

SEMI C2-95 (Withdrawn 0308) SPECIFICATIONS FOR ETCHANTS

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NOTICE: This Document was balloted and approved for withdrawal in 2008.

1 Preface

1.1 The SEMI Committee on Chemical Reagents began its efforts on etchants in 1979. With this publication, the committee establishes definitions for three major types of etchant mixtures; mixed acid etchants, buffer oxide etchants, and phosphoric etchants. Specifications and analytical procedures are introduced for each type.

1.2 Etchants within all of the requirements can be described as ‘meeting SEMI Specifications.’

1.3 Where an analytical procedure different from that provided is substituted by a supplier or user, the burden of proof is on said supplier or user to confirm the equivalency.

2 Definitions

2.1 All definitions set forth in SEMI C1, are adopted for SEMI C2.

2.1.1 *etchant* — the exact definition of an etchant mixture shall be set forth as the initial paragraph of its Standard. The composition shall be expressed as a ratio of the relative volumes taken of the components in an assigned order. In the expression, all the relative volumes shall be reduced to a ratio of the smallest whole numbers. If a component is absent, its relative volume shall be taken as zero.

2.1.2 *composition* — the content of each component of an etchant shall be expressed on a weight/weight basis of the 100% component (e.g., 100% HF, not 49% HF). Any tolerance allowed for the content of a component shall be expressed on a weight/weight basis of that 100% component.

3 General Procedures and Guidelines to Certain Methods

3.1 The general procedures and guidelines for certain methods set forth in SEMI C1, are adopted for SEMI C2.

3.2 Determination of Nitric Acid by Ultraviolet Absorption Spectrophotometry

3.2.1 *Introduction* — The photometric determination of nitric acid in various etchant mixtures is based on the strong absorption of the nitrate ion in the ultraviolet region. For this spectral region fused silica (quartz) cells are required.

3.2.2 *Preparation of Calibration Curve* — Secure a bottle of nitric acid (70%) and determine its assay in duplicate. Accurately weigh 2.8 to 3.0 g dilute with 100 mL of water, add 10 drops of phenolphthalein indicator solution to each solution, and titrate with standardized 1 N sodium hydroxide solution to a pink end-point.

$$\% \text{ Nitric Acid (w/w)} = \frac{\text{mL} \times N \text{ of NaOH} \times 6.302}{\text{Weight of Sample (g)}} \quad (1)$$

If the results do not differ by more than 0.2%, average them. Otherwise, repeat the analysis.

3.2.3 In five previously tared weighing bottles, accurately weigh about 1.0, 1.2, 1.4, 1.6 and 1.8 g of the previously assayed 70% nitric acid. Calculate the weight, in grams, of 100% nitric acid present in each of these five standards. Quantitatively transfer each standard to a separate 100 mL volumetric flask, dilute to volume with water, and mix thoroughly.

3.2.4 Following the manufacturer’s directions, ready a spectrophotometer and set the wavelength to 302 nm. Measure the absorbance of each standard versus water using a 1 cm fused silica cell. On (linear) coordinate graph paper plot the absorbance versus the weight, in grams, of 100% nitric acid for each standard. Draw the best-fitting straight line through the points.

3.2.5 *Application to Acid Etch Mixtures* — Proceed as given under the Standard for the relevant etchant.

4 Reagent and Standard Solutions

4.1 The reagent and standard solutions set forth in SEMI C1, are adopted for SEMI C2.

4.2 *Acetate Buffer for Hydrofluoric Acid Determination* — Dissolve 106 g of sodium acetate trihydrate, $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$, and 137 g of ammonium acetate, $\text{CH}_3\text{COOH} \cdot \text{NH}_4$, in about 700 mL of water. Add 5.1 mL of glacial acetic acid, adjust to pH 5.5-6.6 with dilute ammonium hydroxide (10% NH_3) or dilute acetic acid (20%), and dilute to 1000 mL with water.

4.3 *Aluminum Chloride Standard Solution Preparation* — Dissolve 40.0 g of aluminum chloride hexahydrate, $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$, in about 700 mL of water and 0.5 mL of hydrochloric acid contained in a 1000 mL volumetric flask; dilute to volume with water, and mix thoroughly.

4.3.1 *Standardization* — Transfer 1.5 mL of hydro-fluoric acid to a 250 mL polyethylene beaker containing 50 mL of water. Add 10 drops of phenolphthalein indicator solution. With magnetic stirring, titrate with standardized 1N sodium hydroxide solution to a pink endpoint. Record the volume of base delivered (A). Add 25 mL of Acetate Buffer for Hydrofluoric Acid Determination and 1 g of Eriochrome Cyanine R indicator mixture; mix thoroughly. With magnetic stirring, titrate with the aluminum chloride solution to a pink-purple endpoint. Record the volume of this solution delivered (B).

$$\text{HF Factor} = \frac{A \times N \text{ of NaOH} \times 2.001}{B \text{ of AlCl}_3 \text{ soln.}} \quad (2)$$

4.4 *Eriochrome Cyanine A Indicator Mixture* — Using a mortar and pestle, thoroughly grind and mix 0.1 g of Eriochrome Cyanine R (C.I. 43820) with 100 g of potassium nitrate.

5 Safety

5.1 Because of the continuing evolution of safety precautions, it is impossible in this publication to provide definite statements relating to the safe handling of individual etchants. The user is referred to product labels, product and safety data sheets, government regulations, and other relevant literature.

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