(1) $HOCH_2CH_2OCH_3$	MW: (1) 76.09	CAS: (1) 109-86-4	RTECS: (1) KL5775000
(2) $HOCH_2CH_2OCH_2CH_3$	(2) 90.12	(2) 110-80-5	(2) KK8050000
(3) $HOCH_2CH_2O(CH_2)_3CH_3$	(3) 118.17	(3) 111-76-2	(3) KJ8575000

METHOD: 1403, Issue 3 **EVALUATION: FULL** **Issue 1: 15 August 1990**
Issue 3: 15 March 2003

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

PROPERTIES: See Table 1

SYNONYMS: (1) 2-methoxyethanol: methyl cellosolve, ethylene glycol monomethyl ether, EGME
(2) 2-ethoxyethanol: cellosolve, ethylene glycol monoethyl ether, EGEE
(3) 2-butoxyethanol: butyl cellosolve, ethylene glycol monobutyl ether, 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: 30 days @ 5°C

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: See Table 2.

BIAS: See Table 2.

OVERALL PRECISION ($\hat{S}_{rT}$): See Table 2.

ACCURACY: See Table 2.

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: 2-methoxyethanol, 2-ethoxyethanol, and 2-butoxyethanol

DESORPTION: 1 mL methylene chloride/methanol (95:5) in an ultrasonic bath for 30 minutes

INJECTION VOLUME: 1 µL

TEMPERATURE
-INJECTION: 225°C
-DETECTOR: 300°C
-COLUMN: 40°C (1 min) - 200°C (12°C/min)

CARRIER GAS: He, 2.5-3.0 mL/min

COLUMN: Capillary, fused silica, 30 m x 0.32-mm ID; crossbonded carbowax®-DA or equivalent

CALIBRATION: Solutions of analytes in desorption solvent

RANGE: (1) 2 to 387 µg (2) 2 to 373 µg
(3) 3 to 361 µg

ESTIMATED LOD: (1) 0.8 µg/sample (2) 0.7 µg/sample (3) 1.0 µg/sample

PRECISION ($\hat{S}_r$): (1) 0.024 (2) 0.022 (3) 0.048

APPLICABILITY: The working range for 2-methoxyethanol was 0.095 to 204 ppm (0.33 to 64.5 mg/m^3) for a 6-L sample volume, for 2-ethoxyethanol was 0.053 to 99 ppm (0.20 to 373 mg/m^3) for a 1-L sample, and for 2-butoxyethanol was 0.3 to 36.8 ppm (1.5 to 180 mg/m^3) for a 2-L sample.

INTERFERENCES: Any compounds having similar retention times as the analytes of interest.

OTHER METHODS: This method is an improved update of NMAM 1403, issue 2 (15 August 1994) [1]. NMAM 1403 (15 August 1994) previously combined and replaced methods S76 [2], S79 [2], and S361 [3]. Other less sensitive methods include OSHA 79 [4] and OSHA 83 [5].

REAGENTS:

1. Methylene Chloride, HPLC chromatographic grade.*
2. Methanol, HPLC chromatographic grade.*
3. Desorption solvent: methylene chloride (HPLC chromatographic grade) containing 5% methanol.
4. 2-methoxyethanol, reagent grade.
5. 2-ethoxyethanol, reagent grade.
6. 2-butoxyethanol, reagent grade.
7. Helium, purified and filtered.
8. Hydrogen, filtered.
9. Air, compressed, purified, 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 coconut shell charcoal (100 mg/50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and follows the back section. Tubes are commercially available from SKC, Inc.
2. Personal sampling pump, 0.01 to 0.05 L/min, connected with flexible tubing.
3. Gas chromatograph, FID, integrator, and capillary column (page 1403-1).
4. Autosampler vials, 11-mm glass with crimp caps.
5. Syringes, 10-µL, 25-µL, and 1-mL.
6. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Methylene chloride is a carcinogen [6]. Methanol is very flammable.

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 L (2-methoxyethanol); 1 to 6 L (2-ethoxyethanol); and 2 to 10 L (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. Place the glass wool preceding the front section into the vial containing the front sorbent section. Discard the remaining foam plugs.
6. Add 1.0 mL of the desorption solvent into each vial. Attach crimp caps to each vial.
7. Place the sample vials in an ultrasonic bath for 30 minutes.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards to cover the analytical range. If necessary, additional standards may be added to extend the calibration curve.
   a. Add known amounts of analytes to 10-mL volumetric flasks and dilute to the mark with solvent.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs µg analyte).
9. Determine desorption efficiency (DE) at least once for each lot of charcoal used for sampling in the calibration ranges (step 8).
   a. Prepare three tubes at each of five levels plus three media blanks.
   b. Inject a known amount of DE stock solution directly onto the front sorbent section of each charcoal tube with a microliter syringe.

c. Allow the tubes to air equilibrate for several minutes, then cap the ends of each tube and allow to stand overnight.
d. Desorb (steps 5-7) and analyze together with standards and blanks (steps 11 and 12).
e. Prepare a graph of DE vs µg analyte 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 the gas chromatograph according to manufacturer's recommendations and to conditions given on page 1403-1. Inject a 1-µL sample aliquot manually using the solvent flush technique or with an autosampler.
    NOTE: If peak area is above the linear range of the working standards, dilute with desorption solvent, reanalyze and apply the appropriate dilution factor in the calculations.
12. Measure peak areas.

CALCULATIONS:

13. Determine the 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 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):

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

NOTE: mg/L = mg/m^3

EVALUATION OF METHOD:

Initial Method Development Effort (Issues 1 and 2)

Methods S79 (2-methoxyethanol), S361 (2-ethoxyethanol), and S76 (2-butoxyethanol) were issued on February 14, 1975 [2, 7], March 17, 1978 [3, 8, 9], and February 14, 1975 [2, 7], 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.

Current Method Development Effort (Issue 3) [10]

Issue 3 is an update and improvement of NMAM 1403. Improvements in the method include the use of capillary column chromatography, lower LOD/LOQ values, a new 5 level desorption efficiency (DE) study at lower levels, and a storage stability study at 7, 14, and 30 days.

The average DE determined for 2-methoxyethanol was 97.8% (RSD = 1.0), for 2-ethoxythanol the DE was 100.2% (RSD = 1.2), and for 2-butoxyethanol the DE was 99.9% (RSD = 1.3). The average 30-day storage stability recovery at approximately 0.5x REL for 2-methoxyethanol was 103.8% (RSD = 1.4), for 2-ethoxyethanol was 105.0% (RSD = 1.6), and for 2-butoxyethanol was 82.6% (RSD = 1.4).

The precision and accuracy information listed in Table 2 was calculated by using the data from generated air samples [7, 8] and the analytical data from the current update [10].

REFERENCES:

[1] NIOSH [1994]. Alcohols IV: Method 1403, revised by George Williamson. In: Eller PM, ed. NIOSH Manual of Analytical Methods, 4th rev. ed. Cincinnati, OH: U.S. Department of Health Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113.
[2] NIOSH [1977]. 2-Butoxyethanol: Method S76, and 2-Methoxyethanol: Method S79. In: Taylor DG, ed. NIOSH Manual of Analytical Methods, 2nd ed., Cincinnati, OH: U.S. Department of Health, Education, and Welfare, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 77-157-B.
[3] NIOSH [1979]. 2-Ethoxyethanol: Method S361. In: Taylor DG, ed. NIOSH Manual of Analytical Methods, 2nd ed. Cincinnati, OH: U.S. Department of Health, Education, and Welfare, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 79-141.
[4] OSHA [1990]. Method 79. Salt Lake City, UT: Occupational Safety and Health Administration: Organic Methods Evaluation Branch, OSHA Analytical Laboratory.
[5] OSHA [1990]. Occupational Safety and Health Administration: Method 83. Salt Lake City, UT: Organic Methods Evaluation Branch, OSHA Analytical Laboratory.
[6] NIOSH Recommendations for Occupational Safety and Health, U.S. Department of Health and Human Services, (NIOSH) Publ. 92-100 (January 1992).
[7] Documentation of the NIOSH Validation Tests, S76 and S79, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
[8] Backup Data, S361, available as "Ten NIOSH Analytical Methods, Set 6," Order No. PB288-629 from NTIS, Springfield, VA 22161.
[9] NIOSH Research Report, Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).
[10] Pendergrass SM [1998]. Backup Data Report for Alcohols IV Method Development Effort, NIOSH/ARDB/ACS, January.
[11] NIOSH [1990]. User Check, DataChem Laboratories, NIOSH Sequence #6960-J,K (unpublished, August 15).

METHOD WRITTEN BY:

Issue 3

Stephanie M. Pendergrass, NIOSH/DART

Issue 1 & 2

George Williamson, NIOSH/ DPSE; methods originally validated under Contracts 99-74-75 and 210-76-0123.

TABLE 1. Exposure Limits and Physical Properties

Compound	OSHA (ppm)	NIOSH (ppm)	ACGIH (ppm)	mg/m^3 = 1ppm @ NTP	MW	BP (°C)	Density @ 20°C (g/mL)	VP @ 20°C, KPa (mm Hg)
2-methoxyethanol	25 (skin)	0.1 (skin)	5 (skin)	3.16	76.09	124	0.966	0.8 (6)
2-ethoxyethanol	200 (skin)	0.5 (skin)	5 (skin)	3.75	90.12	135	0.931	0.5 (4)
2-butoxyethanol	50 (skin)	5 (skin)	25 (skin)	4.91	118.17	171	0.902	0.11 (0.8)

TABLE 2. Method Evaluation

Compound	Overall Method[a]					Analytical Method[b]				Storage Stability	
	Range (mg/m^3)	Accuracy	Breakthrough @ 2 x OSHA PEL	Bias	Precision $S_r T$	Range studied (µg/sample)	LOD (µg/sample)	Ave. DE	Measurement Precision S_r	Levels (µg/sample)	Recovery (%)
2-methoxyethanol	44-160	0.41	128 L[c]	NS[e]	0.072	2-387	0.8	97.8	0.024	120.5	103.7
2-ethoxyethanol	340-1460	0.11	> 10 L[d]	NS	0.056	2-373	0.7	100.2	0.022	118.0	101.7
2-butoxyethanol	124-490	0.14	> 44 L[c]	NS	0.071	3-361	1.3	99.9	0.024	90.0	82

a) Generated air sample data in References 7 and 8.
b) Data for analytical method in Reference 10.
c) Testing done in dry air.
d) Testing done in 90% relative humidity.
e) NS = not significant