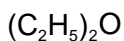


ETHYL ETHER

1610



MW: 74.14

CAS: 60-29-7

RTECS: KI5775000

METHOD: 1610, Issue 3

EVALUATION: FULL

Issue 1: 15 May 1985

Issue 3: 15 March 2003

OSHA : 400 ppm
NIOSH: no REL
ACGIH: 400 ppm; STEL 500 ppm

(1 ppm = 3.03 mg/m³ @ 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		MEASUREMENT	
SAMPLER:	Solid Sorbent Tube (coconut shell charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	ethyl ether
VOL-MIN:	0.25 L @ 400 ppm	DESORPTION:	1.0 mL CS ₂ ; 30 min with agitation
-MAX:	3 L	INJECTION VOLUME:	1 µL
SHIPMENT:	routine	TEMPERATURE:	
SAMPLE STABILITY:	14 days @ 5 °C	-INJECTION:	250 °C
BLANKS:	2 to 10 field blanks per set	-DETECTOR:	300 °C
		-COLUMN:	35 °C (1 min) - 150 °C @ 10 °C/minute, (split 5:1)
		CARRIER GAS:	helium (1-2 mL/min)
		COLUMN:	capillary, fused silica 30 m x 0.32-mm ID; 1-µm film 100% dimethyl polysiloxane, Rtx-1 or equivalent
		CALIBRATION:	solutions of ethyl ether in CS ₂
		RANGE:	2 to 430 µg per sample (capillary column); 1800 to 7100 µg per sample (packed column)
		ESTIMATED LOD:	0.2 µg per sample
		PRECISION (S_r):	0.071 @ 9 to 400 µg per sample
ACCURACY			
RANGE STUDIED:	606 to 2400 mg/m ³ (3-L samples)[2]		
BIAS:	5.2%		
OVERALL PRECISION (S_r):	0.088		
ACCURACY:	± 20%		

APPLICABILITY: The working range is 100 to 2700 mg/m³ (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.[3]

INTERFERENCES: Any compound, such as pentane, with similar retention times.

OTHER METHODS: This revises Method S80 [4] and NMAM 1610 (issue 2) [4].

REAGENTS:

1. Carbon Disulfide, low benzene grade.*
2. Ethyl ether, reagent grade.*
3. Helium, prepurified and filtered.
4. Hydrogen, prepurified and filtered.
5. Air, prepurified and filtered.

* 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 (see page 1610-1).
4. Vials, glass, 2-mL, PTFE-lined caps.
5. Syringe, 10- μ L, 25- μ L, 250- μ L.
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. Carbon disulfide is toxic and flammable (flash point = -30 °C). Prepare samples and standards in a well-ventilated 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. Include the glass wool plug in the vial with the front sorbent section.
6. Add 1.0 mL carbon disulfide 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 from below the LOD to 10 times the LOQ. If necessary, additional standards may be added to extend the calibration curve.
 - a. Add known amounts of ethyl ether to carbon disulfide in 10-mL volumetric flasks and dilute to the mark. Prepare additional standards as needed by serial dilution.
 - b. Analyze together with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (peak area vs. μ g ethyl ether per sample).

9. Determine desorption efficiency (DE) at least once for each lot of charcoal used for sampling in the calibration range (step 8).
 - a. Prepare three tubes at each of five levels plus three media blanks.
 - b. Remove and discard the back sorbent section of a blank sampler.
 - c. Inject a known amount (1 to 20 μL) of ethyl ether or a standard solution of ethyl ether in carbon disulfide directly onto the front sorbent section with a microliter syringe.
 - d. Cap the tube. Allow to stand overnight.
 - e. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - f. Prepare a graph of DE vs. μg 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 sample with carbon disulfide, reanalyze, and apply the appropriate dilution factor in the calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, μg (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):

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

EVALUATION OF METHOD:**Issues 1 and 2**

Method S80 was issued on February 14, 1975 [4], 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 [2]. 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 98% over the range 1.8 to 7.1 mg ethyl ether per sample. Sample storage stability was not determined.

Issue 3

The sensitivity of this method for ethyl ether was lowered to cover a range from approximately 10 times the LOQ (based upon an improved LOD of 0.2 $\mu\text{g}/\text{sample}$) to 500 times (0.25 REL) the LOQ. The desorption efficiency was tested at four levels over the range of 8 - 428 $\mu\text{g}/\text{sample}$. The average recovery for ethyl ether over this range was 102.5%. A 30-day storage stability study for ethyl ether was completed during the method development. The storage stability was determined at a level approximately 0.2 REL. The analyte was determined to be stable for 14 days when stored at 5°C with an average recovery of 90.3% [1].

REFERENCES:

- [1] Pendergrass SM, May LR [2000]. NMAM 1610, Issue 3, Backup Data Report. Cincinnati, OH: National Institute for Occupational Safety & Health, DART (unpublished), April.
- [2] NIOSH [1977]. Documentation of the NIOSH Validation Tests, S80, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185, available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.
- [3] NIOSH/OSHA [1981]. NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services and U.S. Department of Labor, January 1981, Publ. (NIOSH) 81-123, available as GPO Stock #17-033-00337-8 from Superintendent of Documents, Washington, D.C. 20402.
- [4] NIOSH [1977]. NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S80, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B. S80 originally validated under NIOSH Contract CDC-99-74-45.

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