n-BUTYL GLYCIDYL ETHER 1616

$C_7H_{14}O_2$ MW: 130.19 CAS: 2426-08-6 RTECS: TX4200000

METHOD: 1616, Issue 1 **EVALUATION: PARTIAL** **Issue 1: 15 August 1994**

OSHA : 50 ppm
NIOSH: C 5.6 ppm; 15 min
ACGIH: 25 ppm
(1 ppm = 5.32 mg/m^3 @ NTP)

PROPERTIES: liquid; d 0.9087 g/mL @ 20 °C; BP 164 °C; VP 0.43 kPa (3.2 mm Hg, 4200 ppm) @ 20 °C

SYNONYMS: 1,2-epoxy-3-butoxypropane; butoxymethyloxirane; BGE

SAMPLING

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

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 15 L @ 25 ppm
-MAX: 30 L

SHIPMENT: routine (refrigerate at lab)

SAMPLE STABILITY: not determined

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 133 to 542 mg/m^3 [1] (10-L samples)

BIAS: -16.1% @ 133 mg/m^3 [1]
-1.0% @ 259 to 542 mg/m^3

OVERALL PRECISION ($\hat{S}_{rT}$): 0.074 [1]

ACCURACY: ±15.5% @ 259 to 592 mg/m^3
±30.6% @ 133 mg/m^3

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: n-butyl glycidyl ether

DESORPTION: 0.5 mL CS_2, 30 min

TEMPERATURE-INJECTION: 180 °C
-DETECTOR: 275 °C
-COLUMN: 130 °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 n-butyl glycidyl ether in CS_2

RANGE: 1.6 to 6.4 mg per sample

ESTIMATED LOD: not determined

PRECISION ($\hat{S}_r$): 0.016 [1]

APPLICABILITY: The working range is 15 to 60 ppm (80 to 320 mg/m^3) for a 20-L air sample. Because of unexpected bias at the lowest test level and the possibility of poor desorption efficiency at low loadings, a sample volume of at least 15 L is recommended. This method has not been evaluated for short-term exposures at 5.6 ppm. An appropriate capillary column may be used for better resolution and sensitivity.

INTERFERENCES: None identified.

OTHER METHODS: This is Method S81 [2] in a revised format.

REAGENTS:

1. Carbon disulfide* (CS_2), chromatographic quality.
2. n-Butyl 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 1616-1).
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: Both n-butyl glycidyl ether and CS_2 are toxic. In addition, CS_2 is a serious fire and explosion hazard (flash point = -30 °C). 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 15 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 8 mg n-butyl glycidyl ether per sample.
   a. Add a known amount of n-butyl 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 (n-butyl glycidyl ether peak area vs. mg n-butyl glycidyl ether).
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 n-butyl glycidyl ether or standard solution of n-butyl 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 n-butyl 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 1616-1. 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 n-butyl 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 n-butyl 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 S81 was issued on February 14, 1975 [2] and validated over the range 133 to 542 mg/m^3 for 10-L air samples from dynamically generated test atmospheres [1]. The n-butyl glycidyl ether concentrations were independently measured by means of a total hydrocarbon analyzer. The average recoveries for sets of six samples taken at concentrations of 542 and 259 mg/m^3 were 99.8% and 98.2%, respectively. A different method was used to generate concentrations of about 133 mg/m^3. For two sets of six samples taken at the latter concentration, the average recovery was 83.9%. The reason for the low recovery was not determined. Breakthrough was not observed after sampling 44 L from a test atmosphere containing 30 mg/m^3 of n-butyl glycidyl ether. The desorption efficiency decreased from 93.1 to 82.6% as loadings decreased of 6.4 to 1.6 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, S81, 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, S81, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

METHOD REVISED BY:

R.A. Glaser, NIOSH/DPSE. Method S81 was originally validated under NIOSH Contract CDC-99-74-45.