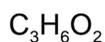


GLYCIDOL

1608



MW : 74.08

CAS: 556-52-5

RTECS: UB4375000

METHOD: 1608, Issue 2

EVALUATION: FULL

Issue 1: 15 May 1985

Issue 2: 15 August 1994

OSHA : 50 ppm
 NIOSH: 25 ppm
 ACGIH: 25 ppm
 (1 ppm = 3.03 mg/m³ @ NTP)

PROPERTIES: liquid; d 1.117 g/mL @ 20 °C; BP 167 °C
 (decomposes); MP - 45 °C; VP 0.12 kPa
 (0.9 mm Hg); 0.12% v/v @ 20 °C

SYNONYMS: 2,3-epoxy-1-propanol; 2-(hydroxymethyl)oxirane; oxiranemethanol

APPLICABILITY: The working range is 16 to 450 mg/m³ (5 to 150 ppm) for a 50-L air sample. During sampling, high humidity may greatly decrease the breakthrough volume.

INTERFERENCES: None identified.

OTHER METHODS: This is Method S70 [2] in a revised format.

REAGENTS:

1. Tetrahydrofuran, chromatographic quality.
2. Glycidol, reagent grade.*
3. Nitrogen, purified.
4. Hydrogen, prepurified.
5. Air, filtered.

* See Special Precautions

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, 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 1 L/min, with flexible connecting tubing.
3. Refrigerant, bagged ("blue ice" or equivalent).
4. Gas chromatograph, flame ionization detector, integrator and column (page 1608-1).
5. Vials, 1-mL, PTFE-lined caps.
6. Syringes, 10- μ L, and other convenient sizes for preparing standards, readable to 0.1 μ L.
7. Volumetric flasks, 10-mL.
8. Pipet, 0.5-mL.

SPECIAL PRECAUTIONS: Glycidol is irritating to the upper respiratory tract, skin, and mucous membranes and will attack some forms of plastics, rubber and coatings [3]. It is absorbed through skin [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 1 L/min for a total sample size of 5 to 100 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment at 4 °C.

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 tetrahydrofuran to each vial. Attach crimp 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 0.1 to 22 mg glycidol per sample.
 - a. Add known amounts of glycidol to tetrahydrofuran 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 glycidol).
9. Determine desorption efficiency (DE) at least once for each batch 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 of glycidol directly onto front sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight in refrigerator at 4 °C.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. mg glycidol recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure 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 1608-1. 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 tetrahydrofuran, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

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

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

EVALUATION OF METHOD:

Method S70 was issued on February 14, 1975 [2], and validated over the range 73 to 310 mg/m³ at 21 °C and 768 mm Hg using a 50-L sample [1]. Overall precision, \hat{S}_{IT} was 0.080 with an average recovery of 0.953, representing a non-significant bias. The concentration of glycidol was independently verified using a total hydrocarbon analyzer calibrated with gas mixtures of glycidol in air. Desorption efficiency was 0.92 in the range 3.8 to 15.0 mg per sample. Desorption efficiencies using carbon disulfide or methylene chloride were much lower. Samples appeared unstable at room temperature. The breakthrough volume (where the concentration of glycidol in the effluent from the 100-mg charcoal bed was 5% of that in the influent) was 152 L; this was determined by sampling dry air containing 300 mg/m³ glycidol at 0.93 L/min.

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S70, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as GPO Stock #017-033-00231-2 from the Superintendent of Documents, Washington, DC 20402.
- [2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 2, S70, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [3] Occupational Health Guidelines for Chemical Hazards, Glycidol, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from

- the Superintendent of Documents, Washington, DC 20402.
[4] The Merck Index, 9th ed., Merck & Co., Rahway, NJ (1976).

METHOD REVISED BY:

James E. Arnold, NIOSH/DPSE; S70 originally validated under NIOSH Contract CDC-99-74-45.