

SEMI C26-0912

SPECIFICATION AND GUIDE FOR HEXAMETHYLDISILAZANE (HMDS)

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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 hexamethyldisilazane 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. This Specification also provides a guide for a grade of hexamethyldisilazane for which a need has been identified. In the case of the guide, the test methods may not have been statistically validated yet.

2 Scope

2.1 The scope of this Specification is for grades of hexamethyldisilazane 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

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

5 Terminology

5.1 None.

6 Properties

Table 1 Properties of Hexamethyldisilazane

Molecular Formula	$(\text{CH}_3)_3\text{SiNHSi}(\text{CH}_3)_3$
Molecular Weight	161.39
CAS Number	999-97-3
Boiling Point	126°C

7 Requirements and Suggested Values

7.1 The requirements for hexamethyldisilazane 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 (refer to SEMI C1). The parameters cited have given satisfactory results.

- Injector Temperature: 200°C, Detector: Thermal Conductivity, Detector Temperature: 300°C, Sample size: 0.4 µL, Carrier Gas: Helium at 8 mL/min., Approximate Retention Times: Hexamethyldisilazane – 14.3 min.
- Column: 30 m × 530 micron I.D. fused silica capillary, coated with 5 micron film of DB-1 or equivalent (100% methylsilicone which has been bonded and cross linked).
- Column Temperature: 40°C isothermal for 2 minutes, then programmed to 220°C at 10°C/min., and isothermal for 10 minutes.

8.2 *Trailing Impurities* — Use the chromatogram developed in the assay (§ 8.1). With the relative retention time for hexamethyldisilazane taken as 1.0, establish the integrated area percents for trailing impurities with relative retention times from 1.3 to 4.0. The total of such trailing impurities shall not exceed 0.1%.

8.3 *Color* — Dilute 2.0 mL of platinum-cobalt stock solution (APHA No. 500, see 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 shall be no darker than the standard.

8.4 *Residue After Evaporation* — Evaporate 130 mL (100 g) of sample to dryness in a hood. Dry at 105°C for one hour, 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 *UV Absorbance* — A UV scan is obtained on all HMDS electronic grade material to determine if any UV absorbing impurities are present: specifically toluene.

8.5.1 *Equipment* — Bausch and Lomb Spectronic 2000 Spectrophotometer or other double beam instrument capable of measurements at 270 nm. Cells – 1 cm path length.

8.5.2 *Reagents* — FC-113 and methanol for UV cell cleaning.

8.5.3 *Procedure* — Using water as the reference and sample cells, establish the baseline between 300 and 270 nm. Remove the water sample from the sample path and replace it with the HMDS sample. Read the absorbance between 300 and 270 nm. The absorbance at 270 nm shall read 0.07 to 0.25.

NOTE 1: Oxygen will contribute to the UV absorbance.

NOTE 2: The sample must be at ambient temperature.

NOTE 3: The UV absorbance will increase or decrease with time.

NOTE 4: HMDS is flammable and corrosive.

8.6 *Chloride* — To a 500 mL separatory funnel, add 260 mL (200 g) of sample, 20 mL of water, and 1 mL of sodium hydroxide reagent solution (see SEMI C1). Shake well for 30 seconds and allow the two layers to separate. Carefully draw off the aqueous (lower) layer into a 250 mL beaker. Repeat the extraction five times and combine the six aqueous extracts. Add 2 mL of nitric acid to acidify the solution and arrange the beaker for magnetic stirring.