

SEMI PV36-0316

SPECIFICATION FOR HYDROGEN PEROXIDE USED IN PHOTOVOLTAIC APPLICATIONS

This Standard was technically approved by the Photovoltaic - Materials Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on December 4, 2015. Available at www.semiviews.org and www.semi.org in March 2016; originally published September 2012.

1 Purpose

1.1 The purpose of this Document is to standardize requirements for hydrogen peroxide used in the photovoltaic (PV) industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results (see Appendix 1 for anion validation).

2 Scope

2.1 The scope of this Document is grades of hydrogen peroxide used in the PV 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

SEMI C35 — Specifications and Guideline for Nitric Acid

4.2 ASTM Standard¹

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 of Hydrogen Peroxide

Table 1 Properties of Hydrogen Peroxide

Chemical Formula	H ₂ O 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 PV Grades 1 and 2 are found in Table 4.

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>

8 PV Grade 1 Procedures

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 20 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.

$$\% \text{Assay} = \frac{\text{Volume of KMnO}_4 (\text{mL}) \times \text{Normality of KMnO}_4 \times 8.500}{\text{Mass of sample (g)}} \quad (1)$$

8.2 Total Oxidizable Carbon (TOC)

8.2.1 Equipment

8.2.1.1 TOC analyzer capable of analyzing TOC in 31% H₂O₂. Validated instrumentation includes TOC analyzer using a high temperature platinum catalyst.

8.2.2 Special Reagents

8.2.2.1 Water — The water used for all of the dilutions, calibrations, and standards shall be the appropriate grade of 18.2 Ω·cm water that meets the criteria for Type E1.2 in ASTM D5127.

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 Analysis

8.2.3.1 Analyze by directly injecting the 31% H₂O₂ into the TOC analyzer after the instrument has been calibrated with 0, 5, 10, and 30 µg/mL of TOC standards.

8.3 Anions

8.3.1 The following method has given satisfactory results in determining anion impurities at the values specified for each of the following anions: chloride (Cl), phosphate (PO₄) and sulfate (SO₄). Alternative methods may be used as long as validity is demonstrated as defined in SEMI C1, Method Validation.

8.3.2 Special Reagents

8.3.2.1 Eluent — Prepare an eluent solution that is 100 mm potassium hydroxide (KOH) in deionized water meeting the criteria for Type E1.2 in ASTM D5127. Prepare second solution consisting of deionized water only meeting the criteria for Type E1.2 in ASTM D5127. Store eluents under a helium gas blanket.

8.3.3 Standard Preparation

8.3.3.1 Prepare the following calibration standard by spiking deionized water meeting the criteria for Type E1.2 in ASTM D5127 with appropriate amounts of individual anion standards: Chloride 200 ppb; Phosphate 1500 ppb; Sulfate 3000 ppb.

8.3.4 Analysis

8.3.4.1 Gradient — Using the prepared eluent solutions, design a gradient as follows, flow rate shall remain 2 mL/min. throughout analysis:

Table 2 Gradient for Anion Analysis

Pump	Time (min)	[mm KOH]
isocratic	0–5	9
ramp	5–7.1	40
ramp	7.1–26	47

8.3.4.2 *Columns* — Precolumn shall be AG15 4 mm (Dionex) or equivalent, separation column shall be AS15 4 mm (Dionex) or equivalent, concentrator column shall be UTAC-LP1 4 mm (Dionex) or equivalent.

8.3.4.3 Load 0.5 mL standard then 0.5 mL water meeting the criteria for Type E1.2 in ASTM D5127 onto concentrator column. Using the prescribed eluent gradient analyze chloride, phosphate, and sulfate in neat 31% H₂O₂ by ion chromatography.

8.4 *Trace Metals Analysis*

8.4.1 The following method has given satisfactory results in determining metal ion impurities at the values specified for each of the following metals: aluminum (Al), 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). Alternative methods may be used as long as method validation as per SEMI C1 has been demonstrated.

8.4.2 *Special Reagents*

8.4.2.1 *Nitric Acid, Ultra Pure* — Use nitric acid of a grade equivalent or better than SEMI Grade 3 (see SEMI C35).

8.4.2.2 *1% Nitric Acid Solution* — Dilute 10 mL of ultra-pure nitric acid to 1 L of water that meets the criteria for Type E1.2 in ASTM D5127.

8.4.2.3 *Water* — The water used for all of the dilutions, calibrations, and standards shall be the appropriate grade of 18.2 Ω·cm water meeting the criteria for Type E1.2 in ASTM D5127.

8.4.2.4 *Indium Internal Standard* — Make up the indium internal standard solution to a concentration of 20 µg/mL (ppm) from the appropriate concentrated indium standard solution.

8.4.3 *Sample Preparation*

8.4.3.1 In a clean environment, place 200 g of sample into a tared FEP bottle and add 20 µL of the indium internal standard.

8.4.4 *Analysis*

8.4.4.1 Using the prepared solutions and b blanks, analyze the aforementioned metals by inductively coupled plasma-mass spectrometry (ICP-MS) using indium as an internal standard. For calibration, the standards are made up with the 1% nitric acid solution and the indium internal standard such that the final concentration is 2 ng/g of indium. Run a reagent blank.

9 **PV 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), nitrate (NO₃), phosphate (PO₄), and sulfate (SO₄). Alternative methods may be used as long as validity is demonstrated as defined in SEMI C1, Method Validation.

9.3.2 *Special Reagents*

9.3.2.1 *Eluent* — Prepare an eluent solution that is 100 mM potassium hydroxide (KOH) in deionized water meeting the criteria for Type E1.2 in ASTM D5127. Prepare second solution consisting of deionized water only meeting the criteria for Type E1.2 in ASTM D5127. Store eluents under a helium gas blanket.

9.3.3 *Standard Preparation*

9.3.3.1 Prepare the following calibration standard by spiking deionized water meeting the criteria for Type E1.2 in ASTM D5127 with appropriate amounts of individual anion standards: Chloride 200 ppb; Nitrate 400 ppb; Phosphate 200 ppb; Sulfate 200 ppb.