

SEMI C25-0312

SPECIFICATION FOR DICHLOROMETHANE (METHYLENE CHLORIDE)

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 December 24, 2011. Available at www.semiviews.org and www.semi.org in March 2012; originally published in 1978; previously published March 2006.

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.

NOTICE: This Standard or Safety Guideline has an Inactive Status because the conditions to maintain Current Status have not been met. Inactive Standards or Safety Guidelines are available from SEMI and continue to be valid for use.

1 Purpose

1.1 The purpose of this Specification is to standardize requirements for dichloromethane (methylene chloride) used in the semiconductor industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results. Alternative methods may be used as long as they comply with SEMI C1 guidelines for method validation.

2 Scope

2.1 The scope of this Specification is for a grade of dichloromethane 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

SEMI C27 — Specifications and Guidelines for Hydrochloric Acid

SEMI C35 — Specifications and Guideline for Nitric Acid

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

5 Terminology

5.1 None.

6 Properties

Table 1 Properties of Dichloromethane

Molecular Formula	CH ₂ Cl ₂
Molecular Weight	84.93
CAS Number	75-09-2
Density at 20°C	1.32 g/mL
Boiling Point	39.8°C

7 Requirements

7.1 The requirements for dichloromethane for Grade 1 are listed in Table 2.

8 Grade 1 Procedures

8.1 *Assay* — Analyze the sample by gas chromatography (refer to SEMI C1). The parameters cited have given satisfactory results.

8.1.1 Injector Temperature: 150°C, Detector: Thermal Conductivity, Detector Temperature: 250°C, Sample Size: 0.2 µL, splitless, Carrier Gas: Helium at 3 mL/min, Approximate Retention Times: Dichloromethane –6 min., Chloroform: –11 min., Carbon Tetrachloride –14 min.

- 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.

8.2 *Color* — Dilute 2.0 mL of platinum-cobalt stock solution (APHA No. 500, refer to SEMI C1) 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 (refer to SEMI C1). Add 0.01 N sodium hydroxide until a slight pink color persists after shaking for one-half minute. Add 38 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 shall be required.

8.4 *Residue After Evaporation* — Evaporate 76 mL (100 g) of sample to dryness. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (refer to SEMI C1, “Determination of Residue After Evaporation”). Residue after evaporation shall be no more than 1.0 mg.

8.5 *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 76 mL (100 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.

$$\% \text{ Water (H}_2\text{O)} = \frac{\text{Volume KF reagent (mL)} \times \text{KF factor (g H}_2\text{O/mL)} \times 100}{\text{Mass of sample (g)}} \quad (1)$$

8.6 *Chloride* — To a 100 mL separatory funnel, add 30 mL (40 g) of sample and 40 mL of water. Shake well for 30 seconds and allow the two layers to separate. Discard the sample (lower) layer. To a 20 mL portion of the water layer add 1 mL of nitric acid and 1 mL of silver nitrate reagent solution (refer to SEMI C1). Any turbidity produced shall be no greater than that of a standard containing 0.01 mg of chloride ion (Cl) in an equal volume of solution containing the amounts of reagents used.

8.7 *Phosphate* — To 7.5 mL (10 g) of sample, add 10 mL of sodium carbonate reagent solution (refer to SEMI C1) 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 (refer to SEMI C1) and 1 mL of p-(methylanino)phenol sulfate reagent solution (refer to SEMI C1). Allow to stand at room temperature for 2 hours. Any blue color produced shall be no greater than that produced when 0.01 mg of phosphate ion (PO₄) is treated as the sample.

8.8 *Heavy Metals (as Pb)* — Evaporate 75 mL (100 g) of sample to dryness on a steam bath in a hood. Dissolve the residue in 3 mL of diluted hydrochloric acid (1 + 1) and dilute with water to 15 mL. If necessary, filter through a small filter and wash the evaporating dish and the filter with 10 mL of water. Dilute to 25 mL with water. For the standard, dilute a solution containing 0.02 mg of lead ion (Pb) and 3 mL of diluted hydrochloric acid (1 + 1) to 25 mL with water. Adjust the pH of both solutions to between 3 and 4 with diluted ammonium hydroxide (10% NH₃) or with acetic acid, and dilute with water to 40 mL. To each solution, add 10 mL of freshly prepared hydrogen sulfide water (refer to SEMI C1) and compare. The sample solution shall be no darker than the standard.

8.9 *Arsenic and Antimony (as As)* — Evaporate 152 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