SEMI C41-0618
SPECIFICATION AND GUIDE FOR 2-PROPANOL

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NOTICE: Paragraphs entitled 'NOTE:' are not an official part of this Standard or Safety Guideline and are not intended to modify or supersede the official Standard or Safety Guideline. These have been supplied by the global technical committee to enhance the usage of the Standard or Safety Guideline.

1 Purpose

1.1 The purpose of this Document is to standardize requirements for 2-propanol 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 covers all grades of 2-propanol used in the semiconductor industry.

2.2 The VLSI grade purity level is typically required by semiconductor devices with geometries of 0.8 to 1.2 microns.

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 Referenced Standards and Documents

3.1 *SEMI Standards and Safety Guidelines*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

3.2 *ASTM Standard*[1]

ASTM D5127 — Standard 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.

4 Terminology

4.1 None.

5 Physical Property (for information only)

Density at 25°C	0.78 g/mL
Boiling Point	82.3°C

6 Requirements

6.1 The requirements for 2-propanol for Grades 1, 2, 3, 4 and VLSI Guidelines, are listed in Table 1.

7 Grade 1 Procedures

NOTE 1: Each laboratory is responsible for verifying the validity of the method within its own operation.

7.1 *Assay* — Analyze the sample by gas chromatography (see SEMI C1, 'Assay by Wide Bore Column Gas Chromatography'). The parameters cited have given satisfactory results.

[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

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- Column: 30 meter × 530 micron I.D. fused silica capillary, coated with 5 micron film of DB-Wax or equivalent (100% methyl silicone which has been surface bonded and cross linked).
- Column Temperature: 40°C isothermal for 5 minutes, then programmed to 200°C at 10°C/min.

Injector Temperature:	150°C
Detector Temperature:	250°C
Sample Size:	0.2 µL splitless
Carrier Gas:	Helium at 3 mL/min
Detector:	Thermal Conductivity
Approximate Retention Times (min):	
Acetone	4.5
2-Propanol	5.0

7.2 *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.

7.3 *Acidity* — To 25 mL of water in a glass-stoppered flask, add 10 mL of sample and 0.1 mL of phenolphthalein indicator solution. Add 0.01 N sodium hydroxide until a slight pink color persists after shaking for one-half minute. Add 64 mL (50 g) of the sample, mix well, and titrate with 0.01 N sodium hydroxide until the pink color is reproduced. Not more than 1.0 mL of the sodium hydroxide solution should be required.

7.4 *Alkalinity* — Add 128 mL (100 g) of sample to 25 mL of water and mix well. Add 0.05 mL of methyl red indicator solution. Titrate with 0.01 N hydrochloric acid until a slight pink color is produced. Not more than 1.0 mL of the hydrochloric acid should be required.

7.5 *Residue after Evaporation* — Evaporate 256 mL (200 g) of sample to dryness. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (see SEMI C1, 'Residue after Evaporation').

7.6 *Water* — Add 25 mL of methanol to a dry titration flask and add Karl Fischer (KF) reagent to a visually or electrometrically determined endpoint that persists for 30 seconds. Add 25 mL (20 g) of sample, taking care to protect the sample and contents of the flask from moisture. Stir vigorously and titrate with Karl Fischer reagent to the same endpoint.

$$\%\ \text{Water}\ (H_2O) = \frac{\text{mL KF reagent} \times \text{KF factor}\ \left(\text{g}\ H_2O/\text{mL}\right) \times 100}{\text{Weight of sample}\ (\text{g})}$$

7.7 *Solubility in Water* — Mix 10 mL of sample with 40 mL of water. Allow to stand 1 hour. The solution should be as clear as an equal volume of water.

7.8 *Chloride* — To 64 mL (50 g) of sample, add 10 mL of sodium carbonate reagent solution and evaporate to dryness on a steam bath in a hood. Dissolve the residue in 10 mL of water, add 1 mL of nitric acid and dilute to 20 mL with water. Add 1 mL of silver nitrate reagent solution. Any turbidity produced should be no greater than that produced when 0.01 mg of chloride ion (Cl) is treated as the sample.

7.9 *Phosphate* — To 26 mL (20 g) of sample, add 10 mL of sodium carbonate reagent solution and evaporate to dryness on a steam bath in a hood. Dissolve the residue 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 produced should be no greater than that produced when 0.01 mg of phosphate ion (PO_4) is treated like the sample.

7.10 *Arsenic and Antimony (as As)* — Evaporate 256 mL (200 g) of sample in a 400 mL beaker to a small volume in a hood. Add 50 mL of water and again evaporate to a small volume. Repeat the evaporation with water addition. Do not allow to go to dryness. Add 5 mL of nitric acid 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 the SEMI C1,

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‘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 should be no greater than that of the standard containing 0.002 mg of arsenic (As).

7.11 *Trace Metal Analysis*

7.11.1 *Boron* — To 128 mL (100 g) of sample, add 0.10 mL of 10% sodium hydroxide, and evaporate to dryness in a nitrogen atmosphere using a covered chamber such as a Thiers assembly or equivalent. Dissolve the residue with 2 mL of water and 0.10 mL of hydrochloric acid. Transfer to a test tube, cool in an ice bath, and add 10 mL of sulfuric acid. Add 10 mL of carminic acid solution (¶ 7.11.1.1) and remove from the ice bath. Allow to stand for 45 minutes. Prepare a standard containing 0.001 mg of boron treated exactly as the sample (¶ 7.11.1.2). Run a complete blank determination on 2 mL of water. Measure the absorbances of the sample and standard against the blank at 585 nm using 5.00 cm cells. The absorbance of the sample solution should be no greater than that of the standard.

7.11.1.1 *Carminic Acid Solution* — Dissolve 0.05 g of carminic acid in 100 mL of sulfuric acid and shake until dissolution is complete.

7.11.1.2 *Boron Standard Solution* — Dissolve 0.572 g of boric acid in water in a 1000 mL volumetric flask. Dilute to the mark with water. Dilute 10.0 mL of this solution with water to the mark in a 100 mL volumetric flask. (1 mL = 0.01 mg boron.)

7.11.2 *Gold (Au)* — Analyze by graphite atomic absorption using the manufacturer’s recommended procedure. This technique has been shown to give satisfactory results using a 1:4 dilution and Zeeman background correction. Each laboratory must determine the appropriate dilution and background correction for their instrument to meet the specification limit.

7.11.3 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), calcium (Ca), chromium (Cr), copper (Cu), 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 they are validated for each trace metal according to SEMI C1, ‘Method Validation’.

7.11.4 *Special Reagents*

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

7.11.4.2 *2% (v/v) Hydrochloric Acid Solution* — Dilute 20 mL of ultra pure 12 M hydrochloric acid to 1 L using water meeting the criteria for Type E1.1 in ASTM D5127.

7.11.5 *Sample Preparation*

7.11.5.1 *Tin* — To 100 g (128 mL) sample in a PTFE dish add 1 mL of 1% oxalic acid solution. Slowly evaporate to about 1 mL. Dissolve the residue in 10% hydrochloric acid and transfer to a 10 mL volumetric flask using 10% hydrochloric acid. Analyze by inductively coupled plasma optical emission spectrometry (ICP-OES) using matrix-matched standards.

7.11.5.2 *All Other Elements* — In a clean environment, place 250 g of sample in a PTFE dish. Slowly evaporate on a hot plate avoiding loss of sample by effervescence or spattering until approximately 1 mL of liquid remains. Take up liquid and all visible residue (from walls of dish) with 1 mL ultra pure, 12 M hydrochloric acid and continue heating until approximately 0.5 mL of liquid remains. No undissolved particulate matter should be observed. Otherwise repeat the addition of hydrochloric acid until all particulate matter is dissolved. Transfer quantitatively to a 50 mL volumetric flask using 2% (v/v) hydrochloric acid and adjust liquid level to mark. Prepare a reagent blank using the same reagents and in the same manner as for the sample concentration.

NOTE 2: Due to the uncertainty of acid concentration in the liquid residue, the final concentration can be estimated to be approximately 2% (v/v). Standard calibration solutions are to use this same acid concentration.

7.11.6 *Analysis*

7.11.6.1 Using the prepared sample and reagent blank, analyze group I elements potassium (K) and sodium (Na) by atomic absorption spectroscopy and all other elements by ICP-OES. If necessary, apply a reagent blank correction to the final determined value of the sample.

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