

SEMI C31-0708

SPECIFICATION FOR METHANOL

This standard was technically approved by the global Liquid Chemicals Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on May 13, 2008. It was available at www.semi.org in June 2008 and on CD-ROM in July 2008. Originally published 1978; previously published March 2001.

1 Purpose

1.1 The purpose of this document is to standardize requirements for methanol 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 all grades of methanol used in the semiconductor industry.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard 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 Standard

SEMI C1 — Guide for the Analysis of Liquid Chemicals

4.2 ASTM Standard¹

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

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

5 Terminology

5.1 None.

6 Physical Property (for information only)

Chemical Formula	CH ₃ OH
CAS Number	67-56-1
Density at 25°C	0.79 g/mL
Boiling Point	64.5°C

7 Requirements

7.1 The requirements for methanol for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

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

8.1 **Assay** — Analyze the sample by gas chromatography (see SEMI C1, § 7.2 Guidelines for Assay by Wide Bore Column Gas Chromatography). The parameters cited have given satisfactory results.

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; <http://www.astm.org>

Column: 30 meter × 530 micron I.D. fused silica capillary, coated with 5 micron film of DB-1 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):	
Methanol	3.0
Ethanol	4.0
Acetone	4.8

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 *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 42 mL (33 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.

8.4 *Alkalinity* — Add 126 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.

8.5 *Residue after Evaporation* — Evaporate 253 mL (200 g) of sample to dryness. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (see SEMI C1, § 7.4, Determination of Residue After Evaporation).

8.6 *Water* — Add 50 mL of sample to a dry titration flask and add Karl Fischer (KF) reagent to a visually or electrometrically determined endpoint that persists for 30 seconds. Add 50 mL (40 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.

$$\% \text{ Water (H}_2\text{O)} = \frac{\text{mL KF reagent} \times \text{KF factor (g H}_2\text{O/mL)} \times 100}{\text{Weight of sample (g)}}$$

8.7 *Solubility in Water* — Mix 15 mL of sample with 45 mL of water. Allow to stand for 30 minutes. The solution should be as clear as an equal volume of water.

8.8 *Boron* — To 127 mL (100 g) of sample, add 0.10 mL of 10% sodium hydroxide solution, 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. Then add 10 mL of carminic acid solution 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. 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.

8.9 *Chloride* — To 63 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.

8.10 *Phosphate* — To 25 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