METHYL CELLOSOLVE ACETATE 1451

$CH_3COOCH_2CH_2OCH_3$ MW: 118.15 CAS: 110-49-6 RTECS: KL5950000

METHOD: 1451, Issue 2 **EVALUATION: FULL** **Issue 1: 6 December 1974**
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OSHA : 25 ppm (skin)
NIOSH: 0.1 ppm (skin)
ACGIH: 5 ppm (skin)
(1 ppm = 4.83 mg/m^3 @ NTP)

PROPERTIES: liquid; BP 145 °C; MP -65 °C; Flash point 49 °C; d 1.005 @ 20 °C; VP 2 mm Hg (1700 mg/m^3) @ 20 °C

SYNONYMS: 2-methoxyethyl acetate; glycol monomethyl ether acetate; ethylene glycol monomethyl ether acetate

SAMPLING

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

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 0.2 L @ 25 ppm
-MAX: 20 L

SHIPMENT: routine

SAMPLE STABILITY: at least 15 days @ 0 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 51 to 214 mg/m^3 [2] (20-L samples)

BIAS: - 0.4%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.078 [2]

ACCURACY: ± 13.3%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: methyl cellosolve acetate

EXTRACTION: carbon disulfide, 1 mL

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTOR: 225 °C
-DETECTOR: 250 °C
-COLUMN: 90 °C

CARRIER GAS: helium, 30 mL/min

COLUMN: 10 ft x 1/8-in stainless steel packed with 5% FFAP stationary phase on 100/200 mesh Supelcoport

CALIBRATION: standard solutions of methyl cellosolve acetate in carbon disulfide

RANGE: 30 to 4500 µg per sample [2]

ESTIMATED LOD: 10 µg per sample [2]

PRECISION ($\bar{S}_r$): 0.0441 [2]

APPLICABILITY: The working range is 0.3 to 47 ppm (1.5 to 225 mg/m^3) for a 20-L air sample. Better sensitivity may be achieved if a capillary column is used in place of a packed column. A 30-m x 0.32-mm fused silica capillary column coate d internally with 1.0 µm of DB-5, with appropriate instrument conditions, is recommended.

INTERFERENCES: High humidity conditions may cause inefficient collection of organic vapors.

OTHER METHODS: This is an adaptation of NIOSH Method S39 [2] and OSHA Method 53, issued 1/85. [1] Operational modifications [3] to the OSHA method are as follows: column -30 m x 0.32-mm I.D. fused silica capillary coated with 1.0 µm DB-5. Column temperature: 40 °C for 4 min and programmed at 4 °C/min. to 85 °C. Desorption: 30 min in 1.0 mL methylene chloride/methanol, 95/5(v/v) containing 1 µL/mL toluene as internal standard.

REAGENTS:

1. Methyl cellosolve acetate, ACS reagent grade.
2. Eluent: carbon disulfide*, chromatographic grade, with 0.1% v/v undecane (optional internal standard).
3. Helium, purified.
4. Hydrogen, prepurified.
5. Air, filtered, compressed.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-m 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 must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph equipped with FID, integrator and column (page 1451-1).
4. Vials, 2-mL, glass, PTFE-lined crimp caps.
5. Syringe, 10 µL, readable to 0.1 µL.
6. Pipet, 1-mL, readable to 0.1 mL.
7. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and a serious fire and explosion hazard (flash point = -30 °C); work with it only in a hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break 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 0.2 to 20 L.
4. Cap the samplers and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL eluent 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 of 10 to 4500 µg per sample.
   a. Add known amounts of methyl cellosolve acetate to 1 mL eluent in sample vials.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area or ratio of peak area of analyte to peak area of internal standard vs. µg methyl cellosolve acetate).
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 calibration stock solution 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 methyl cellosolve acetate.

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 1451-1. 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. If internal standard is used, divide the peak area of analyte by the peak area of internal standard on the same chromatogram.

CALCULATIONS:

13. Determine the mass, µg (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 concentration, C, of methyl cellosolve acetate in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

This method was validated over the range 51 to 214 mg/m^3 at 23 °C and pressure of 762 mm Hg using 20-L samples [2]. Overall sampling and measurement precision, $\hat{S}_{rT}$, was 0.078, with average recovery of 99.8% [2]. Sample stability of methyl cellosolve acetate on charcoal was determined by OSHA to be at least 15 days at 0 °C [1].

REFERENCES:

[1] OSHA Manual of Analytical Methods, Method 53, U.S. Occupational Safety and Health Administration, Salt Lake City, Utah (1985).
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 2, S39, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
[3] Analytical Report for Organics on Charcoal Tubes, Data Chem Sequence # 6227, Unpubl. NIOSH (1988).

METHOD REVISED BY:

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