ISOPROPYL GLYCIDYL ETHER 1620

$C_6H_{12}O_2$ MW: 116.16 CAS: 4016-14-2 RTECS: TZ3500000

METHOD: 1620, Issue 1 **EVALUATION: PARTIAL** **Issue 1: 15 August 1994**

OSHA : 50 ppm
NIOSH: C 50 ppm (15 min)
ACGIH: 50 ppm; STEL 75 ppm
(1 ppm = 4.75 mg/m^3 @ NTP)

PROPERTIES: liquid; d 0.919 g/mL @ 20 °C; VP 1.25 kPa (9.4 mm Hg, 1.2%) @ 25 °C

SYNONYMS: glycidyl isopropyl ether; 1,2-epoxy-3-isopropoxypropane; (1-methylethoxy)methyl)oxirane; IGE

SAMPLING

SAMPLER: SOLID SORBENT TUBE (coconut shell charcoal; 100 mg/50 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 1 L @ 50 ppm
-MAX: 30 L

SHIPMENT: routine (refrigerate at lab)

SAMPLE STABILITY: not determined

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 121 to 484 mg/m^3 [1] (10-L samples)

BIAS: -5.5%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.067 [1]

ACCURACY: ±14.6%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: isopropyl glycidyl ether

DESORPTION: 0.5 mL CS_2, 30 min

TEMPERATURE-INJECTION: 205 °C
-DETECTOR: 270 °C
-COLUMN: 115 °C

INJECTION VOLUME: 5 µL

CARRIER GAS: N_2, 50 mL/min

COLUMN: stainless steel, 3.2-mm ID x 3 m, packed with 10% FFAP on 80/100 mesh Chromosorb W-AW DMCS

CALIBRATION: standard solutions of isopropyl glycidyl ether in CS_2

RANGE: 0.25 to 5 mg per sample

ESTIMATED LOD: not determined

PRECISION ($\hat{S}_r$): 0.020 [1]

APPLICABILITY: The working range is 5 to 105 ppm (25 to 500 mg/m^3) for a 10-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 S77 [2] in a revised format.

REAGENTS:

1. Carbon disulfide* (CS_2), chromatographic quality.
2. Isopropyl glycidyl ether*, reagent grade.
3. Nitrogen, purified.
4. Hydrogen, prepurified.
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 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator, and column (page 1620-1).
4. Vials, 2-mL, with PTFE-lined crimp caps.
5. Microliter syringes, 10-µL and convenient sizes for making dilutions.
6. Flasks, volumetric, 10-mL.
7. Pipets, 0.5-mL.

SPECIAL PRECAUTIONS: Isopropyl glycidyl ether (flash point = 33 °C) and CS_2 (flash point = -30 °C) are serious fire and explosion hazards and are toxic. All work with these compounds must be done 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 1 to 30 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_2 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 0.1 to 15 mg isopropyl glycidyl ether per sample.
   a. Add a known amount of isopropyl glycidyl ether to CS_2 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 (isopropyl glycidyl ether peak area vs. mg isopropyl 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 µL) of isopropyl glycidyl ether or standard solution of isopropyl glycidyl ether in 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 isopropyl glycidyl ether recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and recovery graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1620-1. 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 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 isopropyl 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 isopropyl 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:

Method S77 was issued on February 14, 1975 [2] and was validated over the range 121 to 484 mg/m^3 for 10-L air samples from dynamically generated test atmospheres [1]. The average recoveries ranged from 93 to 95%. The isopropyl glycidyl ether concentrations were independently measured by means of a total hydrocarbon analyzer. Breakthrough was not observed after sampling 44 L from a test atmosphere containing 484 mg/m^3 of isopropyl glycidyl ether. Desorption efficiency decreased from 92.4% to 77.5% with decreasing loading from 4.8 to 1.2 mg isopropyl glycidyl ether per sample. Sample stability was not determined; however, refrigeration of the sample upon receipt by the laboratory is recommended.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S77, 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, S77, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

METHOD REVISED BY:

R.A. Glaser, NIOSH/DPSE. Method S77 was originally validated under NIOSH Contract CDC-99-74-45.