

SEMI C3.40-1000

STANDARD FOR CARBON TETRAFLUORIDE (CF₄), VLSI GRADE

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1 Description

1.1 Carbon tetrafluoride is an odorless, colorless, nonflammable gas. It is a simple asphyxiant. It is also known as tetrafluoromethane.

2 Specifications

QUALITY: 99.997%

Impurities	Maximum Acceptable Level (ppm) *
Carbon Dioxide (CO ₂)	1
Carbon Monoxide (CO)	1
Nitrogen (N ₂)	20
Other Halocarbons (CF ₃ Cl, CF ₂ Cl ₂ , CF ₃ H)	1
Oxygen (O ₂)	5
Sulfur Hexafluoride (SF ₆)	1
Water (H ₂ O) (v/v)	1
TOTAL IMPURITIES	30

* An analysis of significant figures has not been considered. The number of significant figures will be based on analytical accuracy and the precision of the provided procedure.

3 Chemical Specification

Total Hydrolyzable Fluorides as HF	1 ppm max.
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4 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	88.005	88.005
Boiling point at 1 atm	-128.0°C	-198.4°F
Density of gas at 0°C (32°F) and 1 atm	3.946 kg/m ³	0.246 lb/ft ³
Specific gravity of gas at 0°C and 1 atm	3.05	3.05
Density of liquid at -80°C	1317 kg/m ³	82.22 lb/ft ³

5 Analytical Procedures (See Notes 1 and 2, and Figures 1 and 2.)

5.1 *Carbon Dioxide* — This procedure is for the determination of carbon dioxide in carbon tetrafluoride using a gas chromatograph with an ultrasonic detector.

5.1.1 *Detection Limit* — 0.5 ppm (mole/mole).

5.1.2 *Instrument Parameters*

5.1.2.1 Columns:

Column 1:	Haysep D, 100/120 mesh, 4.6 m (15 ft) by 3.2 mm (1/8 in) OD ss or equivalent.
Column 2:	Porapak QS, 100/120 mesh, 2.1 m (7 ft) by 3.2 mm OD ss or equivalent.

5.1.2.2 *Carrier Flow*: 16 mL/min helium.

5.1.2.3 *Temperatures*:

Detector	120°C
Column	50°C
Valve	90°C

5.1.2.4 *Sample Volume*: 1.0 mL

5.1.2.5 *Time Table* — Determine the times for valve switching and signal changes, and enter into the run table.

An example of a run table follows.

Valve	On	Off
1	0.01	*
2	9.20	*
3	0.01	12.00

* Valve left on until end of run.

5.1.3 *Calibration Standard* — 1–5 ppm carbon dioxide and balance helium.

5.1.4 *Operating Procedure*

5.1.4.1 Inject the calibration standard into the column using a gas sampling valve. Record retention times and peak areas. The retention time for carbon dioxide is approximately 16 minutes.

5.1.4.2 Inject the sample to be tested in the same manner as the calibration standard. Record the retention times and peak areas.

5.1.4.3 Repeat 5.1.4.1.

5.1.4.4 Compare the average peak areas of the calibration standard to those of the carbon tetrafluoride

sample being tested. Calculate the concentration of carbon dioxide in the sample, using the formula below. The result may not exceed the specification in Section 2 of this Standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \text{Concentration of Standard} = \text{Concentration of Sample}$$

5.2 Oxygen, Nitrogen and Carbon Monoxide — This procedure is for the determination of oxygen, nitrogen, and carbon monoxide in carbon tetrafluoride using a gas chromatograph with an ultrasonic detector.

5.2.1 Detection Limits — 0.5 ppm (mole/mole) for each impurity.

5.2.2 Instrument Parameters

5.2.2.1 Columns:

Column 3:	Porapak QS, 100/120 mesh, 3.6 m (12 ft) by 3.2 mm (1/8 in) OD ss or equivalent.
Column 4:	Haysep D, 100/120 mesh, 4.6 m (15 ft) by 3.2 mm OD ss or equivalent.
Column 5:	Molecular Sieve 13X, 45/60 mesh, 2.1 m (7 ft) by 3.2 mm OD ss or equivalent.

5.2.2.2 Carrier Flow: 16 mL/min helium.

5.2.2.3 Temperatures:

Detector	120°C
Column	50°C
Valve	90°C

5.2.2.4 Sample Volume: 2.0 mL

5.2.2.5 Time Table — Determine the times for valve switching and signal changes, and enter into the run table.

An example of a run table follows.

Valve	On	Off
1	0.01	*
2	9.20	*
3	0.01	12.00

* Valve left on until end of run.

5.2.3 Calibration Standard — 1–5 ppm (mole/mole) each nitrogen, oxygen and carbon monoxide, balance helium.

5.2.4 Operating Procedure

5.2.4.1 Inject the calibration standard into the column using a gas sampling valve. Record retention times and peak areas. The approximate retention times are:

oxygen 11.1 minutes, nitrogen 11.6 minutes, carbon monoxide 14.5 minutes.

5.2.4.2 Inject the sample to be tested in the same manner as the calibration standard. Record the retention times and peak areas.

5.2.4.3 Repeat 5.2.4.1.

5.2.4.4 Compare the average peak areas of the calibration standard to those of the carbon tetrafluoride sample being tested. Calculate the concentrations of oxygen, nitrogen and carbon monoxide in the sample, using the formula below. The results may not exceed the specifications in Section 2 of this Standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \text{Concentration of Standard} = \text{Concentration of Sample}$$

5.3 Fluorocarbon and Chlorofluorocarbon Impurities — This procedure is for the determination of the volatile organic impurities in carbon tetrafluoride using a gas chromatograph with a flame ionization detector.

5.3.1 Detection Limits — 0.5 ppm (mole/mole) for each impurity.

5.3.2 Instrument Parameters

5.3.2.1 Column: 1% SP-1000 on Carbopak B (60/80), 7.3 m (24 ft) by 3.2 mm (1/8 in) OD ss or equivalent.

5.3.2.2 Carrier Flow: 40 mL/min helium.

5.3.2.3 Support Gases: Set the flow rates as specified by the instrument manufacturer.

5.3.2.4 Temperatures:

Injection Port	200°C
Detector	250°C
Initial Oven	35°C
Pre-Program Hold	7 min
Temperature Rise	10°C/min
Final Oven	150°C
Final Hold	10 min

5.3.2.5 Sample Volume: 2 mL.

5.3.3 Calibration Standard — 1–10 ppm (by volume) desired impurities in helium. Practical impurities may be trifluoromethane (CHF₃), hexafluoroethane (C₂F₆), dichlorodifluoromethane (CCl₂F₂), and chlorotrifluoromethane (CClF₃).

5.3.4 Operating Procedure

5.3.4.1 Attach a stainless steel diaphragm two-stage regulator to the standard cylinder. Connect the regulator to the 6 port chromatographic sampling valve.