

SEMI C45-0699

SPECIFICATION AND GUIDELINE FOR TETRAETHYLORTHOSILICATE (TEOS)

This Standard was technically approved by the Process Chemicals Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on April 23, 1999. Available at www.semi.org in June 1999; originally published in 1991 as SEMI C7.13. This Document replaces SEMI C7.13 in its entirety

1 Purpose

1.1 The purpose of this Document is to standardize requirements for tetraethylorthosilicate used in the semiconductor industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results. This Document also provides guidelines for grades of tetraethylorthosilicate for which a need has been identified. In the case of the guidelines, the test methods may not have been statistically validated yet.

2 Scope

2.1 The scope of this Document is all grades of tetraethylorthosilicate 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 Limitations

3.1 None.

4 Referenced Standards and Documents

SEMI C1 — Specifications for Reagents

SEMI Suggested Guidelines for Pure Water for Semiconductor Processing

5 Terminology

5.1 None.

6 Physical Property (for information only)

6.1 Not applicable.

7 Requirements

7.1 The requirements for tetraethylorthosilicate for Grade 1 and Tier A are listed in Table 1.

8 Grade 1 Procedures

NOTE 1: Each laboratory is responsible for verifying the validity of the method within its own operation.

8.1 Assay — Analyze the sample by gas chromatography (see SEMI C1, Guidelines for Assay by Wide Bore Column Gas Chromatography). The parameters cited have given satisfactory results.

8.1.1 *Column* — 30 meter × 530 micron fused silica capillary, coated with 5 micron film of DB-1 or equivalent (100% methyl silicone which has been surface bonded and cross linked).

8.1.2 *Column Temperature* — 60°C isothermal for 1 minute, then programmed to 200°C at 20°C/min.

Injector Temperature:	120°C
Detector Temperature:	260°C
Sample Size:	1 µL splitless

Carrier Gas:	Helium at 3 mL/min
Detector:	Thermal Conductivity
Retention Times (min)	
Water	2.0
Ethanol	2.5
TEOS	7.8

8.2 *Color* — Dilute 1.0 mL of platinum-cobalt stock solution (APHA No. 500) to 100 mL with water. Compare this standard (APHA No. 5) with 100 mL of sample in Nessler tubes. View vertically over a white background. The sample must be no darker than the standard.

8.3 *Water* — The cell is dried with a dry nitrogen gas stream for twenty minutes. Reagents are added and a blank (no sample) is run. Add 5 g of sample to the dry coulometric Karl Fischer water analyzer. Results are obtained in ppm by dividing the micrograms of water found in the sample by weight of TEOS in grams.

8.4 *Trace Metals Analysis* — The following method has given satisfactory results in determining trace metal impurities at the specified value for each of the following trace metals: aluminum (Al), arsenic (As), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), and zinc (Zn). Alternate methods may be used as long as appropriate studies demonstrate recovery between 75% to 125% of a known sample spike for half of the value of each specified item.

8.4.1 *Special Reagents* — All water used in the *Trace Metals Analysis* and *Boron Analysis* should be a deionized water meeting the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing for 'attainable' or better.

8.4.1.1 *Mixed Acid* — 1% HF to 2% HNO₃ prepared by dilution of ultra pure acids with appropriate weights of deionized water.

8.4.1.2 *Standards* — Composite standard solutions containing 0, 1, 2, and 4 ppb multi-element standards are prepared by diluting with mixed acid of appropriate weights for ICP-MS. Single-element standards are prepared (Ca, Fe, K, Na) for GFAA.

8.4.2 *Sample Preparation* — In a clean environment, place 100 g of sample in a clean PFA Teflon closed bottle equipped with an inlet and outlet tube and evaporate under a nitrogen purge at approximately 110°C. When dry, close the inlet and the outlet tube and digest the residue at 100°C for 30 minutes with 7 mL of mixed acid solution. Cool and transfer the contents with deionized water. Dilute the transferred contents with deionized water to a final volume of 14 mL.

8.4.3 *Analysis* — Analyze the samples by graphite furnace atomic absorption (GFAA) for sodium, calcium, potassium, and iron. Analyze the sample for all other metallic elements by inductively coupled plasma mass spectrometry (ICP-MS).

8.5 *Boron Analysis* — The following method has given satisfactory results in the determination of trace levels of boron (B). Alternate methods may be used as long as appropriate studies demonstrate recovery between 75% to 125% of a known sample spike for half of the specified value.

8.5.1 *Standard* — Standards of 0, 2, and 4 ppm boron are prepared by dilution of an aqueous boron stock solution with deionized water meeting the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing of 'attainable' or better.

8.5.2 *Sample Preparation* — A 100 g sample of TEOS is extracted with 10 mL of deionized water, that meets the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing for 'attainable' or better, by vigorously shaking the solution for minimum of 10 minutes on a mechanical shaker. Allow the layers to separate and remove the aqueous portion to a separate container.

8.5.3 *Analysis* — The 10 mL water extract is analyzed for boron by inductively coupled plasma atomic emission spectroscopy (ICP-AES) at 249.68 nm.

9 Grade 2 Procedures

9.1 This section does not apply to this chemical.