METHYLCYCLOHEXANOL 1404

$CH_3C_6H_{10}OH$ MW: 114.19 CAS: 25639-42-3 RTECS: GW0175000

METHOD: 1404, Issue 1 **EVALUATION: FULL** **Issue 1: 15 August 1994**

OSHA : 100 ppm
NIOSH: 50 ppm
ACGIH: 50 ppm
(1 ppm = 4.67 mg/m^3 @ NTP)

PROPERTIES: liquid; BP 155-180 °C; d 0.92 g/mL @ 20 °C; VP 0.2 kPa (1.5 mm Hg) @ 20 °C

SYNONYMS: hexahydromethylphenol; hexahydrocresol

SAMPLING

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

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 1 L @ 100 ppm
-MAX: 15 L

SHIPMENT: routine

SAMPLE STABILITY: at least 7 days @ 25 °C

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 215 to 920 mg/m^3 [1] (12-L samples)

BIAS: ± 2.9% [1]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.063 [1]

ACCURACY: ± 14.0%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: methylcyclohexanol

DESORPTION: 1.0 mL methylene chloride

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTOR: 230 °C
-DETECTOR: 230 °C
-COLUMN: 140 °C

COLUMN: 10% FFAP on 80/100 mesh Chromosorb W, 12 ft x 1/8 in stainless steel

CALIBRATION: standard solutions of methylcyclohexanol in methylene chloride

RANGE: 0.55 to 17 mg per sample [1]

ESTIMATED LOD: 0.02 mg per sample [2]

PRECISION ($\bar{S}_r$): 0.019 @ 2.7 to 11.3 mg per sample [1]

APPLICABILITY: The working range is 10 to 300 ppm (46 to 1400 mg/m^3) for a 12-L air sample. Methylcyclohexanol is usually present as a 50:50 mixture of meta- and para- isomers.

INTERFERENCES: None identified.

OTHER METHODS: This revises Method S374 [2].

REAGENTS:

1. m-Methylcyclohexanol *, ACS reagent grade.
2. p-Methylcyclohexanol *, ACS reagent grade.
3. Methylene chloride *, distilled in glass.
4. Nitrogen, purified.
5. Hydrogen, prepurified
6. Air, filtered, compressed.
7. Calibration stock solution, 14.8 mg/mL.
   Add 80 µL m-methylcyclohexanol and 80 µL p-methylcyclohexanol to 10 mL methylene chloride.

* See Special Precautions

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends with plastic caps, 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 25 mm/Hg. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 mL/min, with flexible polyethylene or PTFE tubing.
3. Gas chromatograph with a FID detector, recorder, integrator and column (page 1404-1).
4. Vials, 2-mL, PTFE-lined crimp caps.
5. Syringe, 10-µL and other sizes as needed,

SPECIAL PRECAUTIONS: Methylcyclohexanol can irritate the eyes and upper respiratory tract and can cause liver and kidney damage. Methylene chloride is an irritant, can be absorbed through the skin, and is a suspect carcinogen [3]. Prepare samples in a 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. Pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sections of the sampler in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL methylene chloride to each vial. Cap each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards.
   a. Add known amounts of methylcyclohexanol to methylene chloride in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain concentrations in the range 0.02 to 10 mg/mL.
   b. Analyze with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (area vs. mg of methylcyclohexanol).
9. Determine desorption efficiency (DE) at least once for each lot of sorbent used for sampling in the range of interest (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 (2 to 20 µL) of a 50:50 mixture of methylcyclohexanol isomers 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 with working standards (steps 11 and 12)
e. Prepare a graph of DE vs. mg methylcyclohexanol 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 1404-1. 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 an aliquot of the desorbed liquid with methylene chloride, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE), of methylcyclohexanol 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 methylcyclohexanol 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:

This method was evaluated over the range 215 to 920 mg/m^3 and had an overall sampling and measurement precision, $\hat{S}_{rT}$, of 0.063. Concentrations obtained for seventeen generated samples averaged 2.9% higher than the independently determined concentration. Average recoveries of 2.71 mg of meta- and para- isomers of methylcyclohexanol was determined at 942 mg/m^3 while sampling 0.19 L/min. Breakthrough occurred in 116 min corresponding to a capacity of 20.8 mg per sample. Sample stability during storage was evaluated at 5.45 mg per sample. Samples showed 99.8% recovery after eight days of storage at ambient conditions compared to one-day old samples.

REFERENCES:

[1] Backup Data Report for Methylcyclohexanol, prepared under NIOSH Contract 210-76-0123 (1977).
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 4, S374, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
[3] NIOSH Pocket Guide to Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 90-117 (1990), available as GPO Stock #1990-751-238 from Superintendent of Documents, Washington, DC 20402.

METHOD REVISED BY:

M.J. Seymour, NIOSH/DPSE, originally evaluated under NIOSH Contract 210-76--123 (1977).