$C_9H_{14}O$ MW: 138.21 CAS: 78-59-1 RTECS: GW7700000

METHOD: 2508, Issue 2 **EVALUATION: FULL** **Issue 1: 15 May 1984**
Issue 2: 15 August 1994

OSHA : 25 ppm
NIOSH: 4 ppm; Group III Pesticide
ACGIH: C 5 ppm
(1 ppm = 5.65 mg/m^3 @ NTP)

PROPERTIES: liquid; d 0.923 g/mL @ 25 °C; BP 213 °C; VP 26 kPa (0.2 mm Hg; 260 ppm) @ 20 °C; explosive range 0.8 to 3.8% v/v in air

SYNONYMS: 3,5,5-trimethyl-2-cyclohexen-1-one

SAMPLING

SAMPLER: SOLID SORBENT TUBE (petroleum-based charcoal, 100 mg/50 mg)

FLOW RATE: 0.01 to 1 L/min

VOL-MIN: 2 L @ 25 ppm
-MAX: 25 L

SHIPMENT: routine

SAMPLE STABILITY: at least 7 days @ 25 °C

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 67 to 283 mg/m^3 [1] (180-L samples)

ACCURACY: ± 15.3%

BIAS: 5.0%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.059 [1]

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: isophorone

EXTRACTION: 1 mL CS_2; stand 30 min

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTOR: 200 °C
-DETECTOR: 250 °C
-COLUMN: 160 °C

CARRIER GAS: N_2 or He, 30 mL/min

COLUMN: 4 m x 3-mm glass, 10% SP2100/0.1% Carbowax 1500 on 100/120 Supelcoport or equivalent

CALIBRATION: isophorone in CS_2

RANGE: 0.2 to 10 mg per sample [2]

ESTIMATED LOD: 0.02 mg per sample

PRECISION ($\bar{S}_r$): 0.033 [1,2]

APPLICABILITY: The working range for this method is 0.35 to 70 ppm (2 to 400 mg/m ^{3}) for a 12-L air sample. High humidity will greatly decrease breakthrough volume.

INTERFERENCES: None identified. Alternate columns may be used, e.g., 10% SP-1000 or DB-2 fused silica capillary column.

OTHER METHODS: This is Method S367 with a different column recommended [2].

REAGENTS:

1. Carbon disulfide, chromatographic grade.*
2. Isophorone.*
3. Nitrogen, prepurified.
4. Hydrogen, prepurified.
5. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of 20/40 mesh petroleum-based charcoal (100 mg front/50 mg back) 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 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 2508-1).
4. Vials, 2-mL, PTFE-lined caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Volumetric flasks, 10-mL.
7. Pipet, 1-mL, with pipet bulb.

SPECIAL PRECAUTIONS: CS_2 is toxic and acute fire and explosion hazard (flash point = -30 °C). Work with it only in a hood.

Isophorone is a lachymator [3]; work with it should be done in a hood and caution should be taken while it is being used.

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 1.0 L/min for a total sample size of 2 to 25 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 1.0 mL CS_2 to each vial. Attach crimp cap to each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 0.02 to 10 mg isophorone per sample.
   a. Add known amounts of isophorone to CS_2 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 (peak area vs. mg isophorone).

9. Determine desorption efficiency (DE) at least once for each batch 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 of isophorone 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 isophorone 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 2508-1. 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 CS_2, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of isophorone 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 isophorone 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 S367 [2] was validated over the range 69 to 304 mg/m ^{3} using a 12-L sample [1]. Overall precision, $\hat{S}_{rT}$, was 0.059 with an average recovery of 104.9%, representing a non-significant bias. The concentration was independently verified by a direct hydrocarbon analyzer. Desorption efficiency was 0.860 in the range of 0.849 to 3.40 mg per sample. No breakthrough occurred when an atmosphere containing 283 mg/m ^{3} was sampled for 240 min at a rate of 0.19 L/min. At this time the breakthrough test was discontinued. Samples are stable for at least one week at room temperature.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S367-1 to S367-6, U.S. Department of Health and Human Services, Publ. (NIOSH) 77-185 (1977).
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 3, S367, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
[3] Criteria for a Recommended Standard...Occupational Exposure to Ketones, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-173 (1978).

METHOD REVISED BY:

Ardith Grote, NIOSH/DPSE; S367 originally validated under NIOSH Contract CDC-99-74-45.