

SEMI C18-0301

SPECIFICATION FOR ACETIC ACID

This specification was technically approved by the Global Process Chemicals Committee and is the direct responsibility of the North American Process Chemicals Committee. Current edition approved by the North American Regional Standards Committee on October 17, 1999. Initially available at www.semi.org February 2001; to be published March 2001. This document replaces SEMI C1.1 in its entirety. Originally published in 1978; previously published June 1999.

1 Purpose

1.1 The purpose of this document is to standardize requirements for acetic 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 acetic 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 acetic acid used in the semiconductor industry.

2.2 These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

3 Limitations

3.1 None.

4 Referenced Standards

4.1 *SEMI Standards*

SEMI C1 — Specifications for Reagents

4.2 *ASTM Standards*¹

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

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

5 Terminology

5.1 None.

6 Physical Property (for information only)

Density at 25°C	1.05 g/mL
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NOTE 2: This material freezes at about 16°C.

¹ American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555. Website: www.astm.org

7 Requirements

7.1 The requirements for acetic acid for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

NOTE 3: Each laboratory is responsible for verifying the validity of the method within its own operation.

8.1 *Assay* — Place 15 mL (15.8 g) of sample in a 20 × 150 mm test tube in which is centered an accurate thermometer. The sample tube is centered by one or more stoppers in an outer tube about 38 × 20 mm. Cool the entire apparatus, without stirring, in a bath of shaved or crushed ice and sufficient water to immerse the outer tube above the level of the acetic acid. When the thermometer temperature is about 13°C stir to induce freezing and read the thermometer every half minute. The temperature that remains constant for 1 to 2 minutes is the freezing point and should not be below 16°C, indicating not less than 99.7% as CH₃COOH.

8.2 *Color* — Dilute 2.0 mL of platinum-cobalt stock solution (APHA No. 500) to 100 mL with water. Compare this standard (APHA No. 10) with 100 mL of sample in Nessler tubes. View vertically over a white background. The sample must be no darker than the standard.

8.3 *Residue after Evaporation* — Evaporate 95 mL (100 g) of sample in a tared porcelain or silica dish to dryness on a steam bath in a hood. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (see SEMI C1, Section 3.3, Determination of Residue After Evaporation).

8.4 *Solubility in Water* — Mix 10 mL of sample with 30 mL of water. Allow to stand 1 hour. The solution should be as clear as an equal volume of water.

8.5 *Chloride* — Dilute 9.5 mL (10 g) of sample with 10 mL of water. Add 1 mL of silver nitrate reagent solution and evaporate to dryness on a steam bath in a hood. Dissolve the residue in 0.5 mL of ammonium hydroxide, dilute with 20 mL of water and add 1.5 mL of nitric acid. Any turbidity produced should be no greater than that produced when 0.01 mg of chloride ion (Cl) is treated as the sample.

8.6 Phosphate — Evaporate 9.5 mL (10 g) of sample to dryness on a steam bath in a hood. Dissolve the residue, warming if necessary, 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 at room temperature for 2 hours. Any blue color should be no greater than that produced when 0.01 mg of phosphate ion (PO_4) is treated as the sample.

8.7 Substances Reducing Dichromate — To 10 mL (10.5 g) of sample, add 1.0 mL of 0.1 N (0.017 M) potassium dichromate solution and cautiously add 10 mL of sulfuric acid. Cool the solution to room temperature and allow to stand for 30 minutes. Add 50 mL of water slowly and cautiously with continual swirling, allow to cool, and then add 1 mL of freshly prepared potassium iodide reagent solution. Titrate the liberated iodine with 0.1 N (0.1 M) thiosulfate solution using starch as the indicator. Compare the volume of thiosulfate solution required with that for a 10 mL water blank prepared in parallel with the sample. The difference between the titrations for sample and blank should be no greater than 0.40 mL.

8.8 Substances Reducing Permanganate — Dilute 40 mL (42 g) of sample with 10 mL of water. Cool to 15°C, add 0.30 mL of 0.1 N (0.02 M) potassium permanganate, and allow to stand at 15°C for 10 minutes. The pink color should not be entirely discharged.

8.9 Sulfate — To 95 mL (100 g) of sample, add 10 mL of sodium carbonate reagent solution 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, mix and allow to stand for 10 minutes. Any turbidity developed should be no greater than that produced when 0.05 mg of sulfate ion (SO_4) is treated as the sample.

8.10 Arsenic and Antimony (as As) — To 190 mL (200 g) of sample in a 400 mL beaker, add 5 mL of nitric acid and 5 mL of sulfuric acid and evaporate to dense fumes of sulfur trioxide in a hood. 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 the General Method for Arsenic (and Antimony) under SEMI C1, Section 3.4.5, starting with the sentence which begins: "Swirl the flask...." Any red color in the silver diethyldithiocarbamate solution of the sample should be no greater than that of the standard containing 0.001 mg of arsenic (As).

8.11 Acetic Anhydride — Place 52.2 g (50 mL) of sample in a 250 mL titration flask. In a second flask

place 50 mL of glacial acetic acid known to be free from acetic anhydride (see NOTE 3). Into each flask pipet 10 mL of 1.0% solution of 4, 4' -methylenedianiline (4, 4' -diaminodiphenylmethane) in glacial acetic acid (see NOTE 4) and add 0.10 mL of 1.0% solution of crystal violet in glacial acetic acid. Titrate each solution with a 0.1 N solution of perchloric acid in glacial acetic acid (see NOTE 5) to a green endpoint. Subtract the volume for the titration of the sample from the volume for the other titration. One mL of 0.1 N perchloric acid corresponds to 0.0194% (CH_3CO)₂O for a 50 mL sample.

NOTE 4: Glacial acetic acid suspected of containing anhydride may be purified by adding 0.50 mL of water per 100 mL and digesting overnight in a glass-stoppered flask on the steam bath. If this acid is used for comparison in the above test, 0.25 mL of water should be added to the flask containing the test sample just before the endpoint, because water affects the indicator change slightly.

NOTE 5: Dissolve 2.50 g of 4, 4' -methylenedianiline (colorless or only slightly colored) in glacial acetic acid to make 250 mL. Protect the solution from light.

NOTE 6: Slowly add 4.5 mL of 70% perchloric acid to about 400 mL of glacial acetic acid and dilute with glacial acetic acid to 500 mL. Standardize against potassium hydrogen phthalate in glacial acetic acid solution using crystal violet as indicator.

8.12 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), boron (B), calcium (Ca), chromium (Cr), copper (Cu), gold (Au), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), and zinc (Zn). Alternate methods may be used as long as appropriate studies demonstrate recovery between 75 - 125% of a known sample spike for half of the value of each specified item.

8.12.1 Special Reagents

8.12.1.1 Solution A — Glycerol (ACS Reagent Grade) 10 g, Adipic Acid (99+%) 1 g, EDTA Acid (ACS Reagent Grade) 0.1 g. Dilute to 1 L using water meeting the criteria for Type E1 in ASTM D5127.

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

8.12.1.3 2% Nitric Acid Solution — Dilute 10 mL of ultra pure nitric acid to 1 L using water meeting the criteria for Type E1 in ASTM D5127.

8.12.2 Sample Preparation

8.12.2.1 In a clean environment, place 100 grams of sample in a PTFE dish. Add 50 mL of solution A. Slowly evaporate on a hot water bath avoiding loss of