SEMI C28-0306
SPECIFICATIONS FOR HYDROFLUORIC ACID

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1 Purpose

1.1 The purpose of this document is to standardize requirements for hydrofluoric acid used in the semiconductor industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results.

2 Scope

2.1 The scope of this document is all grades of hydrofluoric acid used in the semiconductor industry.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard 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*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

4.2 *ASTM Standards*[1]

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

4.3 *Other Documents*

Dionex Technical Note 45 — Determination of Trace Anions in Concentrated Hydrofluoric Acid

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

5 Terminology

5.1 None.

6 Physical Property (for information only)

Density at 25°C	1.15 g/mL

7 Requirements

7.1 The requirements for hydrofluoric acid for Grades 1, 2, 3 and 4 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* — Accurately weigh 1.4–1.5 mL of sample in polyethylene weighing bottle, sample, stopper immediately, and reweigh. To 50 mL of water in a plastic vessel, add the sample (loosen the stopper of the polyethylene bottle, and add both the container and the stopper with the sample). Add 0.1 mL of phenolphthalein indicator solution, and titrate with standardized 1 N sodium hydroxide to a slight, pink color.

$$\%\text{Assay} = \frac{\text{mL} \times \text{N of NaOH} \times 2.001}{\text{Weight of sample (g)}}$$

8.1.1 Alternately, potentiometric end point detection can be used.

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8.2 *Chloride* — Add 1.7 mL (2.0 g) of sample to 45 mL of water. Filter, if necessary, through a chloride-free filter. Add 1 mL of nitric acid and 1 mL of silver nitrate reagent solution. Any turbidity produced should be no greater than that produced when 0.01 mg of chloride ion (Cl) is treated as the sample.

8.3 *Nitrate* — Weigh 3.3 g of sample in a white plastic 50 mL beaker and dilute with 4.5 mL of water. To another 50 mL plastic beaker, add 6.5 mL of water and 1 mL of standard nitrate solution containing 0.01 mg of nitrate (NO_3) per mL. To each solution, add 2.5 mL of brucine sulfate reagent solution and cautiously add with stirring 20 mL of sulfuric acid. Allow to stand for 10 minutes. The yellow color in the sample solution should be no greater than that in the standard solution.

8.4 *Phosphate* — Evaporate 9 mL (10 g) of sample to dryness in a platinum or other suitable dish on a steam bath in a hood. Dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow to stand for 2 hours at room temperature. Any blue color should be no greater than that produced when 0.01 mg of phosphate ion (PO_4) is treated like the sample.

8.5 *Sulfate and Sulfite (as SO_4)* — To 18 mL (20 g) of sample in a platinum or other suitable evaporating dish, add about 10 mg of sodium carbonate and 1 mL of 30 hydrogen peroxide. Evaporate to dryness on a steam bath in a hood, wash down the sides of the dish with a small volume of water, and add 3 mL of perchloric acid. Evaporate to about 1 mL, dilute with about 15 mL of water, and add 0.01 mL of phenolphthalein indicator solution. Neutralize with ammonium hydroxide, dilute with water to 20 mL, and add 2 mL of dilute hydrochloric acid (1 + 19) and 2 mL of barium chloride reagent solution. Any turbidity should not exceed that produced by 0.1 mg of sulfate ion (SO_4) in an equal volume of solution containing the quantities of reagents used in the test. Compare 10 minutes after adding the barium chloride to the sample and standard solutions.

NOTE 2: For anion determination, alternate method based upon ion chromatography may be used, see ¶10.2.

8.6 *Trace Metal Analysis* — The following method has given satisfactory results in determining trace metal impurities at the value specified for each of the following trace metals: aluminum (Al), antimony (Sb), arsenic (As), barium (Ba), boron (B), cadmium, (Cd), 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). An alternate method is described in ¶9.2. Any other alternate methods may be used as long as method validation according to SEMI C1 can be demonstrated.

8.6.1 *Special Reagents*

8.6.1.1 *Nitric Acid, Ultra Pure* — Use nitric acid specified for ultra low metal ion content.

8.6.1.2 *2% Nitric Acid Solution* — Dilute 20 mL of ultra pure nitric acid to 1 L using water meeting the criteria for Type E1.1 in ASTM D5127.

8.6.2 *Sample Preparation*

8.6.2.1 In a clean environment, place 250 g of sample in a PTFE evaporating dish. Slowly evaporate on a hot plate, avoiding loss of sample by effervescence or spattering until approximately 2 mL of liquid remains.

NOTE 3: Evaporation typically requires 2 1/2 to 4 hours.

Cool. Add 1 mL of ultra pure, 70% nitric acid. While maintaining volume, carefully warm several minutes to dissolve any residue. Cool. Transfer quantitatively to a 50 mL volumetric flask using 2% nitric acid for rinsing and dilution to volume. Run a reagent blank.

8.6.3 *Analysis*

8.6.3.1 Using the acid sample and reagent blank, analyze all specified elements by plasma emission spectroscopy or preferably by ICP-MS.

9 Grade 2 Procedures

NOTE 4: Each laboratory is responsible for verifying the validity of the method within its own operation.

9.1 *Non-Metal Impurities* — See §8, which contains procedures for the following tests:

- Assay,
- Chloride,
- Nitrate,
- Phosphate, and
- Sulfate and Sulfite (as SO_4).

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