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

METHOD: 2007, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 May 1985**
Issue 2: 15 August 1994

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

PROPERTIES: Table 1

COMPOUNDS and SYNONYMS: 2-aminoethanol: ethanolamine; 2-hydroxyethylamine
2-dibutylaminoethanol: 2-n-dibutylaminoethanol; DBAE
2-diethylaminoethanol: 2-hydroxytriethylamine

SAMPLING

SAMPLER: SOLID SORBENT TUBE (silica gel, 300 mg/150 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 4 L
-MAX: 24 L

FIELD TREATMENT: add 20 µL conc. HCl to silica gel immediately after sampling

SHIPMENT: routine

SAMPLE STABILITY: at least 4 weeks @ 25 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: see EVALUATION OF METHOD [1,2]

BIAS: - 2.9%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.056 [2]

ACCURACY: ± 12.1%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: compounds above

DESORPTION: 2 mL 4:1 (v/v) methanol:water; stand 2 h with occasional shaking

INJECTION VOLUME: 3 µL

TEMPERATURE-INJECTION: 150 °C
-DETECTOR: 250 °C
-COLUMN: 90 °C, 3 min; 90 to 225 °C @ 16°/min, hold 6 min

CARRIER GAS: N_2 or He, 30 mL/min

COLUMN: 1.8 m x 2-mm ID silanized glass, 10% Carbowax 20 M + 2% KOH on 80/100 mesh Chromosorb WAW

CALIBRATION: solutions of analyte in 4:1 methanol:water containing 0.12 N HCl

RANGE: 0.1 to 6 mg per sample [2]

ESTIMATED LOD: 0.005 mg per sample [2]

PRECISION ($\hat{S}_r$): 0.026 @ 0.6 to 2.7 mg per sample [2]

APPLICABILITY: The working range is 5 to 300 mg/m^3 for each compound in a 20-L air sample. Water vapor does not significantly affect collection efficiency [3]. Sensitivity may be improved at least ten-fold by using a photoionization or nitrogen-selective detector.

INTERFERENCES: None identified [3]. The chromatographic column or separation conditions may be changed to circumvent interference problems. A DB-5 fused silica capillary column may be used.

OTHER METHODS: This revises P&CAM 270 [3], S140 [4], and Method 2007 (dated 5/15/85).

REAGENTS:

1. Eluent: Four parts methanol to one part distilled water (v/v).
   NOTE: Do not use deionized water which may contain formaldehyde.
2. Analyte, reagent grade.*
3. Alkalinizing solution: 0.20 N NaOH (8.0 g NaOH/L) in eluent.
4. HCl, conc. (38%, 12 N) (needed for field use).*
5. HCl, 0.12 N, in eluent. Dilute 10.0 mL conc. HCl with eluent to make 1 L solution.
6. Benzaldehyde.
7. Calibration stock solutions, 10 mg/mL, in 0.12 N HCl in eluent for each analyte.
8. Nitrogen or helium, purified.
9. Hydrogen, prepurified.
10. Air, filtered.
11. pH paper.
12. KOH, saturated solution.
13. Glass wool.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7-cm long, 8-mm OD, 6-mm ID, flame-sealed ends with plastic caps, containing two sections of 45/60 mesh chromatographic grade silica gel (front = 300 mg; back = 150 mg) separated by a plug of silylated glass wool. A silylated plug of glass wool is at each end of the tube. Pressure drop across the tube at 0.2 L/min airflow must be less than 3.2 kPa. Tubes are commercially available. Do not use sampling tubes constructed with metal parts or urethane foam plugs. Conc. HCl will react with these materials.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Syringe for dispensing HCl, 50-µL with glass barrel, PTFE-tipped plunger and inert (platinum or PTFE) needle (for field use).
4. Gas chromatograph, FID, integrator and column (page 2007-1).
5. Coated capillary injection port liner: Soak liner and small quantity of glass wool in saturated KOH. Air-dry liner and glass wool. Loosely pack glass wool in liner.
6. Vials, 2-mL, glass, PTFE-lined crimp caps.
7. Volumetric flasks, 10-mL.
8. Syringe, 10-µL, readable to 0.1 µL.
9. Micropipets, 10- to 100-µL.
10. Pipets, 0.5- and 2-mL, with pipet bulb.

SPECIAL PRECAUTIONS: The analytes are eye irritants [5]. Avoid skin contact or inhalation of conc. HCl.

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 0.2 L/min for a total sample size of 4 to 24 L.
   NOTE: If samples are to be eluted immediately or will be stored in a freezer for less than 14 days, the following step may be eliminated.
4. Immediately after sampling, stabilize the samples by adding exactly 20 µL conc. HCl to each section of silica gel in each sampler using a syringe.
   NOTE 1: The amount of HCl added must be measured carefully because the sample will be neutralized and made alkaline (step 9) for proper chromatography.
   NOTE 2: Iron impurities in silica gel will turn yellow upon addition of acid.
5. Cap the samplers. Pack securely for shipment.

SAMPLE PREPARATION:

6. Place the first glass wool plug and the front section of the sampler in a vial. Place the second plug and backup section in a separate vial.
7. Add 2.0 mL eluent to each vial. Attach cap to each vial.
   NOTE: If conc. HCl was not added to the silica gel after sampling, desorb the samples in 2.0 mL 0.12 N HCl in eluent. The acid increases desorption efficiency.
8. Allow to stand 2 h with occasional agitation.
9. Transfer a 0.5-mL aliquot of each sample to another vial. Add 0.5 mL alkalinizing solution and attach cap. Mix thoroughly. Check the solution pH with pH paper. If the solution pH is <9 add alkalinizing solution until solution pH is >9.
10. If 2-aminoethanol is present, repeat step 9 with another 0.5-mL aliquot. Add 10 µL benzaldehyde to the basic solution, mix thoroughly and let stand 20 min.
    NOTE: Benzaldehyde derivatizes 2-aminoethanol to 2-benzylideneaminoethanol which has a higher molecular weight, thereby decreasing the GC detection limit. Underivatized 2-aminoethanol can be determined by this method if the sample size is large.

CALIBRATION AND QUALITY CONTROL:

11. Calibrate daily with at least six working standards over the range 0.005 to 6 mg of each analyte.
    a. Add known amounts of analyte or calibration stock solution to 0.12 N HCl in eluent in 10-mL volumetric flasks and dilute to the mark.
    b. Treat the working standards with base and benzaldehyde as needed (steps 9 and 10).
    c. Analyze together with samples and blanks (steps 14 and 15).
    d. Prepare calibration graph (peak area vs. mg analyte).
12. Determine desorption efficiency (DE) at least once for each lot of silica gel used for sampling. 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 or calibration stock solution directly onto front sorbent section with a microliter syringe.
    c. Add 20.0 µL conc. HCl.
    d. Cap the tube. Allow to stand overnight.
    e. Desorb (steps 6 through 10) and analyze with working standards (steps 14 and 15).
    f. Prepare a graph of DE vs. mg analyte recovered.
13. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

14. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 2007-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
    NOTE 1: The temperature program separates all three compounds. Isothermal column conditions are 225, 150, and 90 °C for 2-aminoethanol, 2-dibutylaminoethanol and 2-diethylaminoethanol, respectively. The column packing degrades rapidly at 225 °C; keep operating time at a minimum near this temperature.
    NOTE 2: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze, and apply the appropriate dilution factor in calculations.
    NOTE 3: When using a capillary column system, use the split injection port liner coated with KOH to improve amine peak shape.
15. Measure peak area.

CALCULATIONS:

16. 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.
17. Calculate concentration, C, of analyte in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

All three compounds were evaluated under Method P&CAM 270 using a smaller bed of silica gel (front = 150 mg; back = 150 mg) than given above [3]. Desorption efficiencies for 0.1 mg analyte were found to be 0.97 for 2-aminoethanol, 0.85 for 2-diethylaminoethanol, and 0.93 for 2-dibutylaminoethanol [3]. Only 2-diethylaminoethanol was evaluated under Method S140 using the suggested sorbent tube. In all cases, laboratory testing was performed with spiked samples and generated atmosphere [1,2,6]. All analytes were stable on silica gel up to four weeks when stabilized with acid. Results were:

Compound	Method	Range Studied (mg/m^3)	Sample size (L)	Precision	
				Measurement ($\bar{S}_r$)	Overall ($\hat{S}_{rT}$)
2-aminoethanol	P&CAM 270 [3]	30.4 to 63.6	30	<0.070	0.057
2-dibutylaminoethanol	P&CAM 270 [3]	unknown	unknown	unknown	unknown
2-diethylaminoethanol	S140 [4]	25 to 113	24	0.026	0.056

Method S140 [4] was issued on January 19, 1979, and validated over the range 25 to 113 mg/m^3 at 23 °C and 762 mm Hg using 24-L samples [2,7]. Coast Engineering Laboratory 40/60 mesh silica gel was the collecting medium. Average recovery was 97.1%, representing a non-significant bias. The concentration of 2-diethylaminoethanol was independently verified using midget bubblers containing 15 mL 2% HCl. Desorption efficiency was 0.964 in the range 0.6 to 2.3 mg per sample. Breakthrough (5% on back section) was not achieved after 5.3 h when sampling an atmosphere containing 88 mg/m^3 2-diethylaminoethanol at 0.2 L/min at 82% relative humidity.

REFERENCES:

[1] Wood, G. O. and J. W. Nickols. Development of Air-Monitoring Techniques Using Solid Sorbents, October 1, 1976 - December 31, 1977, Progress Report LA-7295-PR, Los Alamos Scientific Laboratory, Los Alamos, NM, NIOSH IA-77-12 (1978).

[2] Backup Data Report No. S140, prepared under NIOSH Contract 210-76-0123 (NIOSH, unpublished, 1979); Report on NIOSH Sequence #5162 (unpublished, April 2, 1986).

[3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, P&CAM 270, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).

[4] Ibid., Vol. 5, S140, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).

[5] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, Diethylaminoethanol and Ethanolamine, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.

[6] Wood, G. O., R. G. Anderson and J. W. Nickols. Sampling and Analysis of Aminoethanols in Air, Report LA-UR-77-1398, Industrial Hygiene Group, Los Alamos Scientific Laboratory, Los

Alamos, NM (1977) (presented at the 1977 American Industrial Hygiene Conference, May 1977, New Orleans, LA).

[7] 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).

METHOD REVISED BY:

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TABLE 1. STRUCTURAL FORMULAS, MOLECULAR WEIGHTS, PERMISSIBLE EXPOSURE LIMITS AND PROPERTIES.

Compound	CAS RTECS	Formula	MW	mg/m^3 = 1 ppm @ NTP	OSHA NIOSH ACGIH (ppm)	BP (°C)	VP @ 20 °C	Density (g/mL @ 20 °C)
2-aminoethanol	141-43-5 KJ5775000	$HOCH_2CH_2NH_2$	61.08	2.50	3 TWA 3 TWA; 6 STEL 3 TWA; 6 STEL	171	48 kPa (470 ppm)	1.0180
2-dibutylamino-ethanol	102-81-8 KK3850000	$(C_4H_9)_2N(CH_2)_2OH$	173.30	7.09	no standard 2 TWA (skin) 2 TWA (skin)	228	not available	0.859
2-diethylamino-ethanol	100-37-8 KK5075000	$(C_2H_5)_2N(CH_2)_2OH$	117.19	4.79	10 TWA (skin) 10 TWA (skin) 10 TWA (skin)	163	130 kPa (1300 ppm)	0.8921