

SEMI C29-1110

I SPECIFICATIONS AND GUIDE FOR 4.9% HYDROFLUORIC ACID (10:1 v/v)

This Standard was technically approved by the Liquid Chemicals Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on August 27, 2010. Available at www.semi.org in October 2010; originally published June 1999; previously published March 2001.

1 Purpose

1.1 The purpose of this Document is to standardize requirements for 4.9% hydrofluoric acid 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 a guide for grade of 4.9% hydrofluoric acid for which a need has been identified. In the case of the guide, the test methods may not have been statistically validated yet.

2 Scope

2.1 The scope of this Document is grades of 4.9% hydrofluoric acid 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

4.1 SEMI Standards and Safety Guidelines

SEMI C1 — Guide for the Analysis of Liquid Chemicals

SEMI C28 — Specifications for Hydrofluoric Acid

4.2 ASTM Standard¹

ASTM D5127 — Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industry

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

5 Terminology

5.1 None.

6 Physical Property (for information only)

6.1 Not applicable.

7 Requirements and Recommendations

7.1 The requirements for 4.9% hydrofluoric acid for Grades 2 and 3 and recommendations for Tier C are listed in Table 1.

8 Grade 1 Procedures

8.1 This section does not apply to this chemical.

¹ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, <http://www.astm.org>

9 Grade 2 Procedures

9.1 *Non-Metal Impurities* — See SEMI C28 (Specifications for Hydrofluoric Acid) which contains procedures for the following tests:

- Assay
- Chloride
- Phosphate
- Sulfate and Sulfite (as SO₄)

9.1.1 *Analysis of Anions* — Application of the procedures cited above to dilute hydrofluoric acid requires a tenfold increase in the amount of initial sample weight prior to the evaporation steps given in the procedures.

9.2 Trace Metals Analysis

9.2.1 The following method has given satisfactory results in determining metal ion impurities at the values specified for each of the following metals: aluminum (Al), antimony (Sb), arsenic (As), barium (Ba), boron (B), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), vanadium (V), and zinc (Zn). Alternative methods may be used as long as validity has been demonstrated as defined in SEMI C1.

9.3 Special Reagents

9.3.1 *Hydrofluoric Acid, Ultra Pure* — Use hydrofluoric acid specified for low metal ion content.

9.3.1.1 *4.9% Hydrofluoric Acid Solution* — Dilute 20 g of ultra pure hydrofluoric acid to 200 g using water meeting the criteria for Type E1.1 in ASTM D5127.

9.3.1.2 *Water* — The water used for all the dilution, calibration and standards shall meet at a minimum the criteria for Type E1.1 in ASTM D5127 in regard to cation analysis.

9.3.1.3 *Indium Internal Standard* — Make up the internal standard solution to a concentration of 20 µg/mL (ppm) from the appropriate concentrated indium standard solution.

9.3.2 Sample Preparation

9.3.2.1 In a clean environment, place 20.0 g of sample into a tared FEP bottle (30 mL). Add 20 µL of the indium internal standard solution. Run a reagent blank.

9.3.3 Analysis

9.3.3.1 Using the prepared solutions and blanks, analyze sodium, potassium, calcium and iron by graphite furnace atomic absorption (GFAA) and the remaining elements by inductively coupled plasma mass spectrometry (ICP-MS). For calibration, the standards are made up with the 4.9% hydrofluoric acid solution and a final concentration of 20 ng/g of the indium internal standard.

NOTE 1: Analysis of diluted hydrofluoric acid requires the use of special hydrofluoric acid resistant sample introduction systems for ICP-MS. These systems are available from most instrument suppliers.

NOTE 2: Analysis of diluted hydrofluoric acid can produce rapid corrosion of nickel cones commonly used in ICP-MS, platinum cones should be considered as alternative when performing this analysis.

10 Grade 3 Procedures

NOTE 3: The analytical procedures associated with this standard are not intended to be the only acceptable procedure or the best procedure available. The published procedures have been found to meet the required criteria for acceptance of an analytical procedure. Alternate procedures may be used if they meet the same criteria as the published procedures.

10.1 *Non-Metal Impurities* — See SEMI C28, which contains procedures for the following tests:

- Assay
- Chloride
- Phosphate