

SEMI C3.24-95 STANDARD FOR SULFUR HEXAFLUORIDE (SF₆) IN CYLINDERS, 99.97% QUALITY

1 Description

Sulfur hexafluoride is colorless and odorless. It is noncombustible and has a low toxicity.

2 Specifications

QUALITY: 99.97%

Impurities	Maximum Acceptable Level (ppm)*
Air	100
Carbon Tetrafluoride (CF ₄)	100
Hydrogen Fluoride (HF)	1
Water (H ₂ O) (v/v)	8
TOTAL IMPURITIES	209

* 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 Physical Constants (for information only)

	Metric Units	US Units
Molecular weight	146.054	146.054
Boiling point at 1 atm	-63.7°C	-82.7°F
Density of gas at 20°C (68°F) and 1 atm	6.162 kg/m ³	0.385 lb/ft ³
Specific gravity of gas at 21.1°C (70°F) and 1 atm (air = 1)	5.114	5.114
Density of liquid at -50°C	1910 kg/m ³	119.2 lb/ft ³

4 Analytical Procedures (Notes 1, 2, and 3)

4.1 *Air and Carbon Tetrafluoride* — This procedure is for the determination of air and carbon tetrafluoride using a gas chromatograph with a thermal conductivity detector.

4.1.1 *Detection Limit* — 1 ppm (mole/mole)

4.1.2 *Instrument Parameters*

4.1.2.1 *Column*: 2.4 m (8 ft) by 6.4 mm (1/4 in) OD, 5.1 mm (0.201 in) ID stainless steel tubing packed with Porapak Q (80/100 mesh) or equivalent.

4.1.2.2 *Carrier Flow*: 30 mL/min helium.

4.1.2.3 *Sample Volume*: 1 mL.

4.1.2.4 *Temperatures*:

Detector	110°C
Column	70°C

4.1.3 *Calibration Standards* — 90–100 ppm (mole/mole) carbon tetrafluoride, 90–110 ppm (mole/mole) Air (O₂/N₂ blend), balance helium.

4.1.4 *Operating Procedure*

4.1.4.1 Inject the calibration standard. Analyze the standard using the conditions described above. Record the retention times and peak areas.

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

4.1.4.3 Repeat 4.1.4.1.

4.1.4.4 Calculate the concentrations of air and carbon tetrafluoride in the sample, using the formula below. The result may not exceed the specification in Section 2 of this Standard.

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

4.2 *Hydrogen Fluoride* — This procedure is for the determination of hydrogen fluoride by quantitative collection in dilute sodium carbonate (NaHCO₃) and subsequent analysis by ion chromatography.

4.2.1 *Detection Limit* — 0.1 ppm for the method

Equipment Required:

500 mL Greenburgh impinger

50 mL Class A pipet

Type 316 stainless steel delivery system

5' Fluorocarbon tubing, 1/4 in OD by 0.03 in wall

Reagent Grade sodium bicarbonate

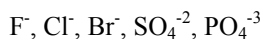
Deionized water

50 mL high-density polyethylene bottles

Assorted Class A volumetric flasks

Ion chromatograph

Anion column which separates:



4.2.2 Ion Chromatograph Parameters:

Instrument:	Dionex 2000 (or equivalent)
Integrator:	Dionex 4270 (or equivalent)
Column:	HDIC AS4A (or equivalent)
Eluent:	0.001 M Na_2CO_3 + 0.001 M NaHCO_3
Flow Rate:	2 mL/minute
Pressure:	960 psia
Detector Conductivity Range:	3.0 microsiemens for less than 10 ppm
Sample:	100 microliters

4.2.2 Calibration — Calibrate the ion chromatograph by dissolving a weighed amount of ammonium fluoride (NH_4F) in deionized water and sequentially diluting it to a fluoride concentration of 0.1 ppmw in Class A volumetric flasks and analyzing it as specified by the instrument manufacturer.

4.2.3 Operating Procedure

4.2.3.1 Prepare a fresh solution of 1.7 mM sodium bicarbonate in deionized water.

4.2.3.2 Pipet 200 mL of the solution into a 500 mL Greenburgh impinger.

4.2.3.3 Bubble approximately 20 L of sulfur hexafluoride (SF_6) through the solution, using the delivery system shown in Figure 1.

4.2.3.4 Analyze the solution as specified by the instrument manufacturer.

4.2.3.5 Calculation — Calculate the concentration of hydrogen fluoride (HF) using the following formula:

$$\text{ppmv HFg} = \frac{(\text{F}^- \text{ ppmw}) \times \text{Vg}}{19.00 \times \text{mg}}$$

Where:

F ⁻ ppm	=	Fluoride concentration in trapped solution.
Vg	=	Volume of collection solution (mL).
mg	=	Moles of SF_6 bubbled through the collecting solution.
19.00	=	Molecular weight of F^- .

The result may not exceed the specification in Section 2 of this Standard.

4.3 Water — This procedure is for the determination of trace moisture (water) in sulfur hexafluoride using a

continuous flowing electrolytic hygrometer. (See Notes 4, and 5.)

4.3.1 Detection Limit — 1.0 ppm (vol/vol) or -76°C (-105°F).

4.3.2 Sample Pressure and Flow — Set in accordance with instrument manufacturer's instructions.

4.3.3 Operation Check — Check the instrument periodically for correct operation. A gas containing a known amount of moisture should be passed through the instrument. Agreement between the hygrometer and the standard should be within their relative accuracies.

4.3.4 Operating Procedure

4.3.4.1 Obtain a continuous flow sample of sulfur hexafluoride source, using a clean, electropolished or passivated stainless steel line which has been purged dry after exposure to ambient moisture. (See Note 5.)

4.3.4.2 After prepurging with a dry gas, allow the sample gas to flow through the sampling system and hygrometer until a stable reading is obtained. The reading may not exceed the specification in Section 2 of this Standard.

4.4 Notes

Note 1: Introduce the calibration standard as many times as necessary to achieve the desired precision.

Note 2: All gases used in the analysis of the sample should not contain more than 10% of the specified value of the component of interest, unless otherwise stated.

Note 3: Observe proper safety procedures for handling and disposing of sulfur hexafluoride (SF_6).

Note 4: The sampling system and hygrometer must be designed to operate at the sample pressure, or the sample pressure must be reduced (by a regulator with a diaphragm of stainless steel or other suitable material) to accommodate the pressure restrictions of the hygrometer.

Note 5: A passivation procedure is described in Metals Handbook, Eighth Edition, Volume 2, ASM International, Metals Park, Ohio.

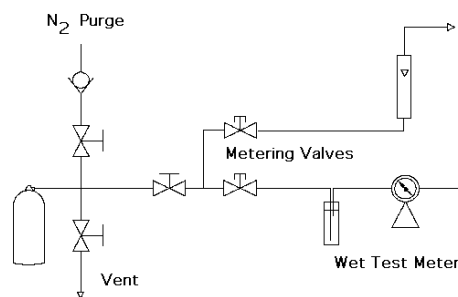


Figure 1