

SEMI C70-1116

SPECIFICATION FOR TUNGSTEN HEXAFLUORIDE

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1 Purpose

1.1 The purpose of this Document is to provide specifications for tungsten hexafluoride (WF_6) that are used in the semiconductor industry.

2 Scope

2.1 This Document covers requirements for tungsten hexafluoride used in the semiconductor industry.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Description

3.1 Tungsten hexafluoride is a colorless gas or colorless liquid. It is shipped as a liquefied gas under its own vapor pressure.

4 Limitations

4.1 None.

5 Referenced Standards and Documents

5.1 *SEMI Standards and Safety Guidelines*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

SEMI C3 — Specifications for Gases

SEMI C10 — Guide for Determination of Method Detection Limits

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

6 Terminology

6.1 Terminology appropriate to this Standard is defined in SEMI C3.

7 Requirements

7.1 Purity and other requirements for tungsten hexafluoride are given in Table 1.

Table 1 Impurity and Other Requirements for Tungsten Hexafluoride (WF_6)

<i>Previous SEMI Reference #</i>	<i>C3.26-0301 (Specification)</i>	<i>C3.52-0200 (Specification)</i>
Grade	99.9%	99.997%
Impurities	Maximum Acceptable Level (ppm) ^{#1}	
Carbon Tetrafluoride (CF_4)	10	1
Carbon Dioxide (CO_2)	--	1
Carbon Monoxide (CO)	--	1
Hydrogen Fluoride (HF)	1000	20
Nitrogen (N_2)	50	5

<i>Previous SEMI Reference #</i>	<i>C3.26-0301 (Specification)</i>	<i>C3.52-0200 (Specification)</i>
Oxygen (O ₂) + Argon (Ar)	50	1
Sulfur Hexafluoride (SF ₆)	10	1
Silicon Tetrafluoride (SiF ₄)	10	1
TOTAL LISTED IMPURITIES	1130	31
Impurities	Maximum Acceptable Level (ppmw)	
Cobalt (Co) and Manganese (Mn)	--	0.01
Lead (Pb)	--	0.02
Chromium (Cr), Zinc (Zn), and Uranium (U)	--	0.05
Calcium (Ca) and Magnesium (Mg)	--	0.10
Nickel (Ni), Copper (Cu), Sodium (Na), and Potassium (K)	--	0.15
Iron (Fe)	--	0.02
Thorium (Th)	--	0.1
Molybdenum (Mo)	--	1

#1 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.

8 Properties of Tungsten Hexafluoride (WF₆)

Table 2 Properties of Tungsten Hexafluoride (for information only)

	<i>Metric Units</i>	<i>US Units</i>
CAS Number	7783-82-6	7783-82-6
Molecular Weight	297.85	297.85
Boiling point at 1 atm	17.1°C	62.8°F
Density of gas at 22.8°C (73°F) and 1 atm	12.9 kg/m ³	0.805 lb/ft ³
Specific gravity of gas	10.8	10.8
Density of liquid at boiling point	3440 kg/m ³	214.8 lb/ft ³

9 Analytical Procedures for Tungsten Hexafluoride, 99.9% Purity

9.1 *Nitrogen and Oxygen + Argon* — This procedure is for the determination of nitrogen and oxygen + argon in tungsten hexafluoride using a gas chromatograph with a thermal conductivity detector.

9.1.1 *Detection Limits* — 10 ppm (mol/mol) nitrogen, and 10 ppm (mol/mol) oxygen + argon.

9.1.2 *Instrument Parameters*

9.1.2.1 *Columns (see Figures 1 and 2)*

- *Column 1* — Porapak® S, 80/100 mesh, 1.5 m (5 ft.) by 6.4 mm (1/4 in.) OD, 5.1 mm (0.2 in.) ID, ss or equivalent.
- *Column 2* — Molecular sieve 5A, 80/100 mesh, 1.8 m (6 ft.) by 4.8 mm (3/16 in.) OD, 3.7 mm (0.147 in.) ID, ss or equivalent.

9.1.2.2 *Column Flow* — 30 mL/min helium.

9.1.2.3 *Sample Volume* — 2 mL.

9.1.2.4 Temperatures

- *Detector* — 70°C.
- *Column* — 40°C.

9.1.3 *Calibration Standard* — 50 ppm (mol/mol) nitrogen, 50 ppm (mol/mol) oxygen, balance helium.

9.1.4 Operating Procedures

9.1.4.1 Determine the times for valve switching and signal changes, and enter into the run table. An example of a run table follows.

Table 3 Example of a Run

<i>Time</i>	<i>Position</i>	<i>Function</i>
0 min.	1	Purge sample through loop. Backflush Porapak [®] column. Connect MS column to the TCD.
1 min.	2	Inject sample onto Porapak [®] column. Allow oxygen + argon and nitrogen to elute from the Porapak [®] column to the MS column. Sequentially elute the oxygen + argon and nitrogen from the MS column to the TCD.
4 min.	1	Backflush Porapak [®] column to vent for 8 minutes.

9.1.4.2 Set the valves in Position 1 (Figure 1).

9.1.4.3 Flow the calibration standard through the 10-port valve. Analyze standard using the conditions described above. Record retention times and peak areas.

9.1.4.4 Flow the sample to be tested through the 10-port valve for 1 minute. Analyze in the same manner as in ¶ 9.1.4.3.

9.1.4.5 The standard is again run as in ¶ 9.1.4.3.

9.1.4.6 Calculate the concentrations of nitrogen and oxygen + argon in the sample, using the formula below. The result may not exceed the specifications in Table 1 of this Standard.

$$\frac{\text{Sample Peak Area}}{\text{Standard Peak Area}} \times \text{Concentration of Standard} = \text{Concentration of Sample} \quad (1)$$

9.2 *Carbon Tetrafluoride, Hydrogen Fluoride, Silicon Tetrafluoride, and Sulfur Hexafluoride* — This procedure is for the determination of these gases in tungsten hexafluoride using infrared spectrophotometry.

NOTE 1: Actual detection limits will be determined by the noise level and the resolution of the spectrometer. One way to reduce the noise level is to use a fourier transform infrared spectrometer (FTIR).

9.2.1 *Detection Limits* — 10 ppm (mol/mol) carbon tetrafluoride, 1000 ppm (mol/mol) hydrogen fluoride, 10 ppm (mol/mol) silicon tetrafluoride and 10 ppm (mol/mol) sulfur hexafluoride.

9.2.2 Instrument Parameters

9.2.2.1 *Cell Path Length* — 10 cm.

9.2.2.2 *Sample Cell Pressure* — 1 atmosphere.

9.2.2.3 Wavenumbers

Table 4 Wavenumber of Chemicals

<i>Component</i>	<i>Wavenumber (cm⁻¹)</i>
Carbon Tetrafluoride (CF ₄)	1283
Hydrogen Fluoride (HF)	4076
Silicon Tetrafluoride (SiF ₄)	1029
Sulfur Hexafluoride (SF ₆)	948

9.2.3 *Calibration Standards* — 1% (mol/mol) hydrogen fluoride in tungsten hexafluoride, 20 ppm (mol/mol) silicon tetrafluoride in nitrogen, 20 ppm (mol/mol) each carbon tetrafluoride and sulfur hexafluoride in nitrogen.

9.2.4 Operating Procedures

9.2.4.1 Flow each calibration gas through the system for 1 minute. Pressurize the cell to 1 atmosphere. Record the absorbance of each calibration standard at the wavenumber listed in ¶ 9.2.2.3.

9.2.4.2 Flow the sample gas through the system for 1 minute. Pressurize the cell to 1 atmosphere. Record the absorbance at each of the appropriate wavenumbers. Calculate the concentration of each gas, using the formula below. The results may not exceed the specification in Table 1 of this Standard.

$$\frac{\text{Measured Absorbance of the Sample}}{\text{Measured Absorbance of the Standard}} \times \frac{\text{Concentration of Standard}}{\text{Concentration of Sample}} = \text{Concentration of Sample} \quad (2)$$

NOTE 2: Tungsten hexafluoride interferes with the direct absorbance reading for silicon tetrafluoride. The absorbance for silicon tetrafluoride is determined by compensating for the interfering absorbance from the tungsten hexafluoride. Compensation is made by subtracting the absorbance contributed to this band by tungsten hexafluoride band at 930 cm⁻¹. This absorbance is halved and subtracted from the total peak absorbance at 1040 cm⁻¹, resulting in the peak absorbance for silicon tetrafluoride.

NOTE 3: Actual detection limits will be determined by the noise level and the resolution of the spectrometer. One way is to reduce the noise level is to use a FTIR.

NOTE 4: 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 5: As tungsten hexafluoride has a low vapor pressure, the cylinder should be kept at room temperature (20°C) for at least 8 hours prior to analysis.

NOTE 6: Observe proper safety procedures for handling and disposing of tungsten hexafluoride.

NOTE 7: Prior to introducing tungsten hexafluoride purge the sample lines and instrument tubing with helium to remove moisture.

NOTE 8: Upon completing the analysis, cap off the manifold and instrument lines or purge the system with helium.