DICHLOROETHYL ETHER

$(ClCH_2CH_2)_2O$ MW: 143.01 CAS: 111-44-4 RTECS: KN0875000

METHOD: 1004, Issue 2 **EVALUATION: FULL** **Issue 1: 15 February 1984**
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OSHA : C 15 ppm (skin)
NIOSH: 5 ppm; STEL 10 ppm (skin); carcinogen
ACGIH: 5 ppm; STEL 10 ppm (skin); carcinogen
(1 ppm = 5.85 mg/m^3 @ NTP)

PROPERTIES: liquid; d 1.218 g/mL @ 20 °C;
BP 178 °C; MP -52 °C;
VP 50 Pa (0.4 mm Hg; 530 ppm) @ 20 °C

SYNONYMS: bis(2-chloroethyl) ether; 1,1'-oxybis[2-chloroethane]; 2,2'-dichlorodiethyl ether; sym-dichloroethyl ether

SAMPLING

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

FLOW RATE: 0.01 to 1 L/min

VOL-MIN: 2 L @ 15 ppm
-MAX: 15 L

SHIPMENT: routine

SAMPLE STABILITY: at least 7 days @ 25 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 45 to 180 mg/m^3 [1]
(15-L samples)

BIAS: - 7.61%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.059 [1]

ACCURACY: ± 15.7%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: dichloroethyl ether

DESORPTION: 1 mL CS_2; stand 30 min

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 200 - 225 °C
-DETECTOR: 250 - 275 °C
-COLUMN: 100 - 135 °C

CARRIER GAS: N_2 or He, 30 mL/min

COLUMN: 3 m x 3-mm ID stainless steel; 10% FFAP on 80/100 mesh DMCS Chromosorb W-AW or equivalent

CALIBRATION: dichloroethyl ether in CS_2

RANGE: 0.15 to 4 mg per sample [2]

ESTIMATED LOD: 0.01 mg per sample [3]

PRECISION ($\hat{S}_r$): 0.007 [1]

APPLICABILITY: The working range is 1.7 to 46 ppm (10 to 270 mg/m^3) for a 15-L air sample. This method may be used for simultaneous analysis of two or more analytes by changing the chromatographic conditions (e.g., temperature programming) . High humidity during sampling will greatly reduce breakthrough volume.

INTERFERENCES: None identified. Alternate columns, e.g., 10% SP-1000 on 80/100 mesh Supelcoport, SP-2100 with 0.1% Carbowax 1500 or DB-1 fused silica capillary column may be used.

OTHER METHODS: This is Method S357 [2] in a revised format.

REAGENTS:

1. Eluent: Carbon disulfide*, chromatographic quality containing 0.1% (v/v) toluene or other suitable internal standard.
2. Dichloroethyl ether (DCEE).
3. Hexane.
4. Calibration stock solution, 0.244 mg/µL. Dilute 2.44 g DCEE (2.0 mL at 20 °C) to 10 mL with hexane. Prepare in duplicate.
5. Nitrogen, purified.
6. Hydrogen, prepurified.
7. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, 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 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detector, integrator and column (page 1004-1).
4. Vials, 2-mL, PTFE-lined caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C); work with it 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 1 L/min for a total sample size of 2 to 15 L.
4. Cap the samplers with plastic (not rubber) caps and 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 eluent to each vial. Attach crimp 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 over the range 0.01 to 4 mg DCEE per sample.
   a. Add known amounts of calibration stock solution to eluent in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (ratio of peak area of analyte to peak area of internal standard vs. mg DCEE).
9. Determine desorption efficiency (DE) at least once for each batch 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 back sorbent section of a media blank sampler.

b. Inject a known amount of calibration stock solution 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 together with working standards (steps 11 and 12).
e. Prepare a graph of DE vs. mg DCEE recovered.

10. Analyze three quality control blind spikes and three analyst spikes to insure 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 1004-1. 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 with eluent, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area. Divide the peak area of analyte by the peak area of internal standard on the same chromatogram.

CALCULATIONS:

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

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

EVALUATION OF METHOD:

Method S357 [2] was issued on May 21, 1976, and validated over the range 45 to 180 mg/m^3 at 24 °C and 762 mm Hg using a 15-L sample [1]. Overall precision, $\hat{S}_{rT}$, was 0.059 with average recovery 90.7%, representing a non-significant bias. The concentration of DCEE was independently verified by a direct hydrocarbon analyzer. Desorption efficiency was 0.94 in the range 0.7 mg to 2.7 mg per sample. Breakthrough (5% on back section) occurred at 115 min when sampling an atmosphere containing 161 mg/m^3 at 0.9 L/min in dry air.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, NIOSH, S357, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
[2] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S357, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
[3] User check, UBTL, NIOSH Sequence #4121-J (unpublished, November 21, 1983).

METHOD REVISED BY:

G. David Foley and Y. T. Gagnon, NIOSH/DPSE; S357 originally validated under NIOSH Contract CDC-99-74-45.