

SEMI C48-0812

SPECIFICATION AND GUIDE FOR 1,1,1-TRICHLOROETHANE*, FURNACE GRADE

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 June 18, 2012. Available at www.semiviews.org and www.semi.org in August 2012; originally published in 1988; previously published July 2006.

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 Document is to standardize requirements for 1,1,1-Trichlorethane, Furnace Grade used in the semiconductor industry and testing procedures to support those standards.

***CAUTION:** This material reacts violently with aluminum and white metals and this material is not be used for vapor degreasing (contains less than 0.05% inhibitors).

2 Scope

2.1 The scope of this Document covers grades of 1,1,1-Trichloroethane 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 — 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.

5 Terminology

5.1 None.

6 Properties

Table 1 Properties for 1,1,1-Trichloroethane*, Furnace Grade

Chemical Formula	C ₂ H ₃ Cl ₃
Molecular Weight	133.4
CAS Number	71-55-6
Density at 25°C	1.33 g/mL
Boiling Point	73°C

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>

7 Requirements and Suggested Values

7.1 Requirements for 1,1,1-Trichloroethane for Grade 1 and suggested values for Tier A are listed in Table 2.

8 Grade 1 Procedures

8.1 *Assay* — Analyze the sample by gas chromatography (see SEMI C1). The parameters below were given satisfactory results.

- 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.

NOTE 1: Calibration standard to be determined between supplier and user.

- Injector Temperature: 150°C; Detector: Thermal Conductivity, Detector Temperature: 250°C, Sample Size: 0.2 µL splitless.
- Approximate Retention Time (min): 1,1,1-Trichloroethane 10.0.

8.2 *Acidity* — To 25 mL of water in a glass-stoppered flask, add 10 mL of sample and 0.1 mL of phenolphthalein indicator solution (see SEMI C1). Add 0.01 N sodium hydroxide until a slight pink color persists after shaking for one-half minute. Add 75 mL (100 g) of the sample, mix well, and titrate with 0.01 N sodium hydroxide until the pink color is reproduced. Not more than 3.0 mL of the sodium hydroxide solution shall be required.

8.3 *Residue after Evaporation* — Evaporate 75 mL (100 g) of sample to dryness. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (see SEMI C1).

8.4 *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 38 mL (50 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.

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

8.5 *Phosphate* — To 75 mL (100 g) of sample, add 10 mL of sodium carbonate reagent solution (see 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 (see SEMI C1) and 1 mL of p-(methylamino)phenol sulfate reagent solution (see SEMI C1). Allow to stand at room temperature for 2 hours. Any blue color produced shall be no greater than that produced when 0.001 mg of phosphate ion (PO₄) is treated like the sample.

8.6 Trace Metal Analysis

8.6.1 *Boron* — To 75 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 (see SEMI C1). Dissolve the residue with 2 mL of water and 0.10 mL of hydrochloric acid (see SEMI C1). Transfer to a test tube, cool in an ice bath, and add 10 mL of sulfuric acid. Add 10 mL of carminic acid solution (§ 8.7.1.1) and remove from the ice bath. Allow to stand for 45 minutes. Prepare a standard containing 0.0001 mg of boron treated exactly as the sample (§ 8.7.1.2). 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 shall be no greater than that of the standard.

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

8.6.1.2 *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 1000 mL volumetric flask (1 mL = 0.001 mg boron).