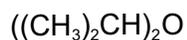


ISOPROPYL ETHER

1618



MW: 102.18

CAS: 108-20-3

RTECS: TZ425000

METHOD: 1618, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 August 1994

Issue 2: 15 March 2003

OSHA : 500 ppm
 NIOSH: 500 ppm
 ACGIH: 250 ppm; STEL 310 ppm

PROPERTIES: liquid; 0.725 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

(1 ppm = 4.18 mg/m³ @ NTP)**Synonyms:** diisopropyl ether; 2-isopropoxypropane; 2,2'-oxybispropane

SAMPLING		MEASUREMENT	
SAMPLER:	Solid Sorbent Tube (coconut shell charcoal; 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.05 L/min	ANALYTE:	Isopropyl ether
VOL-MIN:	0.1 L @ 500 ppm	DESORPTION:	1 mL CS ₂ ; stand 30 min
-MAX:	3 L	INJECTION VOLUME:	1 µL
SHIPMENT:	Routine	TEMPERATURES	
SAMPLE STABILITY:	30 days @ 5 °C	-INJECTION:	250 °C
BLANKS:	2 to 10 field blanks per set	-DETECTOR:	300 °C
		-COLUMN:	35 °C (1 min) - 150 °C ramp (10 °C/min)
		CARRIER GAS:	Helium (1-2 mL/min)
		COLUMN:	Capillary, fused silica, 30-m x 0.32-mm ID; 3µm film 100% dimethyl siloxane
		CALIBRATION:	Solutions of analyte in CS ₂
		RANGE:	2 to 651 µg per sample (capillary column); 1000 to 12000 µg per sample (packed column)
		ESTIMATED LOD:	0.2 µg per sample [1]
		PRECISION (S_r):	0.014 [1]
ACCURACY			
RANGE STUDIED:	992 to 4260 mg/m ³ [2] (3-L samples)		
BIAS:	2.2%		
OVERALL PRECISION (S_r):	0.056		
ACCURACY:	±12.0%		

APPLICABILITY: The working range is 3 to 1170 ppm (16 to 4900 mg/m³) for a 3-L air sample [1]. The sorbent's capacity for the analyte has not been determined under conditions of high relative humidity [2].

INTERFERENCES: Any compound that has a similar retention time under these analytical conditions.

OTHER METHODS: This method is based on and supercedes S368 [3] and NMAM 1618 (Issue 1) [5].

REAGENTS:

1. Eluent: Carbon disulfide* (CS₂), chromatographic quality.
2. Isopropyl ether*, reagent grade.
3. Helium, prepurified and filtered.
4. Hydrogen, prepurified and filtered.
5. Air, prepurified and filtered.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of activated coconut shell charcoal (front = 100 mg, back = 50 mg) separated by a 2-mm urethane foam plug. A silanized glass wool plug precedes the front section, and a 3-mm urethane foam plug follows the back section. Tubes are commercially available.
2. Personal sampling pumps (0.01 to 0.05 L/min) with flexible tubing.
3. Gas chromatograph, FID, integrator, and Rtx®-1 or equivalent capillary column (see page 1618-1).
4. Glass autosampler vials (2-mL) with PTFE-lined caps.
5. Pipettes (1-mL) and pipette bulb.
6. Microliter syringes, 10- μ L and convenient sizes for making dilutions .
7. Volumetric flasks (10-mL).

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and highly flammable (flash point = -30°C); isopropyl ether is flammable and tends to form explosive peroxides [4]. Prepare samples and standards in a well-ventilated 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 the sampler to a 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 and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Include the glass wool plug in the vial with the front sorbent section. Discard the foam plugs.
6. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial immediately.
7. Allow to stand at least 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards from below the LOD to 10 times the LOQ. Additional standards may be added to extend the calibration curve if necessary.
 - a. Add known amounts of isopropyl ether to eluent in 10-mL volumetric flasks and dilute to the mark. Prepare additional standards by serial dilution in 10-mL volumetric flasks.
 - b. Analyze with samples and blanks (steps 11 and 12).
 - c. Prepare a calibration graph (peak area of analyte vs. mg of analyte per sample).
9. Determine the desorption efficiency (DE) at least once for each lot of charcoal used for sampling in the calibration range (step 8).

- a. Prepare three tubes at each of five levels plus three media blanks.
- b. Remove and discard the back sorbent section of a blank sampler.
- c. Inject a known amount of stock solution (1 to 20 μL) directly onto the front sorbent section with a microliter syringe.
- d. Allow the tubes to air equilibrate for several minutes, then cap the tubes and allow to stand overnight.
- e. Desorb (steps 5 through 7) and analyze with standards and blanks (steps 11 and 12).
- f. Prepare a graph of DE vs. μg analyte recovered.
- g. Analyze a minimum of three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.
- h. Analyze at least three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

10. Set the gas chromatograph according to the manufacturer's recommendations and to the conditions given on page 1618-1. Inject a 1- μL aliquot manually using a solvent flushing technique or with an autosampler.
NOTE: If the peak area is above the linear range of the working standards, dilute with solvent, reanalyze and apply the appropriate dilution factor in the calculations.
11. Measure the peak area.

CALCULATIONS:

12. 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.
13. Calculate the concentration, C , of analyte in the air volume, $V(\text{L})$, sampled:

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

EVALUATION OF METHOD:**Issue 1**

S368 was issued on April 21, 1976 [3] and validated over the range 992 to 4260 mg/m^3 for 3-L air samples from dynamically generated test atmospheres [2]. 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.

Issue 2

The desorption efficiency, at levels ranging from 10 times the LOQ to 0.1 times the REL, was determined by spiking known amounts of isopropyl ether (in CS_2) on coconut shell charcoal tubes. The isopropyl ether exhibited acceptable desorption efficiency recovery results (97.1% - 103.4%) over the 20 to 651 μg range evaluated [1]. Isopropyl ether was evaluated for its storage stability. Sorbent tubes were spiked at approximately 615 μg and stored at ambient temperature for 7 days, then transferred to a refrigerator at 5° C. Samples were analyzed after 7, 14, and 30 days. Average recovery was 101.1%, 98.7%, and 94.8% respectively [1]. For this issue, the analytical range is lowered due to increased sensitivity and resolution with capillary column chromatography.

REFERENCES:

- [1] Pendergrass SM, May LR [2002]. NMAM 1618 Backup Data Report, Cincinnati, OH: National Institute for Occupational Safety & Health, DART, February.
- [2] NIOSH [1977]. Documentation of the NIOSH Validation Tests, S368. U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185. Available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.
- [3] NIOSH [1977]. NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, S368, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C. Method S368 was originally validated under NIOSH Contract CDC-99-74-45.
- [4] NIOSH/OSHA [1981]. NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services and U.S. Department of Labor, Publ. (NIOSH) 81-123, January. Available as GPO Stock #17-033-00337-8 from Superintendent of Documents, Washington, D.C. 20402.
- [5] NIOSH [1994]. NIOSH Manual of Analytical Methods, 4th ed., supplement, NMAM 1618, Depart. of Health and Human Services, DHHS (NIOSH) Publication No. 94-113.

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