Table 1 MW: Table 1 CAS: Table 1 RTECS: Table 1

METHOD: 1403, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 February 1984**
Issue 2: 15 August 1994

OSHA : Table 1
NIOSH: Table 1
ACGIH: Table 1

PROPERTIES: Table 1

COMPOUNDS and SYNONYMS: (1) 2-methoxyethanol: methyl cellosolve; ethylene glycol monomethylether; EGME
(2) 2-ethoxyethanol: cellosolve; ethylene glycol monoethylether, EGEE
(3) 2-butoxyethanol: butyl cellosolve; ethylene glycol monobutylether, EGBE

SAMPLING

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

FLOW RATE: 0.01 to 0.05 L/min

	(1)	(2)	(3)
VOL-MIN:	6 L	1 L	2 L
-MAX:	50 L	6 L	10 L

SHIPMENT: routine

SAMPLE STABILITY: unknown; store in freezer

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: see EVALUATION OF METHOD

BIAS: see EVALUATION OF METHOD

OVERALL PRECISION ($\hat{S}_{rT}$): see EVALUATION OF METHOD

ACCURACY: ± 17%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: compounds above

DESORPTION: 1 mL 5% methanol in CH_2Cl_2

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 200 °C
-DETECTOR: 250-300 °C
-COLUMN: (1) 95 °C; (2) 140 °C
(3) 145 °C

CARRIER GAS: N_2 or He, 30 mL/min

COLUMN: glass, 3 m x 2-mm ID, 10% SP-1000 on 80/100 mesh Chromosorb WHP, or equivalent

CALIBRATION: solutions of analyte in eluent (internal standard optional)

RANGE AND PRECISION: see EVALUATION OF METHOD

ESTIMATED LOD: 0.01 to 0.02 mg per sample [1]

APPLICABILITY: This method may be used to determine two or more analytes simultaneously by varying GC conditions (e.g., temperature programming).

INTERFERENCES: High humidity may reduce sampling capacity. The methods were validated using a 3 m x 3-mm stainless steel column packed with 10% FFAP on Chromosorb W-AW; other columns with equal or better resolution (e.g., capillary) ma y be used. Less volatile compounds may displace more volatile compounds on the charcoal.

OTHER METHODS: This method combines and replaces Methods S79 [2], S361 [3], and S76 [2]. Butyl cellosolve and butyl cellosolve acetate are included in OSHA Method 83. Cellosolve and cellosolve acetate are included in OSHA Method 79. Bot h of these OSHA methods are very similar to Method 1403.

REAGENTS:

1. Eluent: methylene chloride with 5% (v/v) methanol and 0.2% (v/v) 1-heptanol, 0.1% (v/v) ethyl benzene or other suitable internal standard.
2. Analyte.
3. Nitrogen, purified.
4. Hydrogen, prepurified.
5. Air, compressed, filtered.

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 0.05 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 1403-1).
4. Vials, glass, 2-mL, PTFE-lined crimp caps.
5. Syringe, 10-µL, readable to 0.1 µL.

SPECIAL PRECAUTIONS: Methylene chloride is a carcinogen [4].

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 0.05 L/min for a total sample size of 6 to 50, 2-methoxyethanol; 1 to 6, 2-ethoxyethanol; 2 to 10, 2-butoxyethanol.
   NOTE: Maximum flow rate for 2-methoxyethanol and 2-butoxyethanol is 0.2 L/min.
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 covering the range of the samples.
   a. Add known amounts of analyte 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 analyte).
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 analyte 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 analyte 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 1403-1. 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 analyte 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 analyte 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:

Methods S79 (2-methoxyethanol), S361 (2-ethoxyethanol) and S76 (2-butoxyethanol) were issued on February 14, 1975 [2,5], March 17, 1978 [3,6,7], and February 14, 1975 [2,5], respectively, and validated using, respectively, 50-, 6- and 10-L air samples of atmospheres generated by calibrated syringe drive. Storage stability of these alcohols was not determined. Precision and recovery were as shown below, representing non-significant bias in each method:

Method	Overall Precision ($\hat{S}_{rT}$)	Recovery (%)	Range Studied mg/m^3	Range Studied mg per sample	Breakthrough @ 2X OSHA	Avg. DE	Measurement Precision ($\bar{S}_r$)
S79 [1,4]	0.068	93	44 to 160	2 to 8	128 L*	0.98	0.008
S361 [2,5,6]	0.059	107	340 to 1460	2 to 7	>10 L**	1.02	0.009
S76 [1,4]	0.060	92	124 to 490	1 to 5	>44 L*	0.99	0.009

*Dry air.
**90% RH.
The method was also successfully checked for spiked samples of 2-butoxyethyl acetate [8].

REFERENCES:

[1] User check, UBTL, NIOSH Sequence #3990-Z (unpublished, November 3, 1983).
[2] NIOSH Manual of Analytical Methods, 2nd ed., V. 2., S76 and S79, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

[3] Ibid, V. 5, S361, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).
[4] NIOSH Recommendations for Occupational Safety and Health, U.S. Department of Health and Human Services (NIOSH) Publ. 92-100 (January, 1992).
[5] Documentation of the NIOSH Validation Tests, S76 and S79, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
[6] Backup Data, S361, available as "Ten NIOSH Analytical Methods, Set 6," Order No. PB 288-629 from NTIS, Springfield, VA 22161.
[7] NIOSH Research Report, Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Dept. of Health and Human Services Publ. (NIOSH) 80-133 (1980).
[8] User Check, DataChem Laboratories, NIOSH Sequence #6960-J,K (unpublished, August 15, 1990).

METHOD REVISED BY:

George Williamson, NIOSH/DPSE; methods originally validated under NIOSH Contracts 99-74-45 and 210-76-0123.

TABLE 1. GENERAL INFORMATION

Compound CAS# RTECS#	Exposure Limits (ppm) OSHA	 NIOSH	 ACGIH	Formula	mg/m^3 = 1 ppm @ NTP	MW	Density @ 20 °C (g/mL)	BP (°C)	VP @ 20 °C, kPa (mm Hg)
2-Methoxyethanol 109-86-4 KL5775000	25 (skin)	0.1 (skin)	5 (skin)	$HOCH_2CH_2OCH_3$	3.11	76.09	0.966	124	0.8 (6)
2-ethoxyethanol 110-80-5 KK8050000	200 (skin)	0.5 (skin)	5 (skin)	$HOCH_2CH_2OCH_2CH_3$	3.68	90.12	0.931	135	0.5 (4)
2-butoxyethanol 111-76-2 KJ8575000	50 (skin)	5 (skin)	25 (skin)	$HOCH_2CH_2O(CH_2)_3CH_3$	4.83	118.17	0.902	171	0.11 (0.8)