

SEMI C3.52-0200 (Withdrawn 0312) STANDARD FOR TUNGSTEN HEXAFLUORIDE, 99.996% QUALITY

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NOTICE: This Document was balloted and approved for withdrawal in 2012; it has been replaced by SEMI C70.

1 Purpose

1.1 To define the specification and analytical methods and validate the specifications for WF₆.

2 Scope

2.1 This Specification relates to a geometry of 0.5 μ or less (0.35 or 0.5 range) and describes analytical techniques using gas phase and liquid phase analysis.

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, odorless gas or a clear liquid.

4 Specifications

4.1 Quality: 99.996%

<i>Impurities</i>	<i>Maximum Acceptable Level</i>
Oxygen (O ₂) and Argon (Ar)	1 ppm
Nitrogen (N ₂)	5 ppm
Carbon Dioxide (CO ₂)	1 ppm
Carbon Tetrafluoride (CF ₄)	1 ppm
Hydrogen Fluoride (HF)	20 ppm
Silicon Tetrafluoride (SiF ₄)	1 ppm
Sulfur Hexafluoride (SF ₆)	1 ppm
Carbon Monoxide (CO)	1 ppm
TOTAL SPECIFIED IMPURITIES	31 ppm
Cobalt (Co) and Manganese (Mn)	0.01 ppm w
Lead (Pb)	0.02 ppm w
Chromium (Cr), Zinc (Zn), and Uranium (U)	0.05 ppm w
Calcium (Ca) and Magnesium (Mg)	0.10 ppm w
Nickel (Ni), Copper (Cu), Sodium (Na), and Potassium (K)	0.15 ppm w
Iron (Fe)	0.02 ppm w
Thorium (Th)	0.1 ppm w
Molybdenum (Mo)	1 ppm w

5 Physical Constants

	<i>Metric Units</i>	<i>U.S. Units</i>
Molecular Weight	297.85	297.85
Boiling Point	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	

6 Analytical Procedures (see Note 1)

6.1 *Carbon Tetrafluoride, Silicon Tetrafluoride, Carbon Dioxide, and Sulfur Hexafluoride* — This procedure is for the determination of carbon tetrafluoride, silicon tetrafluoride, carbon dioxide, and sulfur hexafluoride in tungsten hexafluoride using a gas chromatograph equipped with a helium ionization detector. A backflush is used in order to protect the detector from the main component.

6.1.1 *Detection Limits* — 0.3 ppm (mole/mole) carbon tetrafluoride, 1 ppm (mole/mole) silicon tetrafluoride, 0.1 ppm (mole/mole) carbon dioxide, and 0.5 ppm (mole/mole) sulfur hexafluoride.

6.1.2 Instrument Parameters

6.1.2.1 Columns

- *Column 1* — 10% Kel F Nr10 on Chromosorb T, 4.0 m (13 ft.) by 3.2 mm (1/8 in.) nickel or equivalent.
- *Column 2* — Preconditioned HAYESEP Q, 60/80 mesh, 3 m (10 ft.) by 3.2 mm (1/8 in.) or equivalent. The column should be treated by repeated WF₆ sample injections at 125°C.

6.1.2.2 *Carrier Flow* — 26 mL/min helium, 6.0 grade.

6.1.2.3 Temperatures

- *Detector* — 50°C.
- *Column Oven* — 50°C.

6.1.2.4 *Sample Volume* — 2 mL.

6.1.2.5 *Calibration Standards* — 1 ppm and 5 ppm (mole/mole) carbon tetrafluoride, carbon dioxide, and sulfur hexafluoride in helium (see Figures 1 and 2). 10 ppm silicon tetrafluoride in helium or permeation device (see Figure 3).

6.1.3 Operating Procedure

6.1.3.1 Attach a suitable pressure regulator to the standard cylinders. Connect the regulator to the dedicated WF₆ handling system which is connected to the chromatographic sampling valve.

6.1.3.2 Purge the sampling lines with the standard for at least one minute.

6.1.3.3 Inject the standard into the gas chromatograph. Record the retention times and peak areas. Order of elution for the above mentioned standard is carbon tetrafluoride, silicon tetrafluoride, carbon dioxide, and sulfur hexafluoride.

6.1.3.4 Inject the WF₆ sample to be analyzed in the same manner as the calibration standard. Record the retention times and peak areas.

6.1.3.5 Compare the average peak areas of the calibration standard to that of the detected peak in tungsten hexafluoride. Calculate the concentration of each impurity using the standard and sample peak areas and the standard concentration. The results may not exceed the specifications.

6.2 *Oxygen and Argon, Nitrogen, and Carbon Monoxide* — This procedure is for the determination of oxygen and argon, nitrogen, and carbon monoxide in tungsten hexafluoride using a gas chromatograph equipped with a helium ionization detector. A backflush is used in order to protect the detector from the main component.

6.2.1 *Detection Limits*

6.2.2 *Instrument Parameters*

6.2.2.1 *Columns*

- *Column 1* — 10% Kel F Nr 10 on Chromosorb T, 4.0 m (13 ft.) by 3.2 mm (1/8 in.) nickel or equivalent.
- *Column 2* — Preconditioned HAYESEP Q, 60/80 mesh, 3 m (10 ft.) by 3.2 mm (1/8 in.) or equivalent. The columns should be treated by repeated WF_6 sample injections at 125°C.
- *Column 3* — Molecular sieve 5 A, 60/80 mesh, 2 m (6 ft.) by 3.2 mm (1/8 in.).

6.2.2.2 *Carrier Flow* — 26 mL/min helium, 6.0 grade.

6.2.2.3 *Temperatures*

- *Detector* — 50°C.
- *Column Oven* — 50°C.

6.2.2.4 *Sample Volume* — 2 mL.

6.2.2.5 *Calibration Standards* — 1 ppm and 5 ppm (mole/mole) oxygen, nitrogen, and carbon monoxide in helium (see Figure 4).

6.2.3 *Operating Procedure*

6.2.3.1 Attach a suitable pressure regulator to the standard cylinder. Connect the regulator to the dedicated WF_6 handling system, which is connected to the chromatographic sampling valve.

6.2.3.2 Purge the sampling lines with the standard for at least one minute.

6.2.3.3 Inject the standard into the gas chromatograph. Switch the valves in order to operate in the same conditions as the WF_6 sample analysis. Record the retention times and peak areas. Order of elution for the above mentioned standard is oxygen and argon, nitrogen, and carbon monoxide.

6.2.3.4 Inject the WF_6 sample in the same manner as the calibration standard. Record the retention times and peak areas.

6.2.3.5 Compare the average peak areas of the calibration standard to that of the detected peak in the tungsten hexafluoride. Calculate the concentration of each impurity using the standard and sample areas and the standard concentration. The results may not exceed the specifications.

6.3 *Hydrogen Fluoride* — This procedure is for the determination of hydrogen fluoride in tungsten hexafluoride using Fourier transform infrared (FTIR) analyzer.

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

6.3.2 *Instrument Parameters*

- cell with CaF_2 windows
- HF band: 4038,8 cm^{-1}
- resolution: 2 cm^{-1}
- path length: 12 cm

6.3.3 *Calibration Standard* — Calibrate with low vapor pressure hydrogen fluoride or permeation device.

6.3.4 *Operating Procedure* — Carefully dry the cell before sampling tungsten hexafluoride. Then purge it with tungsten hexafluoride and record the concentration. When it is steady, that means the line is purged and the true hydrogen fluoride concentration is obtained.