$CH_3(CH_2)_7SH$ MW: 146.29 CAS: 111-88-6 RTECS: None

METHOD: 2510, Issue 2 **EVALUATION: FULL** **Issue 1: 15 February 1984**
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OSHA : no PEL
NIOSH: C 0.5 ppm/15 min
ACGIH: no TLV
(1 ppm = 5.98 mg/m^3 @ NTP)

PROPERTIES: liquid; d 0.843 g/mL @ 20 °C; BP 199.1 °C; MP - 49.2 °C; VP 0.026 kPa (1.55 mm Hg; 2040 ppm) @ 37.7 °C

SYNONYMS: n-octanethiol; octyl mercaptan; 1-mercaptooctane; octylthiol.

SAMPLING

SAMPLER: SOLID SORBENT TUBE (Tenax-GC, 100 mg/50 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 1 L @ 0.5 ppm
-MAX: 15 L

SHIPMENT: routine

SAMPLE STABILITY: at least 8 days @ 25 °C

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.32 to 6.55 mg/m^3 [1] (8.5-L samples)

BIAS: - 0.013 [1]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.083 [1]

ACCURACY: ± 17.6%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, SULFUR-SPECIFIC FPD

ANALYTE: 1-octanethiol (sulfur)

DESORPTION: 2 mL acetone; 45 min ultrasonic agitation

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 160 °C
-DETECTOR: 200 °C
-COLUMN: 115 °C

CARRIER GAS: N_2 or He, 50 mL/min

COLUMN: 1.8 m x 4-mm ID glass; 3% OV-1 on 100/120 mesh Gas-Chrom Q or equivalent

CALIBRATION: 1-octanethiol in acetone

RANGE: 3 to 64 µg per sample

ESTIMATED LOD: 0.2 µg per sample

PRECISION ($\bar{S}_r$): 0.030 [1]

APPLICABILITY: The working range is 0.05 to 1 ppm (0.3 to 6 mg/m^3) for a 10-L air sample.

INTERFERENCES: Presence of other volatile compounds may interfere with collection of 1-octanethiol or reduce capacity of sorbent tube.

OTHER METHODS: None evaluated by NIOSH.

REAGENTS:

1. Acetone*.
2. 1-Octanethiol, ≥97% purity.*
3. Calibration stock solution, 0.843 µg/µL. Dissolve 10 µL 1-octanethiol in 10 mL acetone. Prepare fresh daily.
4. Nitrogen or helium, purified.
5. Hydrogen, prepurified.
6. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 8.5 cm long, 6-mm OD, 4-mm ID, sealed with PTFE tape and plastic caps containing front (100-mg) and rear (50-mg) sections of 60/80 mesh Tenax-GC, separated and held in place by three silanized glass wool plugs. (Tenax-GC pretreatment: Sohxlet extraction for 6 hrs with acetone/methanol, 6 hrs with methanol, then drying under vacuum.) Typical pressure drop across tube is 2.4 kPa at 0.05 L/min flow. Similar commercially available tubes, e.g., SKC #226-35-03, are acceptable.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Ultrasonic bath.
4. Gas chromatograph, sulfur-specific FPD, integrator and column (page 2510-1).
5. Vials, glass, 2- and 5-mL, crimp seals or caps, PTFE-lined silicone rubber septa.
6. Pipets, 1-, 2- and 5-mL, with pipet bulb.
7. Syringes, 10- and 100-µL.

SPECIAL PRECAUTIONS: Acetone is highly flammable (flash point = -18 °C).

Thiols are malodorous [2]. Prepare samples and standards in 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 0.01 and 0.2 L/min for a total sample size of 1 to 15 L.
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 2.0 mL acetone to each vial. Attach crimp cap to each vial.
7. Place vials in an ultrasonic bath for 45 min.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 1 to 60 µg 1-octanethiol per sample.
   a. Add known amounts of calibration stock solution to acetone 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 (square root of peak height vs. µg 1-octanethiol).

9. Determine desorption efficiency (DE) at least once for each batch of Tenax-GC 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 calibration stock solution 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. µg 1-octanethiol 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 2510-1. Inject sample aliquot manually using solvent flush technique or with autosampler. Under these conditions, the retention time of 1-octanethiol is about 3.0 min.
    NOTE: If peak area is above the linear range of the working standards, dilute with acetone, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak height.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE) of 1-octanethiol 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 1-octanethiol in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Analytical precision was determined by analyzing spiked sampling media [1]. The average desorption efficiency was 0.94. The breakthrough volume was greater than 15 L for a concentration of 14.6 mg/m^3 at 40 °C and 80% RH. Overall precision, bias and storage stability were determined by sampling and analyzing generated atmospheres. Generated concentrations were independently verified [1].

REFERENCES:

[1] Spafford, R. B., H. K. Dillon, D. H. Love, and W. K. Fowler. Analytical Methods Evaluation and Validation for Vinylidene Fluoride, Vinyl Bromide, Vinyl Fluoride, Benzenethiol, and n-Octanethiol: Research Report for n-Octanethiol and Benzenethiol, NIOSH Contract No. 210-79-0100, Southern Research Institute, Birmingham, AL, available from NTIS, PB 92-128-677, Springfield, VA 22161 (1982).

[2] Criteria for a Recommended Standard...Occupational Exposure to n-Alkane Mono Thiols, Cyclohexanethiol, and Benzenethiol, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-123 (1978).

METHOD WRITTEN BY:

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