

TRIFLUOROBROMOMETHANE

1017

CBrF₃

MW: 148.92

CAS: 75-63-8

RTECS: PA5425000

METHOD: 1017, Issue 2
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EVALUATION: PARTIAL

Issue 1: 15 August

OSHA : 1000 ppm
 NIOSH: 1000 ppm
 ACGIH: 1000 ppm
 (1 ppm = 6.09 mg/m³ @ NTP)

PROPERTIES: gas; vapor density 5 (air = 1);
 BP -57.8 °C; MP -168 °C;
 nonflammable

SYNONYMS: Refrigerant 13B1; bromotrifluoromethane.

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBES (two coconut shell charcoal tubes in series, 400 mg/200 mg and 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.05 L/min	ANALYTE:	trifluorobromomethane
VOL-MIN:	0.1 L @ 1000 ppm	DESORPTION:	5 mL methylene chloride; stand 30 min
-MAX:	1 L	INJECTION VOLUME:	5 µL
SHIPMENT:	separate front and back tubes; in dry ice	TEMPERATURE-INJECTOR:	170 °C
SAMPLE STABILITY:	not determined	-COLUMN:	105 °C
BLANKS:	2 to 10 field blanks per set	-DETECTOR:	230 °C
ACCURACY		CARRIER GAS:	N ₂ , 50 mL/min
RANGE STUDIED:	2890 to 11,500 mg/m ³ [1] (1-L samples)	COLUMN:	stainless steel, 1.2 x 6-mm OD, packed with 50/80 mesh Porapak Q
BIAS:	- 2.9%	CALIBRATION:	standard solutions of analyte in methylene chloride
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.065 [1]	RANGE:	0.5 to 18 mg per sample [1]
ACCURACY:	± 13.46%	ESTIMATED LOD:	0.05 mg per sample
		PRECISION (\hat{S}_r):	0.038 @ 3 to 12 mg per sample [1]

APPLICABILITY: The working range is 80 to 3000 ppm (500 to 18,000 mg/m³) for a 1-L air sample.

INTERFERENCES: None reported.

OTHER METHODS: This revises Method S125 [2].

REAGENTS:

1. Methylene chloride (CH_2Cl_2), chromatographic quality.*
2. Trifluorobromomethane, 99%.
3. Nitrogen, purified.
4. Hydrogen, prepurified.
5. Air, filtered, compressed.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: two glass tubes (9 cm long, 8-mm OD, 6-mm ID, followed by 7 cm long, 6-mm OD, 4-mm ID) connected in series with a short piece of tubing, flame-sealed ends and plastic end caps. Each tube contains two sections of 20/40 mesh activated (600 °C) coconut shell charcoal (front tube = 400 mg + 200 mg; back tube = 100 mg + 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes each front section and a 3-mm urethane foam plug follows each back section. Pressure drop across the tubes at 1 L/min airflow 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, flame ionization detector, integrator, and column (see page 1017-1).
4. Vials, 10-mL, PTFE-lined caps.
5. Syringes, gas tight, 10- μL to 3-mL.
6. Volumetric flasks, 10-mL.
7. Pipet, TD, 5-mL.
8. DE apparatus. Glass tubing, through which nitrogen flows at ca. 200 mL/min, with T-connection and septum. Wrap outlet of tubing with heating tape and connect tubing to inlet of test sampler.

SPECIAL PRECAUTIONS: Methylene chloride is a suspect carcinogen and is metabolized to carbon monoxide, a toxic gas [3].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler tubes immediately before sampling. Attach sampler to personal sampling pump with flexible tubing with the smaller tube nearer the sampling pump.
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 1 L.
4. Separate and cap the tubes. Pack securely for shipment in an insulated container with dry ice.

SAMPLE PREPARATION:

5. Pipet 5.0 mL chilled CH_2Cl_2 into a series of vials.
6. Place the front and back sorbent sections of the front sampler tube in a vial. Discard the glass wool and foam plugs. Immediately cap the vial. Similarly place the front and back sorbent sections of the back sampler tube in a separate vial.
7. Allow to stand 30 min with occasional agitation. Analyze within 6 h.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards.
 - a. Add known volumes of trifluorobromomethane gas with a gas-tight syringe to 5.0 mL CH₂Cl₂ in 10-mL volumetric flasks by slowly bubbling the gas through the liquid with the tip of the syringe needle near the bottom of the volumetric flask. Dilute to the mark. Use serial dilutions as needed to obtain trifluorobromomethane concentrations in the range 0.01 to 3.6 mg/mL.
 - b. Analyze with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (peak area vs. mg trifluorobromomethane).
9. Determine desorption efficiency (DE) at least once for each lot of sorbent used for sampling in the range of interest. Prepare three tubes at each of five levels plus three media blanks.
 - a. Connect a large charcoal tube (400 mg/200 mg) to the outlet of the DE apparatus. Start nitrogen flow (ca. 200 mL/min) through the DE apparatus and into the charcoal tube. Inject a known amount (0.01 to 3 mL; 0.05 to 18 mg @ NTP) of trifluorobromomethane gas into the septum. Allow the N₂ flow to continue for 20 sec.
 - b. Cap the tube. Allow to stand overnight at -10 °C.
 - c. Desorb (steps 5 through 7) and analyze with working standards (steps 11 and 12).
 - d. Prepare a graph of DE vs. mg trifluorobromomethane 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 1017-1. 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 CH₂Cl₂ reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of trifluorobromomethane found in the sample front (W_f) and back (W_b) sorbent tubes, and in the average media blank front (B_f) and back (B_b) sorbent tubes.
NOTE: If W_b > W_f/10, report breakthrough and possible sample loss.
14. Calculate concentration, C, of trifluorobromomethane in the air volume sample, V (L):

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

EVALUATION OF METHOD:

Method S125 was issued on May 9, 1975 [2], and validated over the range 2890 to 11,500 mg/m³ at 25 °C and 760 mm Hg using 1-L samples [1]. Generated concentrations were produced by calibrated gas flows and verified by a total hydrocarbon analyzer, standardized with gas standards. Overall precision, s_r, was 0.065, with average recovery of 100%. Desorption efficiency was 0.81 in the range 3 to 12 mg per sample. Breakthrough (effluent = 5% of test concentration) occurred in 90 min when a concentration of 12,370 mg/m³ was sampled at a rate of 0.046 L/min. The stability of trifluorobromomethane on charcoal was not determined.

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S125, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as Stock No. PB 274-248 from NTIS, Springfield, VA 22161.
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S125, U.S. Department of Health Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [3] NIOSH Current Intelligence Bulletin 46, U.S. Department of Health and Human Services, Publ. (NIOSH) 86-114 (1986).

METHOD REVISED BY:

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