

p-CHLOROBENZOTRIFLUORIDE**1026**C₇H₄ClF₃

MW: 180.56

CAS: 98-56-6

RTECS: XS9145000

METHOD: 1026, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 March 2003

OSHA: Not Established
 NIOSH: Not Established
 ACGIH: Not Established
 (1 ppm = 7.41 mg/m³)

PROPERTIES: liquid; d 1.353 g/mL @ 20 °C; BP 139°C;
 VP 5.3 mm Hg @ 20 °C

SYNONYMS: 4-Chlorobenzotrifluoride, 1-Chloro-4 (trifluoromethyl) benzene, 4-Chloro- α,α,α -trifluorotoluene, PCBTF

SAMPLING		MEASUREMENT	
SAMPLER:	Coconut Shell Charcoal (CSC) (100/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	p-Chlorobenzotrifluoride
VOL-MIN:	0.1 L	DESORPTION:	1 mL of carbon disulfide/methanol (99:1)
-MAX:	10.0 L (25 ppm)	INJECTION VOLUME:	1 μ L
SHIPMENT:	Routine	TEMPERATURE-INJECTION:	250°C
SAMPLE STABILITY:	7 days @ 25°C; 30 days @ 5 °C	-DETECTOR:	300°C
BLANKS:	10% of field samples	-COLUMN:	40°C (1 min) to 150°C (10°C/min)
ACCURACY		CARRIER GAS:	Helium; 2.8 mL/min
RANGE STUDIED:	Not Determined	COLUMN:	capillary, fused silica, 30-m x 0.32 mm ID; 1 μ m film 100% crossbonded dimethyl polysiloxane
BIAS:	Not Determined	CALIBRATION:	Solutions of p-CBTF in solvent
OVERALL PRECISION (\hat{S}_r):	Not Determined	RANGE:	1.8 μ g to 676 μ g [1]
ACCURACY:	Not Determined	ESTIMATED LOD:	0.6 μ g [1]
		PRECISION (\hat{S}_s):	0.0131 [1]

APPLICABILITY: The working range for p-chlorobenzotrifluoride was 0.024 to 9.15 ppm (0.178 to 67.8 mg/m³) for a 10-L air sample. [1]

INTERFERENCES: Any compounds having similar retention times may co-elute with the analyte of interest.

OTHER METHODS: None determined.

REAGENTS:

1. p-Chlorobenzotrifluoride, 98%.
2. Carbon disulfide, low benzene grade*.
3. Methanol, HPLC grade*.
4. Helium, purified.
5. Air, prefiltered and purified.
6. Hydrogen, purified.
7. Calibration stock: Dilute 5 µL of p-CBTF in 10 mL solvent. Prepare remaining standards by serial dilutions.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm ID, 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 silylated 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 pump, 0.01 to 0.2 L/min, with flexible tubing.
3. Gas chromatograph, FID, integrator, and Rtx-1, or equivalent, fused silica capillary column.
4. Autosampler vials, glass, 1.8 mL, with PTFE-lined caps.
5. Syringes, 10-µL to 1-mL.
6. Volumetric flasks, 10-mL and 250-mL.
7. Pipets, various sizes.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and flammable. Methanol is moderately toxic and flammable. Perform all work in a chemical hood and wear appropriate protective equipment. p-CBTF is an irritant compound.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sample tube in line.
2. Break open the ends of the sample tube immediately prior to sampling. Attach sample tube to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size of 0.1 to 10 L.
4. Cap both ends of the sample tubes and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the initial glass wool plug and front sorbent section of the sample tube in an autosampler vial. Place the back sorbent section of the sample tube in a separate autosampler vial. Discard the polyurethane foam separator and the remaining glass wool plug.
6. Add 1.0 mL of carbon disulfide/methanol (99:1) solvent to each vial and cap securely.
7. Allow to stand for 30 minutes with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working calibration standards from below the LOD to 10 times the LOQ. If necessary additional standards may be added to extend the calibration curve.
 - a. Add known amounts of calibration stock solution to carbon disulfide/methanol (99:1) in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 10 and 11).
 - c. Prepare calibration graph (peak area vs. µg p-CBTF).
9. Determine the desorption efficiency (DE) at least once for each batch 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. Inject a known amount of analyte stock solution directly onto the front sorbent section of each charcoal tube.
- c. Allow the tubes to air equilibrate for several minutes, then cap the ends of the tubes and allow to stand overnight.
- d. Desorb (steps 5-7) and analyze together with standards and blanks (steps 10 and 11).
- e. Prepare a graph of DE vs. μg p-CBTF recovered.

MEASUREMENT:

10. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1026-1. Inject a 1- μL sample aliquot manually using the solvent flush technique or with an autosampler.

NOTE: If peak area is above the linear range of the working standards, dilute with carbon disulfide/methanol (99:1), reanalyze and apply the appropriate dilution factor in the calculations.

11. Measure peak areas.

CALCULATIONS:

12. Determine the mass, μg (corrected for DE), for p-chlorobenzotrifluoride 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 concentration, C, of p-chlorobenzotrifluoride in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

This method development was based upon a health hazard evaluation request where p-chlorobenzotrifluoride was being used as a cleaning solvent for automobile parts. After an evaluation of various solid sorbent tubes and solvent/solvent mixtures, coconut shell charcoal tubes and carbon disulfide/methanol (99:1) solvent were selected for use in this method development. The desorption efficiency (DE) was determined for p-chlorobenzotrifluoride at 4 levels ranging from 27.1 μg to 541.2 μg . The average DE determined for p-chlorobenzotrifluoride was 100.6%. The LOD was determined to be 0.6 μg /sample. The analytical method precision, as determined from the pooled relative standard deviation (S_r), was determined to be 0.0131. Para-chlorobenzotrifluoride was determined to have acceptable storage stability (102.6%) for 30 days [1] at a level 0.1 times the recommended exposure level suggested by the manufacturer. [2]

REFERENCES:

- [1] Pendergrass SM [2000]. Backup Data for p-Chlorobenzotrifluoride Method Development, NIOSH/CEMB, Cincinnati, OH., (NIOSH, unpublished report July).
- [2] OCC [1998]. Material safety data sheet for p-chlorobenzotrifluoride. Dallas, Texas: Occidental Chemical Corporation, May.

METHOD WRITTEN BY:

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