SEMI C30-0218
SPECIFICATION FOR HYDROGEN PEROXIDE

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 18, 2017. Available at www.semiviews.org and www.semi.org in February 2018; originally published in 1978; previously published November 2010.

1 Purpose

1.1 The purpose of this Document is to standardize requirements for hydrogen peroxide 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 grades of hydrogen peroxide 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

4.2 *ASTM Standard*[1]

ASTM D5127 — Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries

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

5 Terminology

5.1 *Abbreviations and Acronyms*

5.1.1 *TOC* — total oxidizable carbon

6 Properties (for information only)

Table 1 Properties of Hydrogen Peroxide

Chemical Formula	H_2O_2
CAS Number	7722-84-1
Density at 25°C (at 31% w/w)	1.11 g/mL

7 Requirements

7.1 The requirements for hydrogen peroxide for Grades 1, 2, 3, 4, and 5 are listed in Table 3.

[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, http://www.astm.org

Documents Document Sharing Hub
Full document details: SEMI C30-0218

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 about 1 mL of sample in a tared 100 mL volumetric flask, dilute to volume with water, and mix thoroughly. Transfer exactly 20.0 mL of this solution to a 250 mL conical flask, add 20 mL of dilute sulfuric acid (1 + 15), and titrate with standardized 0.1 N (0.02 M) potassium permanganate to a pink color that persists for 15 seconds.

$$\%\text{Assay} = \frac{\text{mL} \times \text{N of } KMnO_4 \times 8.500}{\text{Weight of sample (g)}}$$

8.2 *Total Oxidizable Carbon (TOC)*

8.2.1 *Equipment*

8.2.1.1 TOC analyzer capable of analyzing TOC in water.

8.2.1.2 Platinum sheet 1 × 1 inch, heated in a muffle oven at 800°C for 15 minutes.

8.2.2 *Special Reagents*

8.2.2.1 *Water* — The water used for all of the dilution, calibration, and standards shall meet, at a minimum, the criteria for Type E1.1 in ASTM D5127 in regard to TOC analysis.

8.2.2.2 *1000 µg/mL TOC Standard (prepare fresh weekly)* — Weigh accurately 0.2128 g of potassium acid phthalate into a 100 mL volumetric flask, add water, shake to dissolve, dilute to volume, and mix well. Lower standards (prepare fresh daily) can be made by diluting an aliquot of the 1000 µg/mL to the appropriate volume.

8.2.3 *Sample Preparation* — Weigh 50 g of hydrogen peroxide, to the nearest 0.01 g, into a 400 mL beaker. Add the platinum sheet to the sample, cover the beaker with a Teflon watch glass, and allow the reaction to go overnight (12 hours minimum). Transfer the solution to a 50 mL volumetric flask, and dilute with water meeting the criteria for Type E1.1 in ASTM D5127 to a final volume of 50 mL.

8.2.4 *Analysis* — Using the prepared solutions, analyze TOC by the TOC analyzer after the instrument has been calibrated with 0, 4, 10, and 20 µg/mL of TOC standards.

8.3 *Chloride* — Dilute 4.5 mL (5 g) of sample with 15 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 (see SEMI C1). 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 *Sulfate* — To 9 mL (10 g) of sample, add 10 mL of sodium carbonate reagent solution (see SEMI C1) 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 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) is treated as the sample.

8.5 *Phosphate* — Evaporate 4.5 mL (5 g) of sample to dryness on the steam bath and dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution (see SEMI C1) and 1 mL of p-(methylamino)phenol sulfate reagent (see SEMI C1) solution. Allow to stand for 2 hours at room temperature. Any blue color shall not exceed that produced in a standard of equal volume containing 0.01 mg of phosphate ion (PO_4) and the quantities of reagents used in the sample.

8.6 *Arsenic and Antimony (as As)* — To 180 mL (200 g) of sample in a 400 mL beaker, add 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 General Method for Arsenic (and Antimony) in SEMI C1, starting with the first sentence which begins: "Swirl the flask...." Any red color in the silver diethyldithiocarbamate solution from the sample shall be no greater than that of the standard containing 0.002 mg of arsenic (As).

8.7 *Trace Metal Analysis*

8.7.1 The following method has given satisfactory results in determining metal ion impurities at the values specified for each of the following metals: aluminum (Al), boron (B), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), lead

Documents
Document Sharing Hub
Full document details: SEMI C30-0218

(Pb), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), and zinc (Zn). Alternative methods may be used as long as method validation as per SEMI C1 has been demonstrated.

8.7.2 *Special Reagents*

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

8.7.2.2 *Hydrochloric Acid, Ultra Pure* — Use hydrochloric acid specified for ultra low metal ion content.

8.7.3 *Sample Preparation*

8.7.3.1 *Sodium and Potassium* — In a clean environment, place 10.0 mL of water in a clean platinum crucible. Slowly add 5.0 mL of hydrogen peroxide. Allow to stand at room temperature until the effervescence ceases (approximately 20 minutes). Swirl. If swirling does not produce any more bubbles, proceed with analysis. If bubbles remain, allow to stand for 5 minutes and repeat the swirl until no bubbles are produced. Run a water blank.

8.7.3.2 *Other Elements* — Two separate samples of hydrogen peroxide are acidified to 2% with hydrochloric acid for the analysis of tin and two additional samples are acidified to 2% with nitric acid for the analysis of the remainder of the (specified elements). Standard additions of tin to the matrix acidified with hydrochloric acid and standard additions of other specified elements to the matrix acidified with nitric acid are added to determine the response for each element in the peroxide matrix. Aqueous acidified standards for tin in 2% hydrochloric acid and other specified elements in 2% nitric acid shall be used to determine instrument responses.

8.7.4 *Analysis*

8.7.4.1 Using the solution from ¶ 8.7.3.1, analyze for specified group 1 elements by flame atomic absorption spectroscopy. All other elements analyzed by plasma emission spectroscopy using the solutions from ¶ 8.7.3.2.

9 Grade 2 Procedures

9.1 *Assay* — See ¶ 8.1.

9.2 *TOC* — See ¶ 8.2.

9.3 *Anions*

9.3.1 The following method has given satisfactory results in determining anion impurities at the values specified for each of the following anions: chloride (Cl), phosphate (PO_4), nitrate (NO_3), and sulfate (SO_4). Alternative methods may be used as long as method validation as per SEMI C1 has been demonstrated.

9.3.2 *Special Reagents*

9.3.2.1 *Eluent* — Prepare an eluent solution that is 2.2 mM sodium carbonate (Na_2CO_3) and 0.75 mM sodium bicarbonate ($NaHCO_3$) in water meeting the criteria for Type E1.1 in ASTM D5127. Store eluent under a helium gas blanket.

9.3.2.2 *Regenerant* — Prepare a 0.025 N sulfuric acid (H_2SO_4) in deionized water meeting the criteria for Type E1.1 in ASTM D5127.

9.3.2.3 *Potassium Carbonate Solution* — Prepare a solution containing 500 mg of reagent grade potassium carbonate (K_2CO_3) into 100 mL of water meeting the criteria for Type E1.1 in ASTM D5127.

9.3.3 *Sample Preparation*

9.3.3.1 In a clean environment, place 40 g of sample into a clean beaker. Add 1 mL of a 5 mg/mL potassium carbonate solution and evaporate carefully (at 100°C) to a volume of 0.5 mL. Dilute with water meeting the criteria for Type E1.1 in ASTM D5127 to a final volume of 20 mL.

NOTE 2: For safety purposes, a decomposition aid such as a platinum strip should be used.

9.3.4 *Analysis*

9.3.4.1 Using the prepared solutions and blanks, analyze chloride, nitrate, phosphate, and sulfate by ion chromatography. Run a reagent blank. Samples and reagent blanks shall be prepared and analyzed in triplicate.

9.3.4.2 *Columns* — Precolumn shall be AG4-A (Dionex) or equivalent and Separation column shall be AS4-A (Dionex) or equivalent.

Documents Document Sharing Hub

Full document details: SEMI C30-0218