

ISOPROPYL ACETATE

1454



MW: 102.14

CAS: 108-21-4

RTECS: AI4930000

METHOD: 1454, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 August 1994

OSHA : 250 ppm
 NIOSH: no REL
 ACGIH: 250 ppm; STEL 310 ppm
 (1 ppm = 4.18 mg/m³ @ NTP)

PROPERTIES: colorless liquid; BP 89.4 °C; d 0.87 g/mL
 @ 20 °C; miscible in most organic
 solvents; VP 6.33 kPa (47.5 mm Hg)
 @ 20 °C

SYNONYMS: methyl isobutyl acetate

SAMPLING		MEASUREMENT	
SAMPLER:	CHARCOAL TUBE (coconut shell charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.02 to 0.2 L/min	ANALYTE:	isopropyl acetate
VOL-MIN:	0.1 L @ 250 ppm	DESORPTION:	carbon disulfide, 1 mL
-MAX:	9 L	INJECTION	
SHIPMENT:	routine	VOLUME:	5 µL
SAMPLE		TEMPERATURE-INJECTION:	225 °C
STABILITY:	not documented	-DETECTOR:	250 °C
BLANKS:	2 to 10 field blanks per set	-COLUMN:	60 °C - isothermal
ACCURACY		CARRIER GAS:	helium, 30 mL/min
RANGE STUDIED:	446 to 1870 mg/m ³ [1] (8-L samples)	COLUMN:	10 ft x 1/8-inch stainless steel, packed with 5% FFAP on 100/120 mesh Supelcoport [1]
BIAS:	1.1%	CALIBRATION:	standard solutions of isopropyl acetate in carbon disulfide
OVERALL PRECISION (\hat{S}_{rT}):	0.067 [1]	RANGE:	0.05 to 15 mg per sample [1]
ACCURACY:	±29.4%	ESTIMATED LOD:	0.01 mg per sample [1]
		PRECISION (\hat{S}_r):	0.007 @ 3.6 to 15 mg per sample [1]

APPLICABILITY: The working range is 1.5 to 450 ppm (6 to 1870 mg/m³) for an 8-L air sample.

INTERFERENCES: None identified.

OTHER METHODS: This is NIOSH Method S50 in a revised format [1]. Operational modifications [2] to this method are as follows: column - 30 m x 0.3-mm I.D. fused silica WCOT capillary coated with 1.0-µm DB-1. Column temperature - 45 °C for 16 min, and programmed at 16 °C/min to 150 °C and held for 8 min. Carrier gas - He, with split ratio of 1:10. Desorption - 1 h or more in 1 mL carbon disulfide with 1 µL/mL p-cymene as internal standard.

REAGENTS:

1. Isopropyl acetate, ACS reagent grade.
2. Eluent: carbon disulfide*, chromatographic grade.
NOTE: Add 0.1% (v/v) undecane or p-cymene as an internal standard, if desired.
3. Undecane or p-cymene, ACS reagent grade.
4. Standard solutions: Isopropyl acetate in carbon disulfide in the range of 0.01 to 15 mg per sample.
5. Helium, purified.
6. Hydrogen, prepurified.
7. Air, filtered, compressed.

* 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 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 must be less than 3.4 kPa at 1 L/min. Tubes are commercially available.
2. Personal sampling pump, 0.02 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 1454-1).
4. Vials, 2-mL glass, PTFE-lined caps.
5. Syringe, 10- μ L, readable to 0.1 μ L.
6. Pipet, 1-mL, readable to 0.1 mL.
7. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C); all work done with it must be performed in a fume 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 0.2 L/min for a total sample size of 0.1 to 9 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 CS₂ to each container and cap tightly. For the internal standard method, the 1.0 mL CS₂ will contain a known amount of the chosen internal standard.
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 0.01 to 15 mg analyte per sample.
 - a. Add known amounts of analyte to CS₂ 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 isopropyl acetate). If internal standard is used, plot ratio of peak area of analyte/peak area of internal standard vs. mg isopropyl acetate.
9. Determine desorption efficiency (DE) at least once per year for each lot of charcoal 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 media blank sampler.
 - b. Inject a known amount of analyte or of a standard solution of analyte in CS₂ 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 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 1454-1. 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 eluent, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass mg (corrected for DE) of isopropyl acetate 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 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:

This method was evaluated over the range 446 to 1870 mg/m³ at 23 °C and pressure of 770 mm Hg using 8-L samples [3]. Overall sampling and measurement precision, \hat{S}_{rT} , was 0.067, with average recovery of 96%. A sample stability evaluation has not been performed, nor has the sample stability been determined. The accuracy of $\pm 29.4\%$ does not meet the NIOSH criterion of $\pm 25\%$.

REFERENCES:

- [1] NIOSH Manual of Analytical Methods, 3rd ed., V. 2, S50, U.S. Department of Health and Human Services, (1984).
- [2] Analytical Report for Organics on Charcoal Tubes, DataChem Sequence #3031-S, Unpubl. NIOSH (1981)
- [3] Documentation of NIOSH Validation Tests, NIOSH Contract No. CDC-99-74-45. Department of Health, Education and Welfare. (NIOSH) Publication 77-185 (1977).

METHOD REVISED BY:

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