SEMI C20-0213 (Reapproved 1218)
SPECIFICATION AND GUIDE FOR AMMONIUM FLUORIDE 40%

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 17, 2018. Available at www.semiviews.org and www.semi.org in December 2018; originally published in 1978; previously published February 2013.

1 Purpose

1.1 The purpose of this Standard is to standardize requirements for ammonium fluoride 40% used in the semiconductor industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results. This Standard also provides guidelines for grades of ammonium fluoride 40% 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 ammonium fluoride 40% 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

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

5 Terminology

5.1 None.

6 Properties

Table 1 Properties of Ammonium Fluoride 40%

Chemical Formula	NH4F
CAS Number	12125-01-8
Density at 25°C	1.11 g/mL

7 Requirements and Suggested Values

7.1 The requirements for ammonium fluoride 40% for Grade 1, and suggested values for Tiers A and B are listed in Table 2.

8 Grade 1 Procedures

8.1 *Assay* — Accurately weigh 3 g of sample in a polyethylene weighing bottle. Transfer with water to a polyethylene beaker and dilute to 100 mL. Add 40 mL of neutralized formaldehyde solution and stir magnetically for 15 minutes or allow to stand for ½ hour. Titrate with standardized 1 N sodium hydroxide to a slight pink color.

$$\%\ \text{Assay} = \frac{\text{Volume of NaOH (mL)} \times \text{Normality of NaOH} \times 3.704}{\text{Mass of Sample (g)}} \quad (1)$$

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NOTE 1: *Neutralized formaldehyde solution:* Dilute 20 mL of 37% formaldehyde solution with 20 mL of water, add 0.1 mL of phenolphthalein indicator solution, and titrate with 0.1 N sodium hydroxide to a slight pink color.

8.2 *pH of a 1% Solution* — Dilute 2.5 mL (2.8 g) of sample to 100 mL with carbon dioxide- and ammonia-free water. Using a pH meter determine the pH at 25°C 1 to 2 minutes after immersion of electrodes. The pH range 6.0 to 7.5 corresponds to 0.2% free hydrogen fluoride (HF) to 0.2% ammonium hydroxide (NH_4OH) in the original solution.

8.3 *Chloride* — Mix 2.2 mL (2.5 g) of sample with 20 mL of water containing 0.5 g of boric acid. Add 1 mL of nitric acid and 1 mL of silver nitrate reagent solution and allow to stand for 5 minutes. Any turbidity produced shall be no greater than that produced when 0.01 mg of chloride ion (Cl) is treated as the sample.

8.4 *Nitrate* — To 2 mL of water in a white plastic beaker, add 0.9 mL (1 g) of sample, 1 mL of sulfuric acid, 0.05 mL of 0.01 N (0.002 M) potassium permanganate, mix, and decolorize with sodium arsenite reagent solution (see SEMI C1). Dilute to 50 mL with brucine sulfate reagent solution (see SEMI C1) and mix. Heat the solution in a preheated (boiling water) bath for 10 minutes. Cool rapidly in an ice bath to room temperature. The yellow color of the sample shall be no greater than that produced when 0.01 mg of nitrate ion (NO_3) is treated as the sample.

8.5 *Phosphate* — To a 9 mL (10 g) sample in a platinum dish, add 1 mL of sodium carbonate reagent solution and 40 mL of nitric acid. Evaporate carefully, to prevent spattering, to near dryness. Cool, wash down the sides of the dish with 5 mL of nitric acid, and evaporate to dryness. Repeat the evaporation with nitric acid two more times to ensure complete removal of all the fluoride. To the cooled dish, add 25 mL of 0.5 N sulfuric acid to dissolve the residue, warming if necessary. Cool, transfer to a color comparison tube, and add 1 mL of ammonium molybdate reagent solution (see SEMI C1), 1 mL of p-(methylamino)phenol sulfate reagent solution(see SEMI C1) and allow to stand at room temperature for 2 hours. Any blue color shall be no greater than that produced when 0.01 mg of phosphate ion (PO_4) is treated as the sample.

8.6 *Sulfate* — To 23 mL (25 g) of sample, add 10 mL of sodium carbonate reagent solution (see SEMI C1) and heat gently until sample has been volatilized. To the residue add 5 mL of hydrochloric acid and evaporate to dryness in a hood. Dissolve the residue in 10 mL of water and 1 mL of dilute hydrochloric acid (1 + 19); filter if necessary. Add 1 mL of barium chloride reagent solution (see SEMI C1) mix, and allow to stand for 10 minutes. Any turbidity developed shall be no greater than that produced when 0.05 mg of sulfate ion (SO_4) solution is treated as the sample.

8.7 *Arsenic and Antimony (as As)* — To 30 mL (33 g) in a fluoroplastic beaker, add 20 mL of nitric acid and 5 mL of hydrochloric acid, and evaporate to dryness in a sand bath in a hood. Completely volatilize the ammonium fluoride, but do not bake. Add 10 mL of water and 5 mL of sulfuric acid and evaporate to dense fumes of sulfur trioxide. Cool, cautiously add 10 mL of water, and again evaporate to dense fumes of sulfur trioxide. Cool, and cautiously wash into a generator flask with water to make a volume of 35 mL. Proceed as described in SEMI C1 under section, Trace Arsenic (and Antimony) Determination, starting with the sentence that begins, "Swirl the flask...." Any red color in the silver diethyldithiocarbamate solution of the sample shall be no greater than that of the standard containing 0.001 mg of arsenic (As).

8.8 *Trace Element Contents* — By a suitable emission spectrometry procedure (see SEMI C1) and determine for each of the specified trace elements that its content is not greater than the stated specification limit.

9 Grade 2 Procedures

9.1 This section does not apply to this chemical.

10 Grade 3 Procedures

10.1 This section does not apply to this chemical.

11 Grade 4 Procedures

11.1 This section does not apply to this chemical.

12 Grade 5 Procedures

12.1 This section does not apply to this chemical.

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