

1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE

1020

CCl₂F - CClF₂

MW: 187.38

CAS: 76-13-1

RTECS: KJ4000000

METHOD: 1020, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 August 1987

Issue 2: 15 August 1994

OSHA : 1000 ppm; STEL 1250 ppm
NIOSH: 1000 ppm; STEL 1250 ppm
ACGIH: 1000 ppm; STEL 1250 ppm
 (1 ppm = 7.66 mg/m³ NTP)

PROPERTIES: clear liquid; d = 1.55 g/mL @ 20 °C;
 BP = 47.6 °C; MP -35 °C; vapor pressure
 = 38 kPa (384 mm Hg, 51% v/v) @ 20 °C

SYNONYMS: Freon 113, Refrigerant 113, Freon TF

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPH, FID
FLOW RATE:	0.01 to 0.05 L/min	ANALYTE:	1,1,2-trichloro-1,2,2-trifluoroethane
VOL-MIN:	0.1 L @ 1000 ppm	DESORPTION:	1 mL CS ₂ ; stand 30 minutes
-MAX:	3 L	INJECTION VOLUME:	1 µL
SHIPMENT:	refrigerated	TEMPERATURE-INJECTION:	250 °C
SAMPLE STABILITY:	undetermined	-DETECTOR:	300 °C
BLANKS:	2 to 10 field blanks per set	-COLUMN:	32° to 90° @ 5°/min °C
ACCURACY		CARRIER GAS:	He; 1 to 2 mL/min
RANGE STUDIED:	3300 to 14,200 mg/m ³ [1] (1.5-L samples)	COLUMN:	DB-Wax/Stabilwax fused silica capillary, 30 m x 0.32-mm ID, split injection 5:1; (<500 µg, splitless).
BIAS:	- 2.3%	CALIBRATION:	standard solutions of analyte in CS ₂
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.07 [1]	RANGE:	0.015 to 14 mg per sample [2]
ACCURACY:	± 14.1%	ESTIMATED LOD:	0.005 mg per sample [2]
		PRECISION (\hat{S}_r):	0.02 [2]

APPLICABILITY: The working range is 10 to 18000 mg/m³ (1.3 to 2350 ppm) for a 1.5-L air sample. This method may be used with diffusive sampler collection, but a larger volume of CS₂ may be needed for desorption.

INTERFERENCES: None studied.

OTHER METHODS: This is a revision of NIOSH 3rd ed. Method 1020 (8/15/90). This method was updated for sequences 6937, 6956, and 6916A [1]. Method S129 [3] was originally evaluated under NIOSH Contract CDC-99-74-45.

REAGENTS:

1. Carbon disulfide (CS₂), chromatographic quality.*
2. 1,1,2-Trichloro-1,2,2-Trifluoroethane (TTE), 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, with flame-sealed ends with plastic caps, containing two sections of 20/40 mesh 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 0.05 L/min, with flexible connecting tubing.
3. Refrigerant, bagged, and insulated shipping container.
4. Gas chromatograph, flame ionization detector, integrator, and column (see page 1020-1).
5. Vials, 2-mL, PTFE-lined caps.
6. Syringes, 10- to 100- μ L, readable to 0.5%.
7. Volumetric flasks, 10-mL.
8. Pipet, TD 1-mL, with pipet bulb.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and a serious fire and explosion hazard (flash point = -30 °C). 1,1,2-Trichloro-1,2,2-Trifluoroethane is a narcotic [4]. Work with these compounds 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 0.05 L/min for a total sample size of 0.1 to 3 L.
4. Cap the samplers. Pack securely for shipment in a refrigerated container.

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 CS₂ 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 in the range of 0.1 to 20 mg/mL.
 - a. Add known amounts of TTE to CS₂ in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze with samples and blanks (steps 11 and 12).

- c. Prepare calibration graph (peak area vs. mg TTE).
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. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount (2 to 20 μL) of TTE or a standard solution of TTE in CS_2 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 TTE.
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 1020-1. 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 CS_2 , reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of TTE 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 TTE 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 S129 was issued on May 9, 1975 [3], and validated with generated atmospheres using calibrated syringe drive and independent verification by gas chromatography [2]. Average recovery was 100.3% (18 samples) in the range 3300 to 14,200 mg/m^3 for 1.5-L samples. Breakthrough (effluent = 5% of test concentration) occurred after sampling for 60 min at 0.046 L/min from an atmosphere containing 14,500 mg/m^3 1,1,2-Trichloro-1,2,2-Trifluoroethane in dry air. Desorption efficiency for 18 samples of SKC lot 105 activated coconut charcoal spiked with 5.6 to 23 mg 1,1,2-Trichloro-1,2,2-Trifluoroethane averaged 1.01 with $\hat{S}_r = 0.02$. Sample stability was not determined.

REFERENCES:

- [1] Pendergrass, S.M., Method Development Efforts, NIOSH/DPSE Analytical Sequence #6937, 6956, and 6916A (unpublished) NIOSH/MRSB (1989).
- [2] Documentation of the NIOSH Validation Tests, S129, 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.
- [3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S129, U.S. Department of Health,

Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

- [4] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as Stock #PB83-154609 from NTIS, Springfield, VA 22161.

METHOD REVISED BY:

S.M. Pendergrass, NIOSH/DPSE; S129 originally validated under NIOSH Contract CDC-99-74-45.