



SEMI C29-0699

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

These specifications and this guideline were technically approved by the Global Process Chemicals Committee and are the direct responsibility of the North American Process Chemicals Committee. Current edition approved by the North American Regional Standards Committee on April 23, 1999. Initially available on SEMI OnLine May 1999; to be published June 1999. This document replaces SEMI C7.4, C8.4, and C12.3 in their entirety. Originally published in 1990, 1992, and 1995 respectively.

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 guidelines for grades of 4.9% hydrofluoric acid 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 4.9% hydrofluoric acid used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

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

8 Grade 1 Procedures

8.1 This section does not apply to this chemical.

9 Grade 2 Procedures

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

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

Assay

Color (APHA)

Fluosilicic Acid

Chloride

Phosphate

Sulfate and Sulfite (as SO_4)

Arsenic and Antimony (as As)

9.1.1 *Analysis of Anions and Fluosilicic Acid* — 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), arsenic (As), antimony (Sb), barium (Ba), beryllium (Be), bismuth (Bi), boron (B), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), gallium (Ga), germanium (Ge), gold (Au), iron (Fe), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), niobium (Nb), potassium (K), silver (Ag), sodium (Na), strontium (Sr), tantalum (Ta), tin (Sn), titanium (Ti), vanadium (V), zinc (Zn), and zirconium (Zr). Alternate methods may be used as long as appropriate studies demonstrate a recovery between 75 - 125% of a known sample spike for half of the value of each specified element.

9.2.2 Special Reagents

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

9.2.2.2 *4.9% Hydrofluoric Acid Solution* — Dilute 20 g of ultrapure hydrofluoric acid to 200 g using water meeting the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing for “attainable” or better.



9.2.2.3 *"Attainable" Water* — The water used for all the dilution, calibration and standards should meet at a minimum the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing for "attainable" in regard to cation analysis.

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

9.2.3 Sample Preparation

9.2.3.1 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.

9.2.4 Analysis

9.2.4.1 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 2: Analysis of diluted hydrofluoric acid requires the use of special hydrofluoric acid resistant sample introduction systems for inductively coupled plasma mass spectrometry. These systems are available from most instrument suppliers.

NOTE 3: Analysis of diluted hydrofluoric acid can produce rapid corrosion of nickel cones commonly used in inductively coupled plasma mass spectrometry, platinum cones should be considered as alternative when performing this analysis.

10 Grade 3 Procedures

NOTE 4: 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.

NOTE 5: Each laboratory is responsible for verifying the validity of each method within its own operation.

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

Assay

Color (APHA)

Fluosilicic Acid

Chlorides

Phosphates

Sulfate and Sulfite (as SO₄)

10.1.1 *Analysis of Anions and Fluosilicic Acid* — 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.

10.2 Trace Metals Analysis

10.2.1 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), cadmium (Cd), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), gold (Au), iron (Fe), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), potassium (K), silver (Ag), sodium (Na), strontium (Sr), tin (Sn), titanium (Ti), vanadium (V), and zinc (Zn). Alternate methods may be used as long as appropriate studies demonstrate a recovery between 75 - 125% of a known sample spike for half of the value of each specified element.

10.2.2 Special Reagents

10.2.2.1 *Hydrofluoric Acid, Ultrapure* — Use hydrofluoric acid specified for low metal ion content.

10.2.2.2 *4.9% Hydrofluoric Acid Solution* — Dilute 20 g of ultrapure hydrofluoric acid to 200 g using water meeting the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing for "attainable" or better.

10.2.2.3 *"Attainable" Water* — The water used for all the dilution, calibration, and standards should meet at a minimum the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing for "attainable" in regard to cation analysis.

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

10.2.3 Sample Preparation

10.2.3.1 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.

10.2.4 Analysis

10.2.4.1 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.