ETHYL ETHER

$(C_2H_5)_2O$ MW: 74.14 CAS: 60-29-7 RTECS: KI5775000

METHOD: 1610, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 May 1985**
Issue 2: 15 August 1994

OSHA : 400 ppm
NIOSH: no recommended standard
ACGIH: 400 ppm; STEL 500 ppm
(1 ppm = 3.03 mg/m^3 @ NTP)

PROPERTIES: liquid; d 0.7134 g/mL @ 20 °C; BP 34.6 °C; MP -116 °C; VP 59 kPa (440 mm Hg; 58% v/v) @ 20°C; explosive range 1.7 to 48% v/v in air

SYNONYMS: diethyl ether; 1,1'-oxybisethane; ethyl oxide

SAMPLING

SAMPLER: SOLID SORBENT TUBES (coconut shell charcoal, 100 mg/50 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 0.25 L @ 400 ppm
-MAX: 3 L

SHIPMENT: routine

SAMPLE STABILITY: unknown

FIELD BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 606 to 2400 mg/m^3 [1] (3-L samples)

BIAS: 5.2%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.053

ACCURACY: ± 14.6%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: ethyl ether

DESORPTION: 1.0 mL ethyl acetate; 30 min

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 195 °C
-DETECTOR: 250 °C
-COLUMN: 175 °C

CARRIER GAS: N_2, 30 mL/min

COLUMN: stainless steel; 1.2 m x 6-mm OD, packed with 50/80 mesh Porapak Q

CALIBRATION: solutions of ethyl ether in ethyl acetate

RANGE: 0.3 to 11 mg per sample

ESTIMATED LOD: 0.01 mg per sample [2]

PRECISION ($\hat{S}_r$): 0.024 @ 1.8 to 7.1 mg per sample [1]

APPLICABILITY: The working range is 100 to 2700 mg/m^3 (33 to 880 ppm) for a 3-L air sample. During sampling, high humidity may greatly decrease the breakthrough volume. A sampling period of 15 min may be monitored for STEL compliance using a flow rate of 0.2 L/min.

INTERFERENCES: Carbon disulfide, hexane, and 2-butanone have similar GC retention times [1]. A 30-m x 0.32-mm ID fused silica capillary column with 1-µm DB-1 at 50 °C may be used [2].

OTHER METHODS: This revises Method S80 [3].

REAGENTS:

1. Ethyl acetate, reagent grade.*
2. Ethyl ether, reagent grade.*
3. Nitrogen, prepurified.
4. Hydrogen, prepurified.
5. Air, filtered, compressed.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends with plastic caps, 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 must be less than 3.4 kPa at 1 L/min airflow. 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 1610-1).
4. Vials, glass, 2-mL, PTFE-lined caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Flasks, volumetric 10-mL.
7. Pipet, volumetric, 1-mL with pipet bulb.

SPECIAL PRECAUTIONS: Ethyl ether is a dangerous fire and explosion hazard (flash point = -45 °C) and tends to form explosive peroxides [4]. Ethyl acetate is flammable (flash point = -4 °C). Work with these compounds only 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 0.25 to 3 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 1.0 mL ethyl acetate to each vial. Attach 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 covering the range 0.01 to 11 mg ethyl ether per sample.
   a. Add known amounts of ethyl ether to ethyl acetate in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain ethyl ether concentrations in the range 0.01 to 11 mg/mL.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs. mg ethyl ether).
9. Determine desorption efficiency (DE) at least once for each lot 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 the back sorbent section of a media blank sampler.
b. Inject a known amount (1 to 20 µL) of ethyl ether or a standard solution of ethyl ether in ethyl acetate directly onto the front sorbent section with a microliter syringe.
c. Cap the tube. Allow to stand overnight.
d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
e. Prepare a graph of DE vs. mg ethyl 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 1610-1. 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 ethyl acetate, reanalyze, and apply the appropriate dilution factor in the calculations.
12. Measure peak area.

CALCULATIONS:

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

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

EVALUATION OF METHOD:

Method S80 was issued on February 14, 1975 [3], and evaluated over the range 630 to 2500 mg/m^3 at 22 °C and 766 mm Hg for 3-L air samples desorbed in 0.5 mL ethyl acetate [1]. The concentrations were verified by using a total hydrocarbon analyzer. Breakthrough (5% on the backup section) occurred at 33 min when sampling a concentration of 2470 mg/m^3 in dry air at a flow rate of 0.185 L/min., corresponding to a capacity of 15 mg. The average DE was 0.98 over the range 1.8 to 7.1 mg ethyl ether per sample. Sample storage stability was not determined.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S80, 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] UBTL, Inc., NIOSH Sequences 3610-J (unpublished, August 23, 1982) and 3726-J (unpublished, December 28, 1982).
[3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S80, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
[4] 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 #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.

METHOD REVISED BY:

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