ETHYL ACETATE

1457

$CH_3COOC_2H_5$ MW: 88.10 CAS: 141-78-6 RTECS: AH5425000

METHOD: 1457, Issue 1 **EVALUATION: FULL** **Issue 1: 15 August 1994**

OSHA : 400 ppm
NIOSH: 400 ppm
ACGIH: 400 ppm
(1 ppm = 360 mg/m^3 @ NTP)

PROPERTIES: liquid, d = 0.8945 g/mL @ 25 °C; BP = 77 °C; VP = 9.7 kPa (73 mm Hg) @ 20 °C

SYNONYMS: acetic ether; acetic ester; ethyl ethanoate

SAMPLING

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

FLOW RATE: 0.01 to 0.2 L/minute

VOL-MIN: 0.1 L @ 1400 mg/m^3
-MAX: 10 L

SHIPMENT: refrigerated

SAMPLE STABILITY: 6 days @ 5 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 704 to 2950 mg/m^3 [2] (6-L samples)

BIAS: -2.1%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.058 [2]

ACCURACY: ±11.8%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: ethyl acetate

EXTRACTION: 1 mL CS_2

INJECTION VOLUME: 1 µL

TEMPERATURE-INJECTION: 250 °C
-DETECTOR: 300 °C
-COLUMN: 35 °C, 2 min; 10 °C/min to 150 °C

COLUMN: DB-Wax; 30 m, 0.32-mm ID, 1-µm film thickness

CARRIER GAS: He, 1 mL/min

MAKEUP GAS: N_2, 30 mL/min

CALIBRATION: standard solutions of ethyl acetate in CS_2

RANGE: 1.5 to 1,000 µg per sample [1]

ESTIMATED LOD: 0.5 µg per sample [1]

PRECISION ($\hat{S}_r$): 0.019 @ 40.5 to 810 µg per sample [1]

APPLICABILITY: The working range is 0.07 to 790 ppm (0.25 to 2800 mg/m^3) for a 6-L air sample [2]. The method may be adapted for other esters with appropriate changes in chromatographic conditions.

INTERFERENCES: Any compounds with similar retention times.

OTHER METHODS: This revises Method S49 [2]. Improved recovery of analyte may be achieved with the addition of 5% butyl carbitol to the CS_2 desorption procedure [3,4].

REAGENTS:

1. Carbon disulfide,* chrom. grade.
2. Ethyl acetate, reagent grade.
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-mm 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 airflow 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. Refrigerant, bagged ("Blue Ice," or equivalent).
4. Gas chromatograph, FID, integrator and column (page 1457-1).
5. Vials, glass, 2-mL, PTFE-lined caps.
6. Syringes, 10-µL and other convenient sizes for preparing standards, readable to 0.1 µL.
7. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C.) Use only in a 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 0.01 and 0.2 L/min for a total sample size of 0.2 to 10 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment with bagged refrigerant.

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.
   NOTE: Decane or other suitable internal standard at 0.1% v/v may be added at this step.
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 8 to 1000 µg analyte per sample.
   a. Add known amounts of analyte 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 of analyte vs. mg ethyl acetate).

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 concentrations plus three media blanks.
10. Determine desorption efficiency (DE) for each lot of charcoal tubes used for sampling in the calibration range (step 9).
    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 ethyl acetate recovered.
11. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

12. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1457-1. 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.
13. Measure peak area.

CALCULATIONS:

14. Determine the mass, µg (corrected for DE), of ethyl 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.
15. Calculate concentration, C, of ethyl acetate in the air volume sampled, V (L), at the sampling site:

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

EVALUATION OF METHOD:

Method S49 [2] was evaluated over the range of 704 to 2950 mg/m^3 for 6-L samples at 23 °C and a pressure of 759 mm Hg. The overall sampling and analytical precision ($\hat{S}_{rT}$) was 0.058 with an average recovery of 97.9% and the breakthrough capacity was found to be 25 mg of ethyl acetate [5]. The method was updated later for a lower range (40.5 to 810 µg/sample) with a capillary column [1]. Desorption efficiencies ranged from 44.5% to 87.5% with an analytical precision ($\bar{S}_r$) of 0.058. Samples of ethyl acetate on coconut charcoal are stable for 6 days at 5 °C with a recovery of 91.0% based on samples analyzed immediately after collection [1].

REFERENCES:

[1] S.M. Pendergrass, Ethyl Acetate Method Development (unpublished report), NIOSH/MRSB, 1990, NIOSH Seq. Report 7716-F (unpublished, Feb. 5, 1993).
[2] NIOSH Manual of Analytical Methods, 2nd ed., V. 2, S49, U.S. Dept. of Health, Education, and Welfare, Publ. (NIOSH) 77-157B (1977).

[3] Analysis of NIOSH Samples for Ethyl Acetate, NIOSH/MRSB/MDS, Sequence #7133 - Cincinnati, Ohio (unpublished, 1991).
[4] Beck, S., T. Stock, and L. Whitehead, Improved Efficiency of Desorption of Oxygenated Solvents from Activated Charcoal Using a New Polar Additive to Carbon Disulfide, Appl. Occup. Environ. Hyg., 5(3), 171-177, 1990.
[5] Documentation of the NIOSH Validation Tests, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185(1977).

METHOD REVISED BY:

S.M. Pendergrass, NIOSH/DPSE.