(1) 2-(DIMETHYLAMINO)ETHANOL 2561
(2) 1-DIMETHYLAMINO-2-PROPANOL

FORMULAS: TABLE 1 MW: TABLE 1 CAS: TABLE 1 RTECS: TABLE 1

METHOD: 2561, Issue 1 **EVALUATION: PARTIAL** **Issue 1: 15 March 2003**

OSHA: no REL
NIOSH: no PEL
ACGIH: no TLV

PROPERTIES: Table 1

COMPOUNDS: (1) 2-(dimethylamino)ethanol: dimethylethanol amine
(2) 1-dimethylamino-2-propanol: dimethylisopropanolamine

SAMPLING

SAMPLER: SOLID SORBENT TUBE (XAD-7, 60 mg/30 mg)

FLOW RATE: 0.02 to 0.1 L/min

VOL – MIN: 10 L
– MAX: 24 L

SHIPMENT: Cold

SAMPLE STABILITY: Stable stored in freezer

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: Not studied

BIAS: Not determined

OVERALL PRECISION (S_{rt}): Not determined

ACCURACY: Not determined

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: Compounds above

DESORPTION: 1 mL methanol, 60 min

INJECTION VOLUME: 1 µL

TEMPERATURE
-INJECTION: 250 °C
-DETECTOR: 300 °C
-COLUMN: 70 °C for 6 min then up to 200 °C for 4 minutes at a rate of 15 °C per minute

CARRIER GAS: Helium, 1-2 mL/min

COLUMN: Capillary, fused silica, 30-m x 0.25-mm ID; 1.0-µm film dimethylpolysiloxane, DB-1 or equivalent

CALIBRATION: Solutions of compounds in methanol

RANGE: 0.004 to 2 mg per sample [1, 2, 3]

ESTIMATED LOD: See Table 2

PRECISION ($\bar{S}_r$): See Table 2

APPLICABILITY: This method can be used for simultaneous analysis of both amino compounds. The method was field tested for almost 300 samples containing both compounds with little or no breakthrough [2, 3] . Up tp 4 mg of dimethylethanolamine (no isopropanolamine present) and up to 1200 µg dimethylisopropanol (140 µg dimethylethanolamine present) were reported on the field samples with little breakthrough. For higher capacity, a larger XAD-7 sorbent bed tube can be used.
NOTE: During method development, a capillary Rtx-5Amine (cross-linked 5% diphenyl/95% dimethyl polysiloxane) column with deactivated injection port liner was also used successfully.

INTERFERENCES: None identified.

OTHER METHODS: This method may be applicable to other amino alcohols such as those in method 2007 [4].

REAGENTS:

1. Eluent: methanol* (chromatographic grade).
2. Analytes, 99+% grade*.
3. Helium, purified.
4. Hydrogen, prepurified.
5. Air, compressed, filtered.

***** See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7-cm long, 6-mm OD, flame-sealed ends, containing two sections of XAD-7 resin (front = 60 mg; back = 30 mg) separated by glass wool. Pressure drop across the tube at 0.1 L/min. airflow must be less than 3.2 kPa. Tubes are commercially available (SKC, Inc. #226-94).
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Refrigerant, bagged ("Blue Ice" or equivalent).
4. Gas chromatograph, FID, integrator and column (page 2561-1).
5. Vials, glass, 2-mL, PTFE-lined crimp caps.
6. Syringes, 10-µL to 500-µL.
7. Volumetric flasks, 10-mL.
8. Pipet, volumetric, 1-mL, with pipet bulb or repipet.

SPECIAL PRECAUTIONS: Methanol is toxic and flammable. The analytes are eye irritants [2]. Wear appropriate protective clothing and work with these compounds in a well ventilated 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 0.02 and 0.1 L/min for a total sample size of 10 to 24 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment with bagged refrigerant.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. The initial glass wool plug may be discarded.
6. Add 1.0 mL of methanol eluent to each vial. Attach crimp cap to each vial.
7. Allow to stand 60 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate with at least six working standards over the range 4 to 4000 µg analyte per sample.
   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 vs. µg analyte).
9. Determine desorption efficiency (DE) at least once for each lot of XAD-7 used for sampling in the calibration range (step 8). Prepare three tubes at each of five concentrations plus three media blanks.
   a. Remove and discard back sorbent section of a 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 ensure 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 2561-1. Inject sample aliquot with autosampler, or manually using solvent flush technique.
    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, 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.
    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):

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

EVALUATION OF METHOD:

Current Validation
This method was developed in response to a request for a method to use in a field study. The method for dimethylethanolamine and dimethylisopropanolamine was validated using analytes fortified on XAD-7 sorbent tubes (SKC, Inc. #226-94, Lot 1994). Desorption efficiency (DE) and precision are listed in Table 2.

Storage stability studies were done at approximately 100 µg each analyte/sample. The samples were stored for up to 5 weeks in the freezer (-4°C). The recovery for dimethylethanolamine (110.9 µg/sample) was 98.4% and for dimethylisopropanolamine (104.6 µg/sample) was 100.2%.

REFERENCES:

[1] Grote AA[2002]. NIOSH Backup Data Report for NIOSH method 2561. National Institute for Occupational Safety and Health, DART/CEMB, Cincinnati, OH. (unpublished report).
[2] NIOSH [2002]. Evaluation of Visual Disturbances Related to Amine Exposure. Cincinnati, OH: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 2001-01444-2867.
[3] DataChem Laboratories [2001]. NIOSH Analytical Reports for NIOSH Sequence 9675-CA through -CH. Cincinnati, OH: National Institute for Occupational Safety and Health, DART/CEMB. (unpublished, May).
[4] NIOSH [1994]. Aminoethanol Compounds I: Method 2007. In: Eller PM, Cassinelli ME, eds. NIOSH Manual of Analytical Methods, 4nd ed. Cincinnati, OH: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Istitute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113.

METHOD WRITTEN BY:

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TABLE 1. Structural Formulas, Molecular Weights, and Properties

Compound	CAS	RTECS	Formula	MW	mg/m³= 1 ppm	BP (ºC)	VP (mm Hg)	Density (g/mL)
2-(dimethylamino)ethanol	108-01-0	KK6125000	$HOCH_2CH_2N(CH_3)_2$	89.14	0.274	133	6.12	0.887
1-dimethylamino-2-propanol	108-16-7	UB3150000	$(CH_3)_2NCH_2CH(OH)CH_3$	103.17	0.237	124	8.0	0.837

TABLE 2. Current Validation

Compound	Range Studied (µg / sample)	LOD (µg / sample)	Average DE[1]	Measurement Precision ($\hat{S}_r$)
2-(dimethylamino)ethanol	22-554	4	0.930	0.026
1-dimethylamino-2-propanol	21-523	4	0.894	0.029

[1] Averaged over mass range shown.