7.6%	<b>CALIBRATION:</b>	standard solutions of phenyl glycidyl ether in CS <sub>2</sub>
<b>OVERALL PRECISION (<math>\hat{S}_{rT}</math>):</b>	0.057 [1]	<b>RANGE:</b>	0.5 to 6 mg per sample
<b>ACCURACY:</b>	± 16.2%	<b>ESTIMATED LOD:</b>	not determined
		<b>PRECISION (<math>\hat{S}_r</math>):</b>	0.022 [1]

**APPLICABILITY:** The working range is 1 to 33 ppm (6 to 200 mg/m<sup>3</sup>) for an 80-L air sample. An appropriate capillary column may be used for better resolution and sensitivity. The sorbent's capacity for the analyte has not been determined under conditions of high relative humidity.

**INTERFERENCES:** None identified.

**OTHER METHODS:** This is Method S74 [2] in a revised format.

**REAGENTS:**

1. Carbon disulfide\* (CS<sub>2</sub>), chromatographic quality.
2. Phenyl glycidyl ether\*, reagent grade.
3. Nitrogen, purified.
4. Hydrogen, purified.
5. Air, compressed, filtered.

\* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler: borosilicate tubes, 7.0 cm long, 6-mm OD, 4-mm ID; flame-sealed ends with plastic caps, containing two sections of 20/40 mesh activated (600 °C) coconut charcoal (front = 100 mg; back = 50 mg) separated by a urethane foam plug. A silanized 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 1.0 L/min air flow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator, and column (page 1619-1).
4. Vials, 2-mL, with PTFE-lined crimp caps.
5. Microliter syringes, 10- $\mu$ L and convenient sizes for making dilutions.
6. Flasks, volumetric, 10-mL.
7. Pipets, 0.5-mL, with bulb.

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**SPECIAL PRECAUTIONS:** Both phenyl glycidyl ether and CS<sub>2</sub> are toxic [3]. In addition, CS<sub>2</sub> is a serious fire and explosion hazard (flash point = -30 °C). All work with these compounds must be done in a hood.

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**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 1 L/min for a total sample size of 80 to 150 L.
4. Cap the samplers. Pack securely for shipment.

**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 0.5 mL CS<sub>2</sub> to each vial. Cap 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 of 0.01 to 6 mg phenyl glycidyl ether per sample.
  - a. Add a known amount of phenyl glycidyl ether to CS<sub>2</sub> in 10-mL volumetric flask and dilute to the mark. Use serial dilutions as needed for smaller concentrations.
  - b. Analyze with samples and blanks (steps 11 and 12).

- c. Prepare calibration graph (phenyl glycidyl ether peak area vs. mg phenyl glycidyl ether per 0.5 mL).
9. Determine desorption efficiency (DE) at least once for each lot of charcoal used for sampling in the range of interest. 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 known amount (1 to 20  $\mu\text{L}$ ) of phenyl glycidyl ether or standard solution of phenyl glycidyl ether in  $\text{CS}_2$  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 with working standards (steps 11 and 12).
  - e. Prepare a graph of DE vs. mg phenyl glycidyl ether 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 1619-1. 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 an aliquot of the desorbed liquid with  $\text{CS}_2$ , reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

#### CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of phenyl glycidyl ether 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 phenyl glycidyl ether in the air volume sampled, V (L):

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

#### EVALUATION OF METHOD:

S74 was issued on February 14, 1975 [2] and validated over the range 31 to 121  $\text{mg/m}^3$  for 50-L air samples from dynamically generated test atmospheres [1]. The average recoveries ranged from 91.2 to 93.7%. The phenyl glycidyl ether concentrations were independently measured by means of a total hydrocarbon analyzer. Breakthrough was not observed after sampling 220 L from a test atmosphere containing 112  $\text{mg/m}^3$  of phenyl glycidyl ether. The average desorption efficiency was 98% over the range of 1.5 to 6 mg per sample. Sample stability was not determined; however, refrigeration of the samples upon receipt by the laboratory is recommended.

#### REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S74, 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] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S74, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

- [3] 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 #17-033-00337-8 from Superintendent of Documents, Washington, D.C. 20402.

**METHOD REVISED BY:**

R.A. Glaser, NIOSH/DPSE. Method S74 was originally validated under NIOSH Contract CDC-99-74-45.