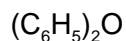


PHENYL ETHER

1617



MW: 170.22

CAS: 101-84-8

RTECS: KN8970000

METHOD: 1617, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 August 1994

OSHA : 1 ppm
 NIOSH: 1 ppm
 ACGIH: STEL 2 ppm
 (1 ppm = 6.96 mg/m³)

PROPERTIES: solid, geranium-like odor; MP 27 °C;
 BP -257 °C; d 1.072 g/mL @ 22 °C;
 VP 2.7 Pa (0.02 mm Hg) @ 25 °C;
 vapor density 5.86 (air = 1);
 flash pt. 115 °C; flammable limits in air
 0.8 to 1.5%

SYNONYMS: diphenyl ether; diphenyl oxide; 1,1'-oxybisbenzene.

SAMPLING		MEASUREMENT	
SAMPLER:	CHARCOAL TUBE (coconut shell charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	phenyl ether
VOL-MIN:	1 L @ 1 ppm	EXTRACTION:	0.5 mL carbon disulfide, 30 min
-MAX:	50 L	INJECTION VOLUME:	5 µL
SHIPMENT:		TEMPERATURE-INJECTION:	230 °C
SAMPLE STABILITY:	not determined	-DETECTOR:	265 °C
BLANKS:	2 to 10 field blanks per set	-COLUMN:	215 °C
ACCURACY		COLUMN:	10 ft x 1/8-in. ID stainless steel column packed with 10% FFAP on 80/100 mesh, acid washed DMCS Chromosorb W
RANGE STUDIED:	3.0 to 13.0 mg/m ³ [1] (10-L samples)	DETECTOR:	flame ionization detector
BIAS:	0.06%	CALIBRATION:	standard solutions of phenyl ether in hexane
OVERALL PRECISION (Ŝ_{r,T}):	0.07 [1,2]	RANGE:	7 to 210 µg per sample [1,2]
ACCURACY:	± 13.7%	ESTIMATED LOD:	0.7 µg per sample [1,2]
		PRECISION (Ŝ_r):	0.048 [1,2]

APPLICABILITY: The working range is 0.1 to 9 ppm (0.7 to 60 mg/m³) for a 10-L air sample. Better sensitivity may be achieved with a capillary column with appropriate adjustment in the instrumental conditions. A DB-wax (polyethelyene glycol) or e equivalent is similar to the FFAP packed column.

INTERFERENCES: None identified.

OTHER METHODS: This revises Method S72 [3].

REAGENTS:

1. Phenyl ether^{*}, ACS reagent grade.
2. Carbon disulfide^{*}, ACS reagent grade.
3. Hexane, distilled in glass.
4. Calibration stock solution, 17.3 mg/mL, of phenyl ether in hexane.
5. Prepurified nitrogen.
6. Prepurified hydrogen.
7. Filtered compressed air.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tubes, 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 at a 1 L/min airflow must be less than 4.3 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible polyethylene or PTFE tubing.
3. PTFE plugs and/or tubing.
4. Vials, glass, 1-mL with PTFE-lined caps.
5. Gas chromatograph with a flame ionization detector, recorder, integrator and column (page 1617-1).
6. Tweezers.
7. Syringes, 5-, 10- and 100- μ L; other sizes as needed.
8. Volumetric flasks, 10- and 25-mL.
9. Pipets, 0.5-, 1-, and 10-mL glass, delivery, with pipet bulb, graduated to 0.1-mL increments.
10. Graduated cylinders, glass, 25-mL.
11. File.

SPECIAL PRECAUTIONS: Phenyl ether is toxic by inhalation. All work with carbon disulfide should be performed in a hood because of its high toxicity and flammability (flash point = -30 °C).

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Immediately before sampling, break the ends of the tube to provide an opening at least one-half the internal diameter of the tube (2 mm). Attach sampler to personal sampling pump with flexible tubing.
3. Sample 1 to 50 L of air at an accurately known flow rate between 0.01 and 0.2 L/min.
4. Record the humidity temperature, and pressure of the atmosphere being sampled, if pressure reading is not available, record the elevation.
5. Cap each tube with plastic plugs and pack securely for shipment.
6. Collect a bulk sample (ca. 1 g) in a glass vial and ship it separately.

SAMPLE PREPARATION:

7. Place front and back sections of the sampler in separate 1-mL vials. Discard the glass wool and foam plugs.
8. Add 0.5 mL carbon disulfide to each sample vial and attach the crimp cap.
9. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate daily with at least six working standards over the range of 7 to 210 µg per sample.
 - a. Add known amounts of calibration stock solution to carbon disulfide in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze working standards together with samples and blanks (steps 13 and 14).
 - c. Prepare a calibration graph of area vs. µg of analyte per sample.
11. Determine desorption efficiency (DE) for each lot of charcoal tubes used for sampling in the concentration 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 a known amount of phenyl ether (or a solution of phenyl ether in hexane) directly onto front (100 mg) sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 7 through 9) and analyze together with standards (steps 13 and 14).
 - e. Prepare a graph of desorption efficiency vs. µg phenyl ether per sample.
12. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

13. Set instrument according to manufacturer's recommendations and to conditions given on page 1617-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
NOTE: If peak area is above linear range of the working standards, dilute an aliquot with carbon disulfide, reanalyze, and apply the appropriate dilution factor in calculations.
14. Measure peak area.

CALCULATIONS:

15. Determine mass, µg (corrected for DE), of analyte found in the sample front (W_f) and back (W_b) sorbent sections and in the average media blanks front (B_f) and back (B_b) sorbent sections.
NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
16. Calculate concentration (C) of analyte in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

This method was evaluated over the range 3.0 to 13.3 mg/m³ with 10-L samples collected from dry (0% RH) air [1,2]. Overall sampling and measurement precision, \hat{S}_{rt} , was 0.07, with average recovery of 100.6%. The test atmosphere concentration of phenyl ether was monitored with a Beckman model 402 total hydrocarbon analyzer. Desorption efficiency (DE) at 7 µg per sample was 80% and approximately 90% in the range 32 to 120 µg per sample. Sample recovery (corrected for DE) was 100.2% over the range 32 to 120 µg. No breakthrough was seen after 4 h when a concentration of 13.1 mg/m³ was sampled at a flow rate of 0.185 L/min. Sample stability was not determined.

REFERENCES:

- [1] Backup Data Report, Phenyl Ether, No. S72, prepared under NIOSH Contract CDC-99-74-45.
- [2] Documentation of NIOSH Validation Tests, NIOSH Contract. CDC-99-74-45.
- [3] NIOSH Manual of Analytical Methods, 2nd. ed., V. 2, S72, U. S. Dept. Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

METHOD REVISED BY:

Y.T. Gagnon, NIOSH/DPSE.