ISOPROPYL ACETATE 1460

$CH_3COOCH(CH_3)_2$ MW: 102.14 CAS: 108-21-4 RTECS: AI4930000

METHOD: 1454, Issue 1 **EVALUATION: PARTIAL** **Issue 1: 15 March 2003**

OSHA : 250 ppm
NIOSH: no REL
ACGIH: 250 ppm; STEL 310 ppm

(1 ppm = 4.18 mg/m^3 @ 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)

SYNONYMS: 2-Propyl Acetate

SAMPLING

SAMPLER: SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg, Lot 2000)

FLOW RATE: 0.02 to 0.2 L/min

VOL -MIN: 0.1 L @ 250 ppm
-MAX: 9 L

SHIPMENT: Routine

SAMPLE STABILITY: 30 Days @ 5°C

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: Not Studied

BIAS: Not Determined

OVERALL PRECISION ($\hat{S}_{rT}$): Not Determined

ACCURACY: Not Determined

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: Isopropyl acetate

DESORPTION: 1 mL of carbon disulfide containing 1% methanol

INJECTION VOLUME: 1 µL

TEMPERATURE
-INJECTION: 250 °C
-DETECTOR: 300 °C
-COLUMN: 40°C (1 min) to 200°C (10°C/min)

CARRIER GAS: Helium, 2.8 mL/min

COLUMN: Capillary, fused silica, 30-m x 0.32-mm ID, 1.00 µm film crossbonded® 100% dimethyl polysiloxane or equivalent

CALIBRATION: Standard solutions of isopropyl acetate in carbon disulfide/ methanol (99:1)

RANGE: 0.6 to 697.6 µg per sample

ESTIMATED LOD: 0.2 µg per sample

PRECISION ($\bar{S}_r$): 0.013

APPLICABILITY: The working range is 0.018 to 20.9 ppm (0.075 to 87.2 mg/m^3) for an 8-L air sample [3].

INTERFERENCES: None identified.

OTHER METHODS: This method was developed as an update for NMAM 1454, Issue 1 which was based upon NIOSH Method S50 from the 2nd edition of the NIOSH Manual of Analytical Methods [1,2].

REAGENTS:

1. Isopropyl acetate, ACS reagent grade.
2. Eluent: carbon disulfide, chromatographic grade.*
3. Methanol, chromatographic grade.*
4. Standard solutions: Isopropyl acetate in carbon disulfide/methanol (99:1) in the range of 0.2 to 700 µg 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 Rtx-1® or equivalent capillary column (page 1460-1).
4. Vials, 2-mL glass, PTFE-lined caps.
5. Syringe, 10-, 25-µL, readable to 0.1 µL.
6. Syringe, 1-mL, readable to 0.1 mL.
7. Pipets, 3-, 5-mL, readable to 0.1 mL.
8. 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. Methanol is flammable.

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 glass wool) and back sorbent sections of the sampler tube in separate vials. Discard the foam plugs.
6. Add 1.0 mL CS_2 / methanol (99:1) to each container and cap tightly.
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 from below the LOD to 10 times the LOQ. If necessary, additional standards may be added to extend the calibration curve.
   a. Add known amounts of isopropyl acetate to solvent 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. µg 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 isopropyl acetate or of a standard solution of isopropyl acetate in solvent 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 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 1460-1. 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 solvent, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass µg (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):

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

NOTE: µg/L=mg/m^3

EVALUATION OF METHOD:

The current method development effort was the result of requests to evaluate and improve problematic gas chromatography methods as part of the NMAM methods update project. Initial evaluations confirmed that Anasorb CSC (coconut shell charcoal) tubes were still the most suitable media for the sampling and analysis of isopropyl acetate. The average DE recoveries for isopropyl acetate, at substantially lower analyte concentration ranges (17 to 698 µg versus 3.75 to 15.4 mg [1,2]) were improved over the previous method (98.4% [3] versus 86.2%[1,2]) when CS_2/methanol (99:1) was used as the desorption solvent. The LOD for isopropyl acetate was lowered to 0.6 µg/sample [3] from the 10 µg/sample [1,2] reported in the previous method. A 30-day storage stability study was completed for isopropyl acetate with an average recovery of 99.9% [3].

REFERENCES:

[1] NIOSH [1977]. NIOSH Manual of Analytical Methods, 2nd ed., V. 2, S50, U.S. Department of Health and Human Services. DHEW (NIOSH) Publication No. 77-157-B.
[2] NIOSH [1977]. Documentation of NIOSH Validation Tests, NIOSH Contract No. CDC-99-74-45. Department of Health, Education and Welfare. (NIOSH) Publication 77-185.
[3] Pendergrass, SM [2000]. Isopropyl Acetate Backup Data Report, (unpublished, November).

METHOD WRITTEN BY:

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