METHYL IODIDE

CH_3I MW: 141.94 CAS: 74-88-4 RTECS: PA 9450000

METHOD: 1014, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 May 1985**
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OSHA : 5 ppm (skin)
NIOSH: 2 ppm (skin; suspect carcinogen)
ACGIH: C 0.2 (skin; suspect carcinogen)
(1 ppm = 5.80 mg/m^3 @ NTP)

PROPERTIES: liquid; d 2.28 g/mL @ 25 °C;
BP 42.5 °C; MP -66 °C; VP 50 kPa
(375 mm Hg; 49% v/v) @ 20 °C;
not combustible

SYNONYMS: iodomethane

SAMPLING

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

FLOW RATE: 0.01 to 1 L/min

VOL-MIN: 15 L @ 5 ppm
-MAX: 50 L

SHIPMENT: routine

SAMPLE STABILITY: unknown

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 17 to 52 mg/m^3 [1]
(53-L samples)

BIAS: - 10.0%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.070 [1]

ACCURACY: ± 20.03%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: methyl iodide

DESORPTION: 1 mL toluene, stand 8 h

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 200 °C
-DETECTOR: 300 °C
-COLUMN: 190 °C

CARRIER GAS: nitrogen, 30 mL/min

COLUMN: 3 m x 3.2-mm OD stainless steel, packed with Chromosorb 101

CALIBRATION: solutions of methyl iodide in toluene

RANGE: 0.5 to 5 mg per sample

ESTIMATED LOD: 0.01 mg per sample [2]

PRECISION ($\bar{S}_r$): 0.045 @ 0.7 to 2.8 mg per sample [2]

APPLICABILITY: The working range of this method is 10 to 100 mg/m^3 (1.7 to 17 ppm) for a 50-L air sample. High humidity during sampling will decrease breakthrough volume.

INTERFERENCES: None identified.

OTHER METHODS: This revises Method S98 [3].

REAGENTS:

1. Toluene, reagent grade (distilled in glass).
2. Methyl iodide, reagent grade.*
3. Calibration stock solution, 250 mg/mL. Prepare in toluene.
4. Nitrogen, purified.
5. Hydrogen, prepurified.
6. Air, compressed, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7-cm long, 6-mm OD, 4-mm ID, flame-sealed ends and plastic caps, containing two sections of 20/40 mesh 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 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 1014-1).
4. Vials, glass, 2-mL, PTFE-lined caps.
5. Syringes, 10-µL, readable to 0.1 µL, 25-, 50- and 100-µL.
6. Volumetric flasks, 10-mL.
7. Pipet, volumetric, 1-mL with pipet bulb.

SPECIAL PRECAUTIONS: Methyl iodide is an acute neurotoxin and may produce severe narcosis. It is a suspected carcinogen [4,5].

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 1 L/min for a total sample size of 15 to 50 L.
4. Cap the samples. Pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials.
6. Add 1.0 mL toluene to each vial. Attach cap to each vial.
7. Allow to stand overnight with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 0.01 to 5 mg methyl iodide per sample.
   a. Add known amounts of calibration stock solution, or a dilution thereof, to toluene in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 10 through 12).
   c. Prepare a calibration graph by plotting peak area vs. mg methyl iodide.
9. Determine desorption efficiency (DE) at least once for each lot of charcoal 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 (1 to 20 µL) of calibration stock solution or a dilution thereof 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 methyl iodide recovered.

10. Analyze three quality control blind spikes or three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph operating parameters according to manufacturer's recommendations and those given on page 1014-1. 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 with toluene, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. 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.
14. Calculate the concentration, C, of analyte in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Method S98 was issued on April 11, 1975 [3] and evaluated using atmospheres containing methyl iodide generated in dry air [1]. The samples were analyzed by gas chromatography along with spiked samplers to correct for desorption efficiency (DE) which was determined to be 0.85, 0.91 and 0.92 at 0.7, 1.4 and 2.8 mg methyl iodide, respectively, from 100 mg of SKC Lot 105 activated coconut shell charcoal. Long-term stability of methyl iodide on the charcoal has not been investigated.

When an atmosphere containing 52 mg/m ^{3} methyl iodide in dry air was sampled at a rate of 1 L/min for 4 hrs, the concentration of methyl iodide in the effluent was 2% of that in the influent, indicating near breakthrough. Therefore, the adsorptive capacity of this lot of charcoal is approximately 12.5 mg methyl iodide, and the breakthrough volume is approximately 240 L under the conditions investigated.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S98, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.
[2] UBTL, Inc., NIOSH Sequence #3428-M (unpublished, April 21, 1982).
[3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S98, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
[4] NIOSH Current Intelligence Bulletin 43, Monohalomethanes, U.S. Department of Health and Human Services, Publ. (NIOSH) 84-117 (1984).

[5] NIOSH-OSHA Occupational Health Guidelines for Chemical Hazards, 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.

METHOD REVISED BY:

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