ALLYL GLYCIDYL ETHER 2545

$CH_2=CHCH_2OCH_2CH(O)CH_2$ MW: 114.14 CAS: 106-92-3 RTECS: RR0875000

METHOD: 2545, Issue 1 **EVALUATION: FULL** **Issue 1: 15 August 1994**

OSHA : C 10 ppm
NIOSH: 5 ppm; STEL 10 ppm (skin)
ACGIH: 5 ppm; STEL 10 ppm (skin)
(1 ppm = 4.67 mg/m^3 @ NTP)

PROPERTIES: liquid; d 0.970 g/mL @ 20 °C; BP 154 °C; VP 0.63 kPa (4.7 mm Hg, 0.62%) @ 25 °C

SYNONYMS: 1-allyloxy-2,3-epoxypropane; ((2-propenyloxy)methyl)oxirane; AGE

SAMPLING

SAMPLER: SOLID SORBENT TUBE (Tenax GC; 100 mg/50 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 1.5 L @ 5 ppm
-MAX: 8 L

SHIPMENT: routine

SAMPLE STABILITY: at least 7 days @ 25 °C

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 19 to 87 mg/m^3 [1] (3-L samples)

BIAS: -1.3% [1]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.057 [1]

ACCURACY: ±12.5%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: allyl glycidyl ether

DESORPTION: 2 mL diethyl ether, 30 min

TEMPERATURE-INJECTION: 200 °C
-DETECTOR: 280 °C
-COLUMN: 150 °C

INJECTION VOLUME: 2 µL

CARRIER GAS: N_2, 30 mL/min

COLUMN: stainless steel, 3.2-mm ID x 6.1 m, packed with 10% FFAP on 100/120 mesh Supelcoport

CALIBRATION: standard solutions of allyl glycidyl ether and internal standard in diethyl ether

RANGE: 30 to 300 µg per sample

ESTIMATED LOD: 10 µg per sample

PRECISION ($\hat{S}_r$): 0.034 [1]

APPLICABILITY: The working range is 2 to 20 ppm (10 to 100 mg/m^3) for a 3-L air sample. An appropriate capillary column may be used for better resolution and sensitivity.

INTERFERENCES: None identified.

OTHER METHODS: This is Method S346 [2] in a revised format.

REAGENTS:

1. Allyl glycidyl ether*, reagent grade.
2. Diethyl ether*, anhydrous, containing approximately 0.1 mg/mL isoamyl alcohol or other suitable internal standard.
3. Hexane.
4. Nitrogen, purified.
5. Hydrogen, prepurified.
6. Air, compressed, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: borosilicate tubes, 10 cm long, 8-mm OD, 6-mm ID; flame-sealed ends with plastic caps, containing two sections of 35/60 mesh Tenax GC (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 2545-1).
4. Vials, 5-mL, with PTFE-lined crimp caps.
5. Microliter syringes, 10-µL and convenient sizes for making dilutions.
6. Flasks, volumetric, 10-mL.
7. Pipets, 2-mL.

SPECIAL PRECAUTIONS: Allyl glycidyl ether is flammable (flash point = 57 °C) and a moderate skin and severe eye irritant [3]. Diethyl ether is a serious fire and explosion hazard (flash point = -45 °C) and may form explosive peroxides during storage. 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 0.02 and 0.2 L/min for a total sample size of 1.5 to 8 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 2.0 mL diethyl ether 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 10 to 800 µg allyl glycidyl ether per sample.
   a. Add a known amount of allyl glycidyl ether to diethyl ether 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 (ratio of allyl glycidyl ether peak area to internal standard peak area vs. µg allyl glycidyl ether per 2 mL).

9. Determine desorption efficiency (DE) at least once per year for each lot of Tenax GC used for sampling in the range of interest. Determine for three samplers at each of five concentrations plus three media blanks.
   a. Transfer front sorbent section of media blank sampler to a 5-mL vial.
   b. Inject known amount (1 to 10 µL) of allyl glycidyl ether or standard solution of allyl glycidyl ether in hexane directly into sorbent in vial with a microliter syringe.
      NOTE: Standard solutions for DE determination should not contain internal standard.
   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. µg allyl 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 2545-1. 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 diethyl ether, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak areas. Divide peak area of allyl glycidyl ether by peak area of internal standard for each chromatogram.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE) of allyl 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 allyl glycidyl ether 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 S346 was issued on January 21, 1977 [2] and was validated over the range 19 to 87 mg/m^3 for 3-L air samples from dynamically generated test atmospheres [1]. The average recoveries ranged from 98.2 to 99.5%. The allyl glycidyl ether concentrations were independently calculated from analyte delivery rate and air dilution ratios. Breakthrough was observed after sampling 12 L from a test atmosphere containing 87 mg/m^3 of allyl glycidyl ether at 90% relative humidity. Desorption efficiency ranged from 101% to 89.8% for loadings of 67 to 269 µg allyl glycidyl ether per sample. A storage stability study gave average recoveries of 96.9% for 7-day storage versus 98.1% for 1-day storage.

REFERENCES:

[1] Backup Data Report for Allyl Glycidyl Ether, No. S346, prepared under NIOSH Contract No. 210-76-0123.
[2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, S346, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
[3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123, available as GPO Stock #17-033-00337-8 from Superintendent of Documents, Washington, D.C. 20402.

METHOD REVISED BY:

R. Alan Lunsford, Ph.D., NIOSH/DPSE. Method S346 was originally validated under NIOSH Contract No. 210-76-0123.