

SEMI C48-0699^E

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

This specification and this guideline were technically approved by the Global Process Chemicals Committee and are the direct responsibility of the North American Process Chemicals Committee. Current edition approved by the North American Regional Standards Committee on April 23, 1999. Initially available on SEMI OnLine May 1999; to be published June 1999. This document replaces SEMI C1.26 and C7.15 in their entirety. Originally published in 1988 and 1991 respectively.

^E This document was editorially modified in March 2000. Changes were made to the table following Section 8.1.

* CAUTION: Reacts violently with Aluminum and white metals and is not to be used for vapor degreasing. (Contains less than 0.05% inhibitors.)

1 Purpose

1.1 The purpose of this document is to standardize requirements for 1,1,1-trichloroethane, furnace grade used in the semiconductor industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results. This document also provides guidelines for grades of 1,1,1-trichloroethane, furnace grade for which a need has been identified. In the case of the guidelines, the test methods may not have been statistically validated yet.

2 Scope

2.1 The scope of this document is all grades of 1,1,1-trichloroethane, furnace grade used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

SEMI C1 — Specifications for Reagents

5 Terminology

5.1 None.

6 Physical Property (for information only)

Density at 25°C	1.33 g/mL
Boiling Point	73°C

7 Requirements

7.1 The requirements for 1,1,1-trichloroethane, furnace grade for Grade 1 and Tier A are listed in Table 1.

8 Grade 1 Procedures

8.1 *Assay* — Determine the assay using a suitable gas chromatograph. Integrate the areas under all stabilizer and impurity peaks and the 1,1,1-trichloroethane peak. Apply appropriate response factors for the column and detector system to correct the results from area percent to weight percent. The assay shall not be less than 99.9% by weight (see SEMI C1, Section 3.1, Guidelines for Assay by Wide Bore Column Gas Chromatography). The parameters cited have given satisfactory results.

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

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):	
1,1,1-Trichloroethane	10.0

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 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 should be required.

8.4 Residue After Evaporation — Evaporate 75 mL (100 g) of sample to dryness in a clean environment. Dry at 105°C for 30 minutes, cool in desiccator and weigh to a precision of 0.1 milligram (see SEMI C1, Section 3.3, Determination of Residue After Evaporation).

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 38 mL (50 g) of sample, taking care to protect the sample and contents of the flask from moisture. Stir and titrate with Karl Fischer reagent to:

% Water (H₂O)=

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

8.6 Phosphate — To 75 mL (100 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. Add 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow to 880 μm in a 1 cm cell should be no greater than that produced when 0.001 mg of phosphate ion (PO₄) is treated as the sample.

9 Grade 2 Procedures

9.1 This section does not apply to this chemical.

10 Grade 3 Procedures

10.1 This section does not apply to this chemical.

11 Grade 4 Procedures

11.1 This section does not apply to this chemical.

12 Grade 5 Procedures

12.1 This section does not apply to this chemical.

13 VLSI Grade Procedures

13.1 This section does not apply to this chemical.

14 Tier A Procedures

14.1 Standardized test methods are being developed for all parameters at the purity levels indicated. Until standardized test methods are published, test methodology shall be determined by user and producer. The Process Chemicals Committee considers a test method to be valid only if there is a documented recovery study showing a recovery of 75 - 125%. Recovery is for a known sample spike at 50% of the specified level.

15 Tier B Procedures

15.1 This section does not apply to this chemical.

16 Tier C Procedures

16.1 This section does not apply to this chemical.

17 Tier D Procedures

17.1 This section does not apply to this chemical.