

SEMI C26-0306

SPECIFICATION AND GUIDELINE FOR HEXAMETHYLDISILAZANE (HMDS)

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 November 29, 2005. It was available at www.semi.org in February 2006 and on CD-ROM in March 2006. Originally published 1983; previously published June 1999.

NOTICE: This document was completely rewritten in 2006.

1 Purpose

1.1 The purpose of this document 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. This document also provides guidelines for grades of hexamethyldisilazane 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 hexamethyldisilazane 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

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 Physical Properties (for information only)

6.1 Density at 25°C 0.77 g/mL

6.2 Boiling Point 126°C

7 Requirements

7.1 The requirements for hexamethyldisilazane for Grade 1 and Tier A are listed in Table 1.

8 Grade 1 Procedures

8.1 *Assay* — Analyze the sample by gas chromatography (see SEMI C1, Guidelines for Assay by Wide Bore Columns Gas Chromatography). 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 8mL/min, Approximate Retention Times: Hexamethyldisilazane –14.3min.

Column: 30 meter × 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 (see ¶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 should not exceed 0.1%.

8.3 *Appearance* — Place 15 mL of the sample in a 20 × 150 mm test tube and compare with water in a similar tube. Viewed across the column by means of transmitted light, the two liquids should be equal in clarity and free from suspended matter.

8.4 *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 should be no darker than the standard.

8.5 *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 (see SEMI C1, Determination of Residue After Evaporation).

8.6 *UV Absorbance* — A UV scan is obtained on all HMDS electronic grade material to determine if any UV absorbing impurities are present: specifically toluene.

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

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

8.6.3 *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 should 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.

8.7 *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. 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.

8.7.1 Using a pH meter fitted with a silver billet electrode (Beckman #89261, Corning #476065, or equivalent) and a silver/silver chloride reference electrode, determine the apparent pH versus mL readings while titrating the continuously stirred sample solution with 0.01 M methanolic silver nitrate solution. Add the silver nitrate solution in 0.1–0.2 mL increments from a 10 mL buret. Run a total blank. Plot the titration data on linear graph paper or use the second-derivative method to establish the endpoint.

ppm Chloride (Cl) =

$$\frac{(\text{mL sample} - \text{mL blank}) \times \text{M of AgNO}_3 \times 35,460}{\text{Weight of sample (g)}}$$

8.7.2 *Standard Silver Nitrate in Methanol Solution* — Dissolve 1.700 grams of silver nitrate in absolute methanol and dilute to 1000 mL. Store this solution in an amber bottle and standardize immediately before use against a standard sodium chloride (NaCl) solution using the above second-derivative method.

8.8 *Arsenic and Antimony (as As)* — 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. Shake well for 30 seconds and allow the two layers to separate. Carefully draw off the aqueous (lower) layer into a 150 mL beaker. Repeat the extraction twice more, combining the three aqueous extracts.

8.8.1 Add 15 mL of nitric acid and 5 mL of sulfuric acid and evaporate to dense fumes of sulfur trioxide. Cool, cautiously add 10 mL of water, and re-evaporate to dense fumes of sulfur trioxide. Cool and cautiously wash into a generator flask with water to make a volume of 35 mL. Proceed as described in the General Method for Arsenic (and Antimony) under SEMI C1, starting with the sentence which begins “Swirl the flask...” Any red color in the silver diethyldithiocarbamate solution of the sample should not exceed that for the standard containing 0.002 mg of arsenic (As).

8.9 *Trace Elements* — By a suitable procedure, determine for each of the specified trace elements that its content is not greater than the stated specification limit (see SEMI C1 for applicable methods e.g. Inductively Coupled Plasma Optical Emission Spectrometry).