ETHYLENE GLYCOL 5500

$HOCH_2CH_2OH$ MW: 62.07 CAS: 107-21-1 RTECS: KW2975000

METHOD: 5500, Issue 2 **EVALUATION: FULL** **Issue 1: 15 February 1984**
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OSHA : no standard
NIOSH: Group III Pesticide
ACGIH: C 50 ppm
(1 ppm = 2.54 mg/m^3 @ NTP)

PROPERTIES: liquid; MP - 13 °C; BP 198 °C;
d 1.1135 g/mL @ 20 °C;
VP 0.007 kPa (0.20 mm Hg) @ 20°C;
explosive limits 3.2 to 15.3% in air

SYNONYMS: 1,2-ethanediol; 1,2-Dihydroxyethane; ethylene alcohol; ethylene dihydrate

SAMPLING

SAMPLER: FILTER + SORBENT
(glass fiber filter + silica gel, 520 mg/260 mg)

FLOW RATE: 0.2 L/min

VOL-MIN: 0.3 L
-MAX: 60 L

SHIPMENT: filter in glass vial with 1 mL 2% (v/v) 2-propanol/H_2O directly after sampling; silica gel tube sealed with plastic caps

SAMPLE STABILITY: at least 15 days (silica gel) @ 25 °C [1]

FIELD BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 45 to 98 mg/m^3 [1]

BIAS: 3.3% [1]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.084 (aerosol); 0.087 (vapor) [1]

ACCURACY: ± 20.4%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: ethylene glycol

SILICA GEL DESORPTION: 1 mL 2% (v/v)
2-propanol in H_2O, sonicate for 15-30 min
(5 min not enough to desorb analyte)

INJECTION VOLUME: 1 µL

TEMPERATURE-INJECTION: 250 °C
-DETECTOR: 300 °C
-COLUMN: 165 °C

CARRIER GAS: N_2 or He, 30 mL/min

COLUMN: glass, 1.9 m x 2-mm; 3% Carbowax 20M on 80/100 Chromosorb 101

CALIBRATION: solutions of ethylene glycol in 2% (v/v) 2-propanol in H_2O

RANGE: 0.02 to 1 mg per sample

ESTIMATED LOD: 4 µg per sample

PRECISION ($\hat{S}_r$): 0.060 (filters); 0.061 (silica gel) [1]

APPLICABILITY: The working range is 7 to 330 mg/m^3 for a 3-L air sample.

INTERFERENCES: A ghosting phenomenon which was described by Spitz [2] caused little or no error in measurements when samples were analyzed with standards at similar concentrations. Improved separation of ethylene glycol and other glycols can be achieved using a 30-m-Rtx-35 (0.53 mm ID; 3.0 µm film) capillary column.

OTHER METHODS: This method originally was designated P&CAM 338 [3] and was evaluated with a reference method which involved sampling with bubblers containing water, oxidation of ethylene glycol to formaldehyde with periodic acid and co lorimetric analysis [1].

REAGENTS:

1. Ethylene glycol.
2. 2-Propanol/water, 2% (v/v). Add 2 volumes 2-propanol to 98 volumes freshly distilled water.
3. Helium or nitrogen, purified.
4. Hydrogen, prepurified.
5. Air, filtered.
6. Calibration stock solution, 10 mg/mL, in 2% 2-propanol/water.

EQUIPMENT:

1. Sampler: 13-mm glass fiber filter, free of binders, in filter holder (Cat. No. SX00 013 00, Millipore Corp. or equivalent) followed by glass tube, 8 cm long, 8-mm OD, 6-mm ID; two sections of 20/40 mesh silica gel (d = 0.72 g/cm^3; surface area, 720 to 760 m^2/g) separated by 3-mm urethane foam plug (front = 520 mg; backup = 260 mg). Filter holder and glass tube connected with two short pieces of plastic tubing. One piece (7 mm long) fits tightly around outlet of filter holder; other piece fits over first piece and inlet of glass tube.
2. Personal sampling pump, 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 5500-1).
4. Vials, glass, 1-mL, rubber caps.
5. Syringes, 10-µL, readable to 0.1 µL.
6. U-tube, glass, 25 cm x 15-mm ID.
7. Constant temperature bath, 75 °C.
8. Volumetric flasks, 10-mL

SPECIAL PRECAUTIONS: None.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at 0.2 L/min for a total sample size of 0.3 to 60 L.
3. Immediately after sampling, disassemble the sampler and transfer the filter to a vial containing 1 mL 2% 2-propanol/water. Seal the vial.
4. Seal the ends of the silica gel tubes with plastic caps. Pack securely for shipment.

SAMPLE PREPARATION:

5. Transfer front and backup sections of silica gel to separate vials. Add 1 mL 2% 2-propanol/water to each vial.
6. Place samples in ultrasonic bath for 15 to 30 minutes.

CALIBRATION AND QUALITY CONTROL:

7. Calibrate daily with at least six working standards over the range 0.001 to 1 mg ethylene glycol per sample.
   a. Add known amounts of calibration stock solution to 2% 2-propanol/water in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 10 through 12).
   c. Prepare calibration graph (peak area or height vs. mg ethylene glycol).
8. Determine recovery from filters in the calibration range (step 7). Prepare three filters at each of three levels plus three media blanks.
   a. Place a filter in a clean vial.

b. Add a known quantity of ethylene glycol in 5 µL water solution to the filter.
c. Seal the vial; ultrasonicate for 15 to 30 minutes.
d. Prepare and analyze the filters together with working standards (steps 5, 6, and 10 through 12).
e. Calculate recovery (ethylene glycol recovered/ethylene glycol taken).

9. Determine DE from silica gel for each lot of silica gel in the range of interest. Prepare three tubes at each of five levels plus three media blanks.
   a. Draw ethylene glycol vapor with a pump at 0.2 L/min into sampler tubes with a pump from a U-tube partly immersed in a constant temperature bath at 75 °C.
      NOTE: Values of DE for liquid and vapor spikes may be different [3].
   b. Desorb (steps 5 and 6) and analyze (steps 10 through 12) together with working standards.
   c. Prepare a graph of DE vs. mg ethylene glycol recovered.

MEASUREMENT:

10. Set gas chromatograph according to conditions given on page 5500-1. Set flow rates of hydrogen and air according to manufacturer's instructions.
11. Inject sample aliquot manually using solvent flush technique or with autosampler. t_r = 4 min for ethylene glycol under these conditions.
12. Measure peak area or peak height.

CALCULATIONS:

13. Determine the mass, mg (corrected for recovery or DE) of ethylene glycol found on the filter (W) and in the sample tube front (W_f) and back (W_b) sorbent sections, and in the average media blank filter (B), and 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 ethylene glycol in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

The method was tested with spiked samplers and atmospheres generated with a diffusion cell and verified with bubblers of water followed by colorimetric analysis for 54 to 98 mg/m^3. S_r = 0.084 (six samples) for 6-L samples at 14 mg/m^3 (aerosol); $\bar{S}_r$ = 0.065 (17 samples, pooled) for 6-L samples at 45 to 84 mg/m^3 (vapor). Breakthrough volume (79 mg/m^3, 0.2 L/min) = 261 L; recovery from filters (20 to 900 mg per sample) = 1.02; DE from silica gel (20 to 3120 µg per sample) = 0.81 to 0.87; filter storage stability = 49% of 85 mg evaporated from filters during 4 h at 24 °C, 78 µg stable on silica gel for 15 days at 25 °C. Area samples were collected side by side for 4 h at a field location by this method and a reference method (bubblers and colorimetric analysis); concentrations by this method, 0.63, 0.59, and 0.23 mg/m^3, were 1.4, 0.7 and 26.5% higher than corresponding concentrations by the reference method.

REFERENCES:

[1] Tucker, S. P., and G. J. Deye. Anal. Lett., 14 (A12), 959-976 (1981).
[2] Spitz, H. D. J. Pharm. Sci., 1339-1340 (1972).
[3] NIOSH Manual of Analytical Methods, 2nd ed., V. 7, P&CAM 338, U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1981).

METHOD WRITTEN BY:

Samuel P. Tucker, Ph.D., and Gregory J. Deye, NIOSH/DPSE.