

DICHLOROFLUOROMETHANE

2516

CHCl₂F

MW: 102.92

CAS: 75-43-4

RTECS: PA8400000

METHOD: 2516, Issue 2

EVALUATION: FULL

Issue 1: 15 May 1985

Issue 2: 15 August 1994

OSHA : 1000 ppm
NIOSH: 10 ppm
ACGIH: 10 ppm
 (1 ppm = 4.21 mg/m³ @ NTP)

PROPERTIES: gas; BP 8.9 °C; d 1.405 g/mL @ 9 °C;
 vapor density (air = 1) 3.82;
 not combustible

SYNONYMS: Freon 21; Halon 112; dichloromonofluoromethane

APPLICABILITY: The working range is 120 to 1500 ppm (500 to 6300 mg/m³) for a 2-L air sample.

INTERFERENCES: None known.

OTHER METHODS: This revises Method S109 [2].

REAGENTS:

1. Eluent: carbon disulfide,* chromatographic quality, containing 0.4% v/v pentane as internal standard.
2. Dichlorofluoromethane, 99%.*
3. Nitrogen, purified.
4. Hydrogen, prepurified.
5. Air, filtered, compressed.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: two glass tubes connected in series with a short piece of plastic tubing; each tube 10 cm long, 8-mm OD, 6-mm ID, with plastic caps, containing 20/40 mesh activated (600 °C) coconut shell charcoal (front tube = 400 mg; back tube = 200 mg). A plug of silylated glass wool is placed at each end of each tube. Pressure drop across sampler less than 3.4 kPa (2.5 cm Hg) at 1 L/min airflow. 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 2516-1).
4. Vials, glass, serum, 10-mL, with PTFE-lined septa and crimp caps.
5. Syringe, 10- μ L.
6. Syringes, gas-tight, 10-mL and 100- μ L.
7. Syringe needles, 22-gauge.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an extreme fire and explosion hazard (flash point = -30 °C); work with it only in a hood. Dichlorofluoromethane may cause irregular heartbeat if inhaled [3].

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 0.25 to 3 L.
4. Separate the front and back tubes immediately after sampling. Cap the tubes. Pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler in separate vials. Discard the glass wool plugs. Attach crimp cap to each vial. Insert a syringe needle vent through the septum to serve as a vent.
6. Add 5.0 mL eluent to each vial with a syringe. Remove the syringe needle vent from the septum.
NOTE: To avoid loss of analyte, do not add eluent to the charcoal before sealing the 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 0.05 to 35 mg (0.01 to 8.2 mL at 20 °C and 101 kPa) dichlorofluoromethane per sample.
 - a. Add 5.0 mL eluent to each of a series of vials. Attach crimp cap to each vial.

- b. Immediately before adding dichlorofluoromethane, withdraw from each vial an amount of air equal to that of dichlorofluoromethane to be added.
 - c. Slowly bubble a measured amount of dichlorofluoromethane into the liquid in each vial using a gas-tight syringe.
 - d. Analyze together with samples and blanks (steps 11 and 12).
 - e. Prepare calibration graph (ratio of peak area of analyte to peak area of internal standard vs. mg dichlorofluoromethane).
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 levels plus three media blanks.
 - a. Inject a known amount of dichlorofluoromethane slowly and directly into a media blank front sorbent section with a gas-tight syringe.
 - b. Cap the tube. Allow to stand overnight.
 - c. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - d. Prepare a graph of DE vs. mg dichlorofluoromethane 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 2516-1. Inject sample aliquot manually using solvent flush technique or with autosampler. $t_r = 6$ min under these conditions.

NOTE 1: If an autosampler is used, transfer an aliquot of the sample solution to a sealed empty vial using a syringe needle as a vent.

NOTE 2: 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. Divide the peak area of analyte by the peak area of internal standard of the same chromatogram.

CALCULATIONS:

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

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

EVALUATION OF METHOD:

Method S109 was issued on September 30, 1976 [2], and validated over the range 2100 to 12,600 mg/m^3 by analyzing 18 spiked samples and 18 samples collected from dynamically-generated atmospheres of dichlorofluoromethane gas [1,4]. These recoveries were 100.9 and 102.9%, respectively. A combined storage migration study determined that samples of 17 and 35 mg had a 100% recovery after seven days if stored without backup sections. At the same levels, those front sections stored with backup sections had 83.2% recovery. Breakthrough (5%) in an atmosphere of 9120 mg/m^3 and 90% RH, sampling at 0.187 L/min, occurred at 23 min. giving a breakthrough volume of 4.3 L and tube capacity of 39 mg. In a dry atmosphere of 8850 mg/m^3 , breakthrough occurred after 64 min sampling at 0.187 L/min, giving a breakthrough volume of 12.0 L and tube capacity of 102 mg. Desorption efficiencies averaged 101% over the range 5.9 to 34 mg dichlorofluoromethane per sample.

REFERENCES:

- [1] Backup Data Report, S109, available as "Ten NIOSH Analytical Methods, Set 1," Order No. PB 271-712 from NTIS, Springfield, VA 22161.
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S109, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, Dichlorofluoromethane, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.
- [4] NIOSH Research Report - Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

METHOD REVISED BY:

Y.T. Gagnon, NIOSH/DPSE; S109 originally validated under NIOSH Contract 210-76-0123.