ISOPROPYL ETHER 1618

$((CH_3)_2CH)_2O$ MW: 102.18 CAS: 108-20-3 RTECS: TZ425000

METHOD: 1618, Issue 1 **EVALUATION: FULL** **Issue 1: 15 August 1993**

OSHA : 500 ppm
NIOSH: 500 ppm
ACGIH: 250 ppm; STEL 310 ppm
(1 ppm = 4.18 mg/m^3 @ NTP)

PROPERTIES: liquid; d 0.723 g/mL @ 20 °C; BP 67.5 °C; VP 15.9 kPa (119 mm Hg, 15.7% v/v) @ 20 °C; explosive range 1.4 to 7.9% v/v in air

SYNONYMS: diisopropyl ether; 2-isopropoxypropane; 2,2'-oxybispropane

SAMPLING

SAMPLER:	SOLID SORBENT TUBE (coconut shell charcoal; 100 mg/50 mg)
FLOW RATE:	0.01 to 0.05 L/min
VOL-MIN:	0.1 L @ 500 ppm
-MAX:	3 L
SHIPMENT:	routine
SAMPLE STABILITY:	at least 5 days @ 25 °C
BLANKS:	2 to 10 field blanks per set

ACCURACY

RANGE STUDIED:	992 to 4260 mg/m^3 [1] (3-L samples)
BIAS:	2.2% [1]
OVERALL PRECISION ($\hat{S}_{rT}$):	0.056 [1]
ACCURACY:	±12.0%

MEASUREMENT

TECHNIQUE:	GAS CHROMATOGRAPHY, FID
ANALYTE:	isopropyl ether
DESORPTION:	1 mL CS_2, 30 min
TEMPERATURE-INJECTION:	150 °C
-DETECTOR:	220 °C
-COLUMN:	80 °C
INJECTION VOLUME:	5 µL
CARRIER GAS:	N_2, 50 mL/min
COLUMN:	stainless steel, 3.2-mm ID x 6.1 m, packed with 10% FFAP on 80/100 mesh Chromosorb W-AW
CALIBRATION:	standard solutions of isopropyl ether and internal standard in CS_2
RANGE:	0.05 to 12 mg per sample
ESTIMATED LOD:	0.01 mg
PRECISION ($\bar{S}_r$):	0.009 [1]

APPLICABILITY: The working range is 3 to 1170 ppm (16 to 4900 mg/m^3) for a 3-L air sample. An appropriate capillary column may be used for better resolution and sensitivity. The sorbent's capacity for the analyte has not been determined under conditions of high relative humidity [1].

INTERFERENCES: None identified.

OTHER METHODS: This is Method S368 [2] in a revised format.

REAGENTS:

1. Eluent: carbon disulfide* (CS_2), chromatographic quality, containing 1% (v/v) octane, undecane, or other suitable internal standard.
2. Isopropyl ether*, reagent grade.
3. Nitrogen, purified.
4. Hydrogen, purified.
5. Air, compressed, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: borosilicate tubes, 7.0 cm long, 6-mm OD, 4-mm ID; flame-sealed ends with plastic caps, containing two sections of 20/40 mesh activated (600 °C) coconut charcoal (front = 100 mg; back = 50 mg) separated by a urethane foam plug. A silanized glass wool plug held in place with a metal spring precedes the front section and a urethane foam plug follows the back section. Pressure drop across the tube at 1.0 L/min air flow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.05 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator, and column (page 1618-1).
4. Vials, 2-mL, with PTFE-lined crimp caps.
5. Microliter syringes, 10-µL and convenient sizes for making dilutions.
6. Flasks, volumetric, 10-mL.
7. Pipets, 1-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and a serious fire and explosion hazard (flash point = -30 °C); isopropyl ether is flammable and tends to form explosive peroxides [3]. All work with these compounds must be done 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 0.05 L/min for a total sample size of 0.1 to 3 L.
4. Cap the samplers. 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 eluent to each vial. Cap 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 0.01 to 12 mg isopropyl ether per sample.
    a. Add a known amount of isopropyl ether to eluent in 10-mL volumetric flask and dilute to the mark. Use serial dilutions as needed for smaller concentrations.
    b. Analyze with samples and blanks (steps 11 and 12).
    c. Prepare calibration graph (ratio of isopropyl ether peak area to internal standard peak area vs. mg isopropyl ether).

9. Determine desorption efficiency (DE) at least once for each lot of charcoal used for sampling in the range of interest. 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 known amount (1 to 20 µL) of isopropyl ether or standard solution of isopropyl ether in eluent 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 with working standards (steps 11 and 12).
   e. Prepare a graph of DE vs. mg isopropyl ether recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure 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 1618-1. 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 an aliquot of the desorbed liquid with eluent, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area. Divide peak area of isopropyl ether by peak area of internal standard for each chromatogram.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of isopropyl ether 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 isopropyl ether in the air volume sampled, V (L):

$$C = \frac{W_f + W_b - B_f - B_b}{V} \cdot 10^3, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

S368 was issued on April 21, 1976 [2] and validated over the range 992 to 4260 mg/m^3 for 3-L air samples from dynamically generated test atmospheres [1]. The isopropyl ether concentrations were verified by GC/FID analysis. Breakthrough (5% on the backup section) occurred at 26 min when sampling a test atmosphere containing 4260 mg/m^3 of isopropyl ether in dry air at a flow rate of 0.198 L/min. Average recovery for generated samples stored five days was 102.2%.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S368, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977). Available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.

[2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, S368, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

[3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services and U.S. Department of Labor, January 1981, Publ. (NIOSH) 81-123, available as GPO Stock #17-033-00337-8 from Superintendent of Documents, Washington, D.C. 20402.

METHOD REVISED BY:

Charles V. Cooper, NIOSH/DPSE. Method S368 was originally validated under NIOSH Contract CDC-99-74-45.