

SEMI C37-0699

SPECIFICATION FOR PHOSPHORIC ETCHANTS

This specification was technically approved by the Global Process Chemicals Committee and is 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 C2.3 in its entirety. Originally published in 1981.

1 Purpose

1.1 The purpose of this document is to standardize requirements for phosphoric etchants 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 phosphoric etchants 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 phosphoric etchants used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

SEMI C1 — Specifications for Reagents

SEMI C2 — Specifications for Etchants

5 Terminology

5.1 *phosphoric etchant* — any combination of phosphoric, nitric, and acetic acids with the relative composition expressed in terms of 85% phosphoric acid, 70% nitric acid, and glacial acetic acid, respectively. In the expression, all the relative volumes shall be reduced to a ratio of the smallest whole numbers. For example, a 16:1:2 phosphoric etchant would imply a mixture of 16 volumes of 85% phosphoric acid, 1 volume of 70% nitric acid, and 2 volumes of glacial acetic acid. If a component is absent, its relative volume shall be taken as zero. For example, a 16:1:0 phosphoric etchant implies that no acetic acid is present.

6 Composition

6.1 The content of each of the components of a phosphoric etchant shall be expressed on a weight/weight basis of the 100 percent acid in the total mixture. For the volume-to-weight conversion, the densities shall be taken as 1.695, 1.415, and 1.050 g/mL for 85% phosphoric acid, 70% nitric acid, and glacial acetic acid, respectively.

7 Tolerances

7.1 The tolerances allowed for the absolute percentage of each of the components of a phosphoric etchant shall be:

Phosphoric Acid:	± 1.0% (\pm 0.20 meq/g)
Nitric Acid:	± 0.5% (\pm 0.08 meq/g)
Acetic Acid:	± 0.5% (\pm 0.08 meq/g)

8 Requirements

8.1 The requirements for phosphoric etchants for Grade 1 are listed in Table 1.

9 Grade 1 Procedures

9.1 *Total Acidity* — Weigh accurately 2.5 to 3.0 g of sample in a tared weighing bottle. Transfer with water to a 25 mL beaker and dilute to approximately 100 mL. Add 10 drops of thymolphthalein indicator solution and titrate with standardized 1 N sodium hydroxide solution to the blue endpoint. Calculate the total acidity (A):

$$\text{Total Acidity} \left(\frac{\text{meq}}{\text{g}} \right) = \frac{\text{mL} \times \text{N of NaOH}}{\text{Weight of sample (g)}} = A$$

9.2 *Phosphoric Acid* — Weigh accurately 2.5 to 3.0 g of sample in a tared weighing bottle. Quantitatively transfer the sample with 40 to 60 mL of water into a platinum or polyfluorocarbon dish. Evaporate the solution on a steam bath in a hood (1 to 2 hours). Rinse down the inner sides of the dish with 10 to 15 mL of water and re-evaporate for 1 hour. Cool and dilute with 50 mL of water. Add 10 drops of thymolphthalein indicator solution and titrate with standardized 1 N sodium hydroxide solution to the blue endpoint.

$$\% \text{ Phosphoric Acid} = \frac{\text{mL} \times \text{N of NaOH} \times 4.900}{\text{Weight of sample (g)}}$$

9.3 *Nitric Acid* — Weigh accurately a sample containing 0.9 to 1.1 g of nitric acid in a tared 100 mL volumetric flask. Dilute to volume with water and mix thoroughly. Following the manufacturer's directions, ready a spectrophotometer and set the wavelength to 302 nm. Transfer the sample solution to a 1 cm fused silica cell and measure the absorbance versus water. Read from a previously established calibration curve the grams of nitric acid present in the sample solution (see

SEMI C2, Section 3.2, Determination of Nitric Acid by Ultraviolet Absorption Spectrophotometry).

$$\% \text{ Nitric Acid} = \frac{\text{grams of HNO}_3 \text{ calibration curve} \times 100}{\text{Weight of sample (g)}}$$

9.4 Acetic Acid — The acetic acid content is calculated by deducting the acidity found for each of the other components from the value for the total acidity.

$$\% \text{ Acetic Acid} = \left[A - \frac{\% \text{H}_3\text{PO}_4}{4.900} - \frac{\% \text{HNO}_3}{6.302} \right] \times 6.005$$

9.5 Heavy Metals (as Pb) — Dilute 6 g of sample to 30 mL with water. For the standard, add 0.02 mg of lead ion (Pb) to a 5 mL aliquot of this solution and dilute to 25 mL with water. For the sample, use the remaining 25 mL portion. Adjust the pH to between 3 and 4 (using a pH meter) with 1 N acetic acid or dilute ammonium hydroxide (10% NH₃), dilute to 40 mL with water, and mix. Add 10 mL of freshly prepared hydrogen sulfide water to each and mix. Any color in the solution of the sample should not exceed that in the standard.

9.6 Antimony — Dilute 20 mL (20 g) of stock solution to volume with water in a 100 mL volumetric flask and mix. For the standard, dilute 0.4 mg of antimony ion (Sb) to volume with water in a second 100 mL volumetric flask and mix. Following the manufacturer's directions, ready an atomic absorption spectrometer, use an air-acetylene flame, position the antimony hollow cathode lamp, and set the monochromator to the antimony resonance line of 217.6 nm. Employ deuterium lamp correction. Aspirate water and set to zero absorbance. Then aspirate, in succession, the standard, water, and sample. The absorbance of the sample should not exceed that of the standard. (see SEMI C1, Section 3.6, Guidelines for Determination of Trace Elements by Atomic Absorption Spectrometry.)

9.6.1 Stock Solution for Sections 9.6 through 9.9 — To 200 g of sample in a 400 mL beaker, add 100 mL of water. Heat to boiling on a hot plate in a hood, and boil off approximately 100 mL. Cool slightly, add 100 mL of water, and again boil off about 100 mL. Cool, transfer to a 200 mL volumetric flask, and dilute to volume with water. One mL contains 1 gram of sample.

9.7 Arsenic — To 10 mL (10 g) of stock solution in a 125 mL arsine generator flask, add 30 mL of water 10 mL of ferric ammonium sulfate reagent solution, and then 2% potassium permanganate reagent solution dropwise to a permanent pink color. Add 1.5 g of sodium chloride and mix to dissolve. Heat nearly to boiling, remove from heat, and add 1 mL of stannous chloride reagent solution. Dilute to 60 mL and cool to 25°C ± 3°C. For the standard, add 20 mL of dilute sulfuric acid (1 + 4) to 0.005 mg of arsenic ion (As) in a

second arsine generator flask and treat as above. Assemble the arsenic test apparatus (see SEMI C1, Section 3.4.2). Pack each scrubber tube with two pledgets of lead acetate-impregnated glass wool previously moistened with lead acetate solution, freed from excess solution by squeezing, and dried in a vacuum. Allow a small space between the two pledgets. Place 3.0 mL of silver diethyldithiocarbamate solution in each absorber tube and 10 g of zinc (granular) in each generator flask. Immediately connect the scrubber absorber assemblies to the sample and standard generator flasks. Place the flasks in a water bath maintained at 25°C ± 3°C. Swirl each flask occasionally. After 30 minutes disconnect the tubing from the generator flasks and transfer the silver diethyldithiocarbamate solutions to separate 1 cm photometer cells. Measure both sample and standard absorbances at 540 nm using the silver diethyldithiocarbamate solution as a blank. The absorbance of the sample should not exceed that of the standard.

9.8 Manganese — Add 25 mL (25 g) of stock solution to 100 mL of dilute sulfuric acid (1 + 9). For the standard, add 5 mL (5 g) of stock solution and 0.01 mg of manganese ion (Mn) to 100 mL of dilute sulfuric acid (1 + 9). To each solution, add 20 mL of nitric acid, heat to boiling, and continue boiling gently for 5 minutes. Cool slightly, add 0.25 g of potassium periodate, and again boil for 5 minutes. Any pink color in the sample solution should not exceed that in the standard.

9.9 Copper, Iron, and Nickel — Dilute 1 mL (1 g) of stock solution with 50 mL of water in a 100 mL beaker. Add 0.5 mL of ammonium hydroxide and cool. For the three-element standard, add 0.002 mg of copper ion (Cu), 0.01 mg of iron ion (Fe), and 0.003 mg of nickel ion (Ni) to 50 mL of water in a second 100 mL beaker.

9.9.1 Treat each solution as follows: Adjust the pH to 2.5 (using a pH meter) with dilute ammonium hydroxide (10% NH₃) or 10% hydrochloric acid. Transfer to a polyfluorocarbon-stoppered 250 mL separatory funnel. Rinse the beaker with water adjusted to pH 2.5 and add the rinsings to the funnel (the final volume should not exceed 100 mL). Add 10 mL of freshly prepared 1% ammonium 1-pyrrolidinecarbodithiolate solution and mix thoroughly. Add 10.0 mL of water-saturated methyl isobutyl ketone (that is, 4-methyl-2-pentanone), stopper, and shake vigorously for 3 minutes. Allow the layers to separate. Drain and discard the lower, aqueous layer; save the upper, organic layer.

9.9.2 Following the manufacturer's directions, ready the atomic absorption spectrometer for measurements in the absorbance mode and with the air-acetylene flame adjusted for aspiration of an organic solvent. For copper, align the copper hollow cathode lamp, set the wavelength to 324.7 nm, and zero the absorbance while

aspirating methyl isobutyl ketone. Then aspirate the extract of the standard solution, solvent, and the extract of the sample solution. Record the absorbances. Proceed similarly for iron and nickel with measurements at 248.3 and 232.0 nm, respectively. For each of the three elements the absorbance of the sample extract should not exceed that of the standard extract (see SEMI C1, Section 3.6, Guidelines for Determination of Trace Elements by Atomic Absorption Spectrometry).

9.10 Sodium, Lithium, Potassium, Calcium, Strontium, and Magnesium

9.10.1 Sample Solutions for the Determination of Sodium, Lithium, Potassium, Calcium, Strontium, and Magnesium by Atomic Absorption Spectrometry — Thoroughly clean four 100 mL volumetric flasks and label them 1, 2, 3, 4, respectively. Transfer 20 mL (20 g) samples of stock solution into the four volumetric flasks. Add the quantities of cations as listed as follows:

CATION	FLASK	FLASK	FLASK	FLASK
	1	2	3	4
Milligrams of Cation				
Sodium Ion (Na)	0.00	0.15	0.30	0.60
Lithium Ion (Li)	0.00	0.01	0.02	0.04
Potassium Ion (K)	0.00	0.10	0.20	0.40
Calcium Ion (Ca)	0.00	0.20	0.40	0.80
Strontium Ion (Sr)	0.00	0.05	0.10	0.20
Magnesium Ion (Mg)	0.00	0.05	0.10	0.20

9.10.2 General Instructions — Dilute each solution to volume with water and mix thoroughly. Following the manufacturer's directions, ready the atomic absorption spectrometer for measurements in the absorbance mode (see SEMI C1, Section 3.6, Guidelines for Determination of Trace Elements by Atomic Absorption Spectrometry). Determine each of the elements, using the conditions specified in Sections 9.10.3 through 9.10.8.

9.10.2.1 For each element plot on linear coordinate paper the absorbance versus the added quantity in milligrams of the cation added. Draw a straight line through the four points and extrapolate to zero absorbance. This intercept corresponds to the absolute amount, in milligrams, of the element being determined in Solution 1.

$$\text{ppm element} = \frac{\text{mg found} \times 1000}{\text{Weight of Sample (g)}}$$

9.10.3 Sodium — Using an air-acetylene flame and a sodium hollow cathode lamp, set the wavelength to

589.0 nm and zero the absorbance with water. Aspirate, and record the absorbances of each of the solutions, zeroing the absorbance with water between aspiration of the solutions. Calculate the amount of the sodium ion (Na) in Solution 1; this should not exceed 0.30 mg.

9.10.4 Potassium — Using an air-acetylene flame and a potassium hollow cathode lamp, set the wavelength to 766.5 nm and zero the absorbance using water. Aspirate, and record the absorbances of each of the four solutions, zeroing the absorbance with water between aspiration of the solutions. Calculate the amount of potassium ion (K) in Solution 1; this should not exceed 0.20 mg.

9.10.5 Lithium — Using an air-acetylene flame and a lithium hollow cathod lamp, set the wavelength to 670.8 and zero the absorbance with water. Aspirate and record the absorbances of each of the four solutions, zeroing the absorbance with water between aspiration of the solutions. Calculate the amount of lithium ion (Li) in sample Solution 1; this should not exceed 0.02 mg.

9.10.6 Strontium — Using a nitrous oxide-acetylene flame and a strontium cathode lamp, set the wavelength to 460.7 nm and zero the absorbance with water. Aspirate, and record the absorbances of each of the four solutions, zeroing the absorbance with water between aspiration of the solutions. Calculate the amount of strontium ion (Sr) in Solution 1; this should not exceed 0.10 mg.

9.10.7 Calcium — Using a nitrous oxide-acetylene flame and a calcium hollow lamp, set the wavelength to 422.7 nm and zero the absorbance with water. Aspirate, and record the absorbances of each of the four solutions, zeroing the absorbance with water between aspiration of the solutions. Calculate the amount of calcium ion (Ca) in Solution 1; this should not exceed 0.40 mg.

9.10.8 Magnesium — Thoroughly clean four 50 mL volumetric flasks and label them 1a, 2a, 3a, and 4a. Transfer 20 mL (4 g) from each of the four 100 mL volumetric flasks into the corresponding 50 mL flasks. Dilute each to volume and mix thoroughly. Using an air-acetylene flame, background corrections, and a magnesium hollow lamp, set the wavelength to 285.2 nm and zero the absorbance with water. Aspirate, and record the absorbances of each of the four solutions, zeroing the absorbance with water between aspiration of the solutions. Calculate the amount of magnesium ion (Mg) in Solution 1a; this should not exceed 0.02 mg.

10 Grade 2 Procedures

10.1 This section does not apply to this chemical.

11 Grade 3 Procedures

11.1 This section does not apply to this chemical.

12 Grade 4 Procedures

12.1 This section does not apply to this chemical.

13 Grade 5 Procedures

13.1 This section does not apply to this chemical.

14 VLSI Grade Procedures

14.1 This section does not apply to this chemical.

15 Tier A Procedures

15.1 This section does not apply to this chemical.

16 Tier B Procedures

16.1 This section does not apply to this chemical.

17 Tier C Procedures

17.1 This section does not apply to this chemical.

18 Tier D Procedures

18.1 This section does not apply to this chemical.

Table 1 Impurity Limits and Other Requirements for Phosphoric Etchants

Previous SEMI Reference #	C2.3-95
	Grade 1 (Specification)
Heavy Metals (as Pb)	5 ppm max
Antimony (Sb)	15 ppm max
Arsenic (As)	0.2 ppm max
Calcium (Ca)	15 ppm max
Copper (Cu)	1.2 ppm max
Iron (Fe)	6 ppm max
Lithium (Li)	0.3 ppm max
Magnesium (Mg)	2 ppm max
Manganese (Mn)	0.2 ppm max
Nickel (Ni)	1 ppm max
Potassium (K)	6 ppm max
Sodium (Na)	10 ppm max
Strontium (Sr)	2 ppm max
Particles in bottles: size, #/mL	≥1.0 µm, 25 max

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SEMI C38-0699

GUIDELINE FOR PHOSPHORUS OXYCHLORIDE

This guideline was technically approved by the Global Process Chemicals Committee and is 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 C7.12 in its entirety. Originally published in 1991.

1 Purpose

1.1 The purpose of this document is to standardize requirements for phosphorus oxychloride 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 phosphorus oxychloride 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 phosphorus oxychloride used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

4.1 None.

5 Terminology

5.1 None.

6 Physical Property (for information only)

6.1 Not applicable.

7 Requirements

7.1 The requirements for phosphorus oxychloride for Tier A are listed in Table 1.

8 Grade 1 Procedures

8.1 This section does not apply to this chemical.

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.

Table 1 Impurity Limits and Other Requirements for Phosphorus Oxychloride

Previous SEMI Reference #	C7.12-91
	Tier A
	(Guideline)
Assay	99.9% min
Color (APHA)	5 max
Hydroxyl	0.1 absorbance units (see NOTE 1)
Aluminum (Al)	10 ppb max
Arsenic (As)	7 ppb max
Barium (Ba)	1 ppb max
Bismuth (Bi)	1 ppb max
Calcium (Ca)	6 ppb max
Chromium (Cr)	1 ppb max
Cobalt (Co)	1 ppb max
Copper (Cu)	3 ppb max
Gallium (Ga)	1 ppb max
Gold (Au)	1 ppb max
Iron (Fe)	5 ppb max
Lead (Pb)	3 ppb max
Lithium (Li)	6 ppb max
Magnesium (Mg)	5 ppb max
Manganese (Mn)	1 ppb max
Mercury (Hg)	8 ppb max
Nickel (Ni)	1 ppb max
Niobium (Nb)	1 ppb max
Potassium (K)	3 ppb max
Silver (Ag)	1 ppb max
Sodium (Na)	5 ppb max
Strontium (Sr)	1 ppb max
Tin (Sn)	1 ppb max
Titanium (Ti)	3 ppb max
Zinc (Zn)	10 ppb max
Particles in bottles: size, #/mL	≥0.3 µm, 50 max

NOTE 1: Absorbance for the 4000 to 2700 cm⁻¹ for 5 mm optical path length.



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SEMI C39-0699

SPECIFICATION FOR POTASSIUM HYDROXIDE PELLETS

This specification was technically approved by the Global Process Chemicals Committee and is 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 C1.14 in its entirety. Originally published in 1978.

1 Purpose

1.1 The purpose of this document is to standardize requirements for potassium hydroxide pellets 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 potassium hydroxide pellets 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 potassium hydroxide pellets 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)

6.1 Not applicable.

7 Requirements

7.1 The requirements for potassium hydroxide pellets for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

8.1 *Assay* — Weigh accurately 35 to 40 g of sample in a closed weighing bottle. Transfer to a 500 mL volumetric flask, dissolve in carbon dioxide-free water, cool to 25°C, dilute to the mark with carbon dioxide free water, and mix thoroughly. Transfer 25.0 mL of this solution to a 500 mL glass-stoppered flask, dilute with 175 mL of carbon dioxide-free water, add 5 mL of barium chloride reagent solution, shake, and allow to stand for five minutes. Add 0.15 mL of phenolphthalein indicator solution and titrate with 1 N hydrochloric

acid. (Save this solution for the potassium carbonate test.)

$$\% \text{ Assay} = \frac{\text{mL} \times \text{N of HCl} \times 112.2}{\text{Weight of sample (g)}}$$

8.2 *Potassium Carbonate (K_2CO_3)* — To the above titrated solution, add 0.15 mL of methyl orange indicator solution and continue the titration with 1 N hydrochloric acid. The additional hydrochloric acid is equivalent to that consumed by the potassium carbonate.

$$\% K_2CO_3 = \frac{\text{mL} \times \text{N} \times \text{HCl} \times 138.2}{\text{Weight of sample (g)}}$$

8.3 *Ammonium Hydroxide Precipitate* — Weigh about 10 g of sample and dissolve in about 100 mL of water.

8.3.1 Cautiously add 12 mL of sulfuric acid to 15 mL of water, cool, then cautiously add the mixture to the solution of the sample and evaporate to dense fumes. Cool, dissolve the residue in 130 mL of hot water, and add ammonium hydroxide until the solution is just basic to methyl red.

8.3.2 Heat to boiling, filter, wash with hot water, and ignite. The weight of the residue should not exceed 2 mg.

8.4 *Insoluble Matter* — Dissolve 50 g of sample in 250 mL of carbon dioxide-free water in a 500 mL stoppered flask and cool to 25°C. The solution should be clear and colorless. Filter through a tared, medium-porosity filtering crucible and wash thoroughly with hot water. Dry the crucible at 105°C for 1 hour, cool, and weigh the residue. The weight of the residue should not exceed 2.5 mg.

8.5 *Stock Solution for 8.6-8.11, 8.13-8.14* — Dissolve 50 g of sample in 250 mL of carbon dioxide-free water and dilute with such water to 500 mL. One mL contains 0.10 g of sample. Store in a plastic container.

8.6 *Nitrogen Compounds* — Dilute 20 mL of the Stock Solution with 50 mL of water in a flask connected through a spray trap to a condenser, the end of which dips beneath the surface of 10 mL of 0.1 N hydrochloric acid. For the standard, take 50 mL of water in a similar flask, and add 10 mL of the Stock Solution and 1 mL of the nitrogen standard solution. To each flask add 0.5 g of aluminum wire in small pieces,

allow to stand for 1 hour, and slowly distill about 35 mL. To each distillate add 2 mL of freshly boiled 10% sodium hydroxide reagent solution, dilute with water to 50 mL, and add 2 mL of Nessler reagent. Any color in the solution of the sample should not exceed that in the standard.

8.7 Chloride — Dilute 10 mL of the Stock Solution with water to 50 mL. Filter if necessary through a chloride-free filter. To 10 mL of this solution, add 5 mL of water, 2 mL of nitric acid, and 1 mL of silver nitrate reagent solution. Any turbidity developed should not exceed that produced by 0.02 mg of chloride ion (Cl⁻) in an equal volume of solution containing the quantities of reagent used in the test.

8.8 Phosphate — To 40 mL of the Stock Solution, add 10 mL of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow to stand for 2 hours at room temperature. Any blue color developed should not exceed that produced when 0.02 mg of phosphate ion (PO₄³⁻) is treated as the sample.

8.9 Sulfate — To 20 mL of the Stock Solution, add 5 mL of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 20 mL of water and evaporate again to dryness. Dissolve the residue in 20 mL of water, filter if necessary, and add 1 mL of dilute hydrochloric acid (1 + 19) and 1 mL of barium chloride reagent solution. Allow to stand for 10 minutes. Any turbidity developed should not exceed that produced when 0.04 mg of sulfate ion (SO₄²⁻) is treated as the sample.

8.10 Heavy Metals (as Ag) — To 60 mL of the Stock Solution cautiously add 15 mL of nitric acid. For the standard add 0.05 mg of silver ion (Ag) to 10 mL of the Stock Solution and cautiously add 15 mL of nitric acid. Evaporate both solutions to dryness over a low flame or on an electric hot plate. Dissolve each residue in about 20 mL of water, filter if necessary through a chloride-free filter, and dilute with water to 25 mL. Adjust the pH of the standard and sample solutions to between 3 and 4 (using a pH meter) with 1 N acetic acid or ammonium hydroxide (10% NH₃). Dilute each solution with water to 40 mL and stir. Add 10 mL of freshly prepared hydrogen sulfide water to each solution and stir again. Any color developed in the sample should not exceed that in the standard.

8.11 Iron — Neutralize 10 mL of the Stock Solution with hydrochloric acid, using phenolphthalein indicator solution, add 2 mL in excess, and dilute with water to 50 mL. Add 30 to 50 mg of ammonium peroxydisulfate crystals and 3 mL of ammonium thiocyanate reagent

solution. Any red color developed should not exceed that produced when 0.01 mg of iron ion (Fe) is treated as the sample.

8.12 Mercury — To each of two thoroughly clean 125 mL conical flasks add 20 mL of water and 1 mL of 4% potassium permanganate solution. To one add 5.5 g of solid sample. To the other add 0.5 g of sample and 0.5 mg of mercury ion (Hg). To each flask slowly add 18 mL of hydrochloric acid, with constant swirling. Heat to boiling, allow to cool, and dilute to 100 mL. Determine mercury in 10 mL aliquots by the cold vapor (flameless) atomic absorption method using 1 mL of 10% hydroxylamine hydrochloride reagent solution and 2 mL of 10% stannous chloride reagent solution for the reduction. The peak obtained with the sample solution should not be larger than that from the standard mercury solution.

8.13 Nickel — Dilute 20 mL of the Stock Solution to 50 mL with water and neutralize with hydrochloric acid. Dilute to 85 mL and add ammonium hydroxide to make the solution barely basic (pH 8). Add 5 mL of bromine water and 5 mL of alcoholic 1% dimethylglyoxime solution. Any red color developed should not exceed that produced when 0.02 mg of nickel ion (Ni) is treated as the sample.

8.14 Sodium — Dilute 10 mL of the Stock Solution with water to 100 mL to form the Sample Solution. To prepare the Standard Solution, add 0.5 mg of sodium ion (Na) to 10 mL of the Stock Solution and dilute with water to 100 mL.

8.14.1 Using flame emission spectroscopy, observe the emission of the Standard Solution at the 589 nm sodium line. Observe the emission of the Sample Solution at the 589 nm sodium line and also at a wavelength of 580 nm. The difference between the intensities observed for the Sample Solution at 580 nm and 589 nm should not exceed the difference observed at 589 nm between the Sample Solution and the Standard Solution (see SEMI C1, Section 3.7, Flame Emission Spectroscopy).

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 This section does not apply to this chemical.

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.

Table 1 Impurity Limits and Other Requirements for Potassium Hydroxide Pellets

Previous SEMI Reference #	C1.14-95
	Grade 1
	(Specification)
Assay (KOH)	85.0% min
Potassium Carbonate (K_2CO_3)	1.0% max
Ammonium Hydroxide Precipitate	0.02% max
Insoluble Matter	50 ppm max
Nitrogen Compounds (as N)	10 ppm max
Chloride (Cl)	0.01% max
Phosphate (PO_4)	5 ppm max
Sulfate (SO_4)	20 ppm max
Heavy Metals (as Ag)	10 ppm max
Iron (Fe)	10 ppm max
Mercury (Hg)	0.1 ppm max
Nickel (Ni)	10 ppm max
Sodium (Na)	0.05% max

NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

The user's attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any

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SEMI C40-0699

SPECIFICATION FOR POTASSIUM HYDROXIDE, 45% SOLUTION

This specification was technically approved by the Global Process Chemicals Committee and is 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.

1 Purpose

1.1 The purpose of this document is to standardize requirements for 45% potassium hydroxide solution 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 potassium hydroxide, 45% solution 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 45% potassium hydroxide solution 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)

6.1 Not applicable.

7 Requirements

7.1 The requirements for 45% potassium hydroxide solution for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

NOTE 1: Each laboratory is responsible for verifying the validity of the method within its own operation.

8.1 *Assay* — Accurately weigh 70 to 80 g of sample in a closed weighing bottle. Transfer to a 500 mL volumetric flask, add in carbon dioxide-free water, cool to 25°C, dilute to the mark with carbon dioxide-free water, and mix thoroughly. Transfer 25.0 mL of this solution to a 500 mL glass-stoppered flask, dilute with 175 mL of carbon dioxide-free water, add 5 mL of

barium chloride reagent solution, shake and allow to stand for five minutes. Add 0.15 mL of phenolphthalein indicator solution and titrate with standardized 1 N hydrochloric acid. (Save this solution for the potassium carbonate test.)

$$\% \text{ Assay} = \frac{\text{mL} \times \text{N of HCl} \times 112.2}{\text{Weight of Sample (g)}}$$

8.2 *Potassium Carbonate (K_2CO_3)* — To the above titrated solution, add 0.15 mL of methyl orange indicator and continue the titration with 1 N hydrochloric acid, making sure that no insoluble carbonate remains. The additional hydrochloric acid is equivalent to that consumed by the potassium carbonate.

$$\% K_2CO_3 = \frac{\text{mL} \times \text{N of HCl} \times 138.2}{\text{Weight of Sample (g)}}$$

8.3 *Ammonium Hydroxide Precipitate* — Weigh 40 g of sample into a 250 mL beaker and dissolve in about 100 mL of water.

8.3.1 Cautiously add 26 mL of sulfuric acid to 30 mL of water in a 150 mL glass beaker, cool; then cautiously add the mixture to the solution of the sample, and evaporate to dense fumes. Cool, dissolve the residue in 130 mL of hot water, and add ammonium hydroxide until the solution is just basic to methyl red.

8.3.2 Heat to boiling, filter, wash with hot water and ignite. The weight of the residue should not exceed 2 mg.

8.4 *Insoluble Matter* — Dilute 100 g of sample in 250 mL of carbon dioxide-free water in a 500 mL stoppered flask and cool to 25°C. The solution should be clear and colorless. Filter through a tared, medium porosity filtering crucible and wash thoroughly with hot water. Dry the crucible at 105°C for 1 hour, cool and weigh the residue. The weight of the residue should not exceed 5.0 mg.

8.5 *Stock Solution for Sections 8.6 through 8.13* — Add 100 g of sample to 250 mL of carbon dioxide-free water and dilute with such water to 500 mL. One mL contains 0.20 g of sample. Store in a plastic container.

8.6 *Nitrogen Compounds (as N)* — Dilute 20 mL of the stock solution with 50 mL of water in a flask

connected through a spray trap to a condenser, the end of which dips beneath the surface of 10 mL of 0.1 N hydrochloric acid. For the standard, take 50 mL of water in a similar flask, and add 10 mL of the stock solution and 1 mL of the nitrogen standard solution (0.01mg N/mL). To each flask add 0.5 g of aluminum wire in small pieces, allow to stand for 1 hour, and slowly distill to about 35 mL. To each distillate add 2 mL of freshly boiled 10% sodium hydroxide reagent solution, dilute with water to 50, add 2 mL of Nessler reagent, and mix. Any color in the solution of the sample should not exceed that in the standard.

8.7 Chloride — Dilute 10 mL of the stock solution with water to 50 mL. Filter, if necessary, through a chloride-free filter. To 10 mL of this solution, add 5 mL of water, 2 mL of nitric acid, and 1 mL of silver nitrate reagent solution. Any turbidity developed should not exceed that produced by 0.02 mg of chloride (Cl) ion in an equal volume of solution containing the quantities of reagent used in the test.

8.8 Phosphate — To 50 mL of the stock solution, add 15 mL of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow the solution to stand for 2 hours at room temperature. Any blue color developed should not exceed that produced when 0.02 mg of phosphate (PO_4) ion is treated as the sample.

8.9 Sulfate — To 20 mL of the stock solution, add 5 mL of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 20 mL of water and evaporate again to dryness. Dissolve the residue in 20 mL of water, filter if necessary, and add 1 mL of dilute hydrochloric acid (1+19) and 1 mL of barium chloride reagent solution. Allow to stand for 10 minutes. Any turbidity developed should not exceed that produced when 0.04 mg of sulfate (SO_4) ion is treated as the sample.

8.10 Heavy Metals (as Ag) — To 60 mL of the stock solution cautiously add 15 mL of nitric acid. For the standard add 0.05 mg of silver ion (Ag) to 10 mL of the stock solution and cautiously add 15 mL of nitric acid. Evaporate both solutions to dryness over a low flame or on an electric hot plate. Dissolve each residue in about 20 mL of water, filter if necessary through a chloride-free filter, and dilute with water to 25 mL. Adjust the pH of the standard and sample solutions to between 3 and 4 (using a pH meter) with 1 N acetic acid or ammonium hydroxide (10% NH_3). Dilute each solution with water to 40 mL and stir. Add 10 mL of freshly prepared hydrogen sulfide water to each solution and stir again. Any color developed in the sample should not exceed that in the standard.

8.11 Iron — Neutralize 10 mL of the stock solution with hydrochloric acid, using a phenolphthalein indicator solution, add 2 mL of HCl in excess, and dilute with water to 50 mL. Add 30 to 50 mg of ammonium peroxydisulfate crystals and 3 mL of ammonium thiocyanate reagent solution. Any red color developed should not exceed that produced when 0.01 mg of iron (Fe) ion is treated as the sample.

8.12 Nickel — Dilute 20 mL of the stock solution to 50 mL with water and neutralize with hydrochloric acid using a pH meter. Dilute to 85 mL with water and add ammonium hydroxide to make the solution barely basic (pH 8). Add 5 mL of bromine water and 5 mL of alcoholic 1% dimethylglyoxime solution. Any red color developed should not exceed that produced when 0.02 mg of nickel (Ni) ion is treated as the sample.

8.13 Sodium — Dilute 10 mL of the stock solution with water to 100 mL to form the sample solution. To prepare the standard solution, add 0.6 mg of sodium (Na) ion to 10 mL of the stock solution and dilute with water to 100 mL.

8.13.1 Using flame emission spectroscopy, observe the emission of the standard solution at the 589 nm sodium line. Observe the emission of the sample solution at the 589 nm sodium line and also at a wavelength of 580 nm. The difference between the intensities observed for the sample solution at 580 nm and 589 nm should not exceed the difference observed at 589 nm between the sample solution and standard solution (see SEMI Cl, Section 3.7, Flame Emission Spectroscopy).

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 This section does not apply to this chemical.

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.

Table 1 Impurity Limits and Other Requirements for Potassium Hydroxide, 45% Solution

Previous SEMI Reference #	--
	Grade 1
	(Specification)
Assay (KOH)	45.0 - 47.0%
Insoluble Matter	50 ppm max
Nitrogen Compounds (as N)	5 ppm max
Ammonium Hydroxide Precipitate	50 ppm max
Chloride (Cl)	50 ppm max
Phosphate (PO ₄)	2 ppm max
Potassium Carbonate (K ₂ CO ₃)	0.5% max
Sulfate (SO ₄)	10 ppm max
Heavy Metals (as Ag)	5 ppm max
Iron (Fe)	5 ppm max
Nickel (Ni)	5 ppm max
Sodium (Na)	0.03% max

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SEMI C41-0705

SPECIFICATIONS AND GUIDELINES FOR 2-PROPANOL

This specification was technically approved by the global Liquid Chemicals Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on April 7, 2005. It was available at www.semi.org in June 2005 and on CD-ROM in July 2005. Originally published in 1978; previously published in 2003.

NOTICE: This document was completely rewritten in 2005.

NOTICE: Paragraphs entitled NOTE are not an official part of this document and are not intended to modify or supercede it.

1 Purpose

1.1 The purpose of this document is to standardize requirements for 2-propanol used in the semiconductor industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results.

2 Scope

2.1 The scope of this document covers all grades of 2-propanol used in the semiconductor industry.

2.2 The VLSI grade purity level is typically required by semiconductor devices with geometries of 0.8–1.2 microns.

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 Referenced Standards and Documents

3.1 SEMI Standards

SEMI C1 — Guide for the Analysis of Liquid Chemicals

3.2 ASTM Standards¹

ASTM D5127 — Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry

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

4 Terminology

None.

5 Physical Property (for information only)

Density at 25°C	0.78 g/mL
Boiling Point	82.3°C

6 Requirements

6.1 The requirements for 2-propanol for Grades 1, 2, 3, 4 & VLSI Guidelines, are listed in Table 1.

7 Grade 1 Procedures

NOTE 1: Each laboratory is responsible for verifying the validity of the method within its own operation.

7.1 *Assay* — Analyze the sample by gas chromatography (see SEMI C1, ‘Assay by Wide Bore Column Gas Chromatography’). The parameters cited have given satisfactory results.

¹ American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555, www.astm.org



Column: 30 meter \times 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.

Injector Temperature:	150°C
Detector Temperature:	250°C
Sample Size:	0.2 μ L splitless
Carrier Gas:	Helium at 3 mL/min
Detector:	Thermal Conductivity
Approximate Retention Times (min):	
Acetone	4.5
2-Propanol	5.0

7.2 *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.

7.3 *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 0.01N sodium hydroxide until a slight pink color persists after shaking for one-half minute. Add 64 mL (50 g) of the sample, mix well, and titrate with 0.01N sodium hydroxide until the pink color is reproduced. Not more than 1.0 mL of the sodium hydroxide solution should be required.

7.4 *Alkalinity* — Add 128 mL (100 g) of sample to 25 mL of water and mix well. Add 0.05 mL of methyl red indicator solution. Titrate with 0.01 N hydrochloric acid until a slight pink color is produced. Not more than 1.0 mL of the hydrochloric acid should be required.

7.5 *Residue after Evaporation* — Evaporate 256 mL (200 g) of sample to dryness. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (see SEMI C1, ‘Residue after Evaporation’).

7.6 *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 25 mL (20 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.

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

7.7 *Solubility in Water* — Mix 10 mL of sample with 40 mL of water. Allow to stand 1 hour. The solution should be as clear as an equal volume of water.

7.8 *Chloride* — To 64 mL (50 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 10 mL of water, add 1 mL of nitric acid and dilute to 20 mL with water. Add 1 mL of silver nitrate reagent solution. Any turbidity produced should be no greater than that produced when 0.01 mg of chloride ion (Cl) is treated as the sample.

7.9 *Phosphate* — To 26 mL (20 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 and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow to stand at room temperature for 2 hours. Any blue color produced should be no greater than that produced when 0.01 mg of phosphate ion (PO_4^{3-}) is treated like the sample.

7.10 *Arsenic and Antimony (as As)* — Evaporate 256 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 trioxide. Cool, cautiously add 10 mL of water, and again 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 SEMI C1, ‘Trace Arsenic (and Antimony) Determination’, starting with the sentence that begins, “Swirl the



flask...." Any red color in the silver diethyldithiocarbamate solution of the sample should be no greater than that of the standard containing 0.002 mg of arsenic (As).

7.11 Trace Metal Analysis

7.11.1 *Boron* — To 128 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. Dissolve the residue with 2 mL of water and 0.10 mL of hydrochloric acid. Transfer to a test tube, cool in an ice bath, and add 10 mL of sulfuric acid. Add 10 mL of carminic acid solution (¶7.11.1.1) and remove from the ice bath. Allow to stand for 45 minutes. Prepare a standard containing 0.001 mg of boron treated exactly as the sample (¶7.11.1.2). 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 should be no greater than that of the standard.

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

7.11.1.2 *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 100 mL volumetric flask. (1 mL = 0.01 mg boron.)

7.11.2 *Gold (Au)* — Analyze by graphite atomic absorption using the manufacturer's recommended procedure. This technique has been shown to give satisfactory results using a 1:4 dilution and Zeeman background correction. Each laboratory must determine the appropriate dilution and background correction for their instrument to meet the specification limit.

7.11.3 The following method has given satisfactory results in determining trace metal impurities at the value specified for each of the following trace metals: aluminum (Al), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), and zinc (Zn). Alternate methods may be used as long as they are validated for each trace metal according to SEMI C1, 'Method Validation'.

7.11.4 Special Reagents

7.11.4.1 *Hydrochloric Acid, Ultra Pure* — Use hydrochloric acid specified for ultra low metal ion content.

7.11.4.2 *2% (v/v) Hydrochloric Acid Solution* — Dilute 20 mL of ultra pure 12 M hydrochloric acid to 1 L using water meeting the criteria for Type E1.1 in ASTM D5127.

7.11.5 Sample Preparation

7.11.5.1 *Tin* — To 100 g (128 mL) sample in a PTFE dish add 1 mL of 1% oxalic acid solution. Slowly evaporate to about 1 mL. Dissolve the residue in 10% hydrochloric acid and transfer to a 10 mL volumetric flask using 10% hydrochloric acid. Analyze by inductively coupled plasma optical emission spectrometry (ICP-OES) using matrix-matched standards.

7.11.5.2 *All Other Elements* — In a clean environment, place 250 g of sample in a PTFE dish. Slowly evaporate on a hot plate avoiding loss of sample by effervescence or spattering until approximately 1 mL of liquid remains. Take up liquid and all visible residue (from walls of dish) with 1 mL ultra pure, 12 M hydrochloric acid and continue heating until approximately 0.5 mL of liquid remains. No undissolved particulate matter should be observed. Otherwise repeat the addition of hydrochloric acid until all particulate matter is dissolved. Transfer quantitatively to a 50 mL volumetric flask using 2% (v/v) hydrochloric acid and adjust liquid level to mark. Prepare a reagent blank using the same reagents and in the same manner as for the sample concentration.

NOTE 2: Due to the uncertainty of acid concentration in the liquid residue, the final concentration can be estimated to be approximately 2% (v/v). Standard calibration solutions are to use this same acid concentration.

7.11.6 Analysis

7.11.6.1 Using the prepared sample and reagent blank, analyze group I elements potassium (K) and sodium (Na) by atomic absorption spectroscopy and all other elements by ICP-OES. If necessary, apply a reagent blank correction to the final determined value of the sample.



8 Grade 2 Procedures

NOTE 3: Each laboratory is responsible for verifying the validity of the method within its own operation.

8.1 *Non-Metal Impurities* — See §7, which contains procedures for the following tests:

- Assay
- Water
- Color (APHA)
- Chloride
- Phosphate
- Arsenic and Antimony (as As)
- Boron

8.2 *Trace Metals Analysis*

8.2.1 The following method has given satisfactory results in determining trace metal impurities at the values specified for each of the following metals: aluminum (Al), barium (Ba), beryllium (Be), bismuth (Bi), cadmium (Cd), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), gallium (Ga), germanium (Ge), gold (Au), iron (Fe), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), niobium (Nb), potassium (K), silver (Ag), sodium (Na), strontium (Sr), tantalum (Ta), thallium (Tl), tin (Sn), titanium (Ti), vanadium (V), zinc (Zn), and zirconium (Zr). Alternate methods may be used as long as they are validated for each trace metal according to SEMI C1, ‘Method Validation’.

8.2.2 *Special Reagents*

8.2.2.1 *Nitric Acid, Ultra Pure* — Use nitric acid specified for low metal ion content.

8.2.2.2 *1% Nitric Acid Solution* — Dilute 10 mL of ultrapure nitric acid to 1 L using water meeting the criteria for Type E1.1 in ASTM D5127.

8.2.2.3 *Water* — The water used for all the dilution, calibration and standards should meet at a minimum the criteria for Type E1.1 in ASTM D5127 in regard to trace metal analysis.

8.2.2.4 *Indium Internal Standard* — Make up an indium internal standard solution to a concentration of 20 mg/mL (ppm) from an appropriate concentrated indium standard solution.

8.2.2.5 *Mannitol Powder* — Mannitol powder, reagent grade (ACS)

8.2.2.6 *5% Mannitol Solution* — Prepare a 5% (by weight), dissolve and dilute 5 g of reagent grade Mannitol powder (ACS) to 100 g using water meeting the criteria for Type E1.1 in ASTM D5127.

8.2.3 *Sample Preparation*

8.2.3.1 *All Elements* — In a clean environment, place 200 g of sample into a PTFE dish. Add 5 mL of the 5% Mannitol solution and 100 mL of the 1% nitric acid solution. Slowly evaporate on a hot plate to dryness avoiding loss of sample by effervescence or spattering until 1 to 2 mL remain. Dissolve the residue with 5 mL of the 1% nitric acid solution by heating on a hot plate at low temperature for several minutes. Cool to room temperature, dilute to 50 mL with 1% nitric acid, add an appropriate concentration of the indium internal standard and mix well. Prepare a reagent blank.

8.2.4 *Analysis*

8.2.4.1 Using the prepared solutions and blanks, analyze potassium, calcium and iron by graphite furnace atomic absorption (GFAA) and the remaining elements by inductively coupled plasma mass spectrometry (ICP-MS). For calibration, the standards are made up with the 1% nitric acid solution and a suitable concentration of the indium internal standard.

NOTE 4: Current ICP-MS technology (2004) allows analysis of all trace metals, see Grade 3 Procedures and SEMI C1, ‘ICP-MS’.



9 Grade 3 Procedures

NOTE 5: Each laboratory is responsible for verifying the validity of the method within its own operation.

9.1 See §7, which contains procedures for the following tests:

- Assay
- Color (APHA)
- Residue after Evaporation
- Water (H_2O)

9.2 Analysis of Anions

9.2.1 Ion chromatography has been used successfully for the determination of anion impurities at the values specified for each of the following: chloride (Cl^-), nitrate (NO_3^-), phosphate (PO_4^{3-}) and sulfate (SO_4^{2-}). Alternate methods may be used as long as they are validated for each anion according to SEMI C1, ‘Method Validation’.

9.2.2 The ion chromatography columns used were as follows²: ASRS-ULTRA Suppressor (2 mm), IonPac AG9-HC Concentrator (4 mm), IonPac AG9-HC Guard (2 mm), IonPac AS9-HC Analytical (2 mm), IonPac ATC-1 Anion Trap (4 mm).

9.2.3 The following ion chromatography conditions were used:

Eluent	8.0 mM sodium carbonate/1.5mM sodium hydroxide
Eluent Flow Rate	0.25 mL/min
Rinsing Flow Rate	2.0 mL/min
Sample Volume	5 mL
Detection	Suppressed Conductivity
Suppressor	ASRS, Autosuppression External Water Mode
Current Setting	100 mA
Sample	2-propanol (high purity)
Anion Spike Level	5 ppb

9.3 Trace Metals Analysis

9.3.1 ICP-MS has been used successfully for the determination of trace metal impurities at the values specified for each of the following: aluminium (Al), arsenic (As), antimony (Sb), barium (Ba), boron (B), cadmium (Cd), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), Vanadium (V) and zinc (Zn). Alternate methods may be used as long as method validation according to SEMI C1 can be demonstrated.

9.3.2 Special Reagents

9.3.2.1 *Nitric Acid, Ultrapure* — Use nitric acid specified for low metal content.

9.3.2.2 *Water* — The water used for all dilution, calibration and standards should meet, at a minimum, the criteria for Type E1.2 in ASTM D5127 in regard to trace metal analysis.

9.3.2.3 *1% Nitric Acid Solution* — Dilute 10 mL of nitric acid with water to 1 L.

9.3.2.4 *Indium Internal Standard* — Make up an indium internal standard solution in 1% nitric acid to a concentration of 20 mg/mL from an appropriate indium standard solution.

9.3.3 Sample Preparation for Trace Metals

9.3.3.1 *All Elements* — In a clean environment, place 200 g of sample into a PTFE dish. Add 100 mL of 1% nitric acid solution. Slowly evaporate avoiding loss of sample by effervescence or spattering until 1 to 2 mL remain. Add 5 mL 1% nitric acid solution, heat at a low temperature for several minutes to ensure dissolution. Cool to room

2 Dionex, Sunnyvale, CA, (www.dionex.com) or equivalent



temperature, dilute to 50 mL with 1% nitric acid, add an appropriate volume of indium internal standard and mix well. Prepare a reagent blank.

9.3.4 Analysis

9.3.4.1 Using the prepared solutions and reagent blank, analyze all trace metals by ICP-MS using analytical conditions optimized for each element. Calibration standards are made in 1% nitric acid solution with the same concentration of indium internal standard as in ¶9.3.3.1.

10 Grade 4 Procedures

NOTE 6: Each laboratory is responsible for verifying the validity of the method within its own operation.

10.1 See §7, which contains procedures for the following tests:

- Assay
- Color (APHA)
- Residue after Evaporation
- Water (H_2O)

10.2 See §9, which contains procedures for the following tests:

- Assay of Anions
- Assay of Trace Metals

11 Grade 5 Procedures

11.1 This section does not apply to this chemical.

12 VLSI Grade Procedures

12.1 Specific procedures for this grade do not exist. Refer to §7 and §8 for available procedures.

13 Tier A Procedures

13.1 This section does not apply to this chemical.

14 Tier B Procedures

14.1 This section does not apply to this chemical.

15 Tier C Procedures

15.1 This section does not apply to this chemical.

16 Tier D Procedures

16.1 This section does not apply to this chemical.



Table 1 Impurity Limits and Other Requirements for 2-Propanol

Previous SEMI Reference #	C1.15-96	C7.7-93	C11.7-0698	C8.7-92	-
	Grade 1	Grade 2	VLSI Grade	Grade 3	Grade 4
	(Specification)	(Specification)	(Guideline)	(Specification)	(Specification)
Assay ($\text{CH}_3\text{CHOHCH}_3$)	99.5% min	99.8% min	99.7% min	99.8% min	99.8% min
Color (APHA)	10 max	10 max	10 max	10 max	10 max
Residue after Evaporation	5 ppm max	5 ppm max	5 ppm max	5 ppm max	5 ppm max
Resistivity	-	-	15 MegOhm cm min	-	-
Solubility in Water	To pass test	-	-	-	-
Chloride (Cl)	0.2 ppm max	200 ppb max	0.2 ppm max	50 ppb max	50 ppb max
Nitrate (NO_3^-)	-	-	-	50 ppb max	50 ppb max
Phosphate (PO_4^{3-})	0.5 ppm max	500 ppb max	0.5 ppm max	50 ppb max	50 ppb max
Sulfate (SO_4^{2-})	-	-	1 ppm max	50 ppb max	50 ppb max
Aldehydes and Ketones (as CH_3COCH_3)	-	-	100 ppm max	-	-
Water (H_2O)	0.05% max	0.05% max	0.05% max	0.05% max	0.05% max
Acidity	0.2 $\mu\text{eq/g}$ max	-	0.2 $\mu\text{eq/g}$ max	-	-
Alkalinity	0.1 $\mu\text{eq/g}$ max	-	0.1 $\mu\text{eq/g}$ max	-	-
Aluminum (Al)	0.1 ppm max	10 ppb max	0.05 ppm max	1 ppb max	100 ppt max
Antimony (Sb)	-	-	0.01 ppm max	1 ppb max	100 ppt max
Arsenic (As)	-	-	0.01 ppm max	1 ppb max	100 ppt max
Arsenic and Antimony (as As)	0.01 ppm max	10 ppb max	-	-	-
Barium (Ba)	-	10 ppb max	0.02 ppm max	1 ppb max	100 ppt max
Beryllium (Be)	-	10 ppb max	0.01 ppm max	-	-
Bismuth (Bi)	-	10 ppb max	0.02 ppm max	-	-
Boron (B)	0.01 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Cadmium (Cd)	-	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Calcium (Ca)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Chromium (Cr)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Cobalt (Co)	-	10 ppb max	0.01 ppm max	-	-
Copper (Cu)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Gallium (Ga)	-	10 ppb max	0.01 ppm max	-	-
Germanium (Ge)	-	10 ppb max	-	-	-
Gold (Au)	0.1 ppm max	5 ppb max	0.02 ppm max	-	-
Indium (In)	-	-	0.01 ppm max	-	-
Iron (Fe)	0.1 ppm max	5 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Lead (Pb)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Lithium (Li)	-	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Magnesium (Mg)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Manganese (Mn)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Molybdenum (Mo)	-	10 ppb max	0.01 ppm max	-	-
Nickel (Ni)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Niobium (Nb)	-	10 ppb max	-	-	-
Platinum (Pt)	-	-	0.02 ppm max	-	-
Potassium (K)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Silicon (Si)	-	-	-	-	-
Silver (Ag)	-	5 ppb max	0.01 ppm max	-	-
Sodium (Na)	0.1 ppm max	10 ppb max	0.1 ppm max	1 ppb max	100 ppt max



Strontium (Sr)	-	10 ppb max	0.01 ppm max	-	-
Tantalum (Ta)	-	10 ppb max	-	-	-
Thallium (Tl)	-	10 ppb max	0.01 ppm max	-	-
Tin (Sn)	0.1 ppm max	10 ppb max	0.02 ppm max	1 ppb max	100 ppt max
Titanium (Ti)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Vanadium (V)	-	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Zinc (Zn)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Zirconium (Zr)	-	10 ppb max	0.01 ppm max	-	-
Particles in bottles (size, #/mL)	#1	#1	#1	#1	#1

^{#1} Due to the limitations of current particle counters, particle size and number are to be agreed upon between supplier and user. See SEMI C1, 'Calibration and Measurement Method for Particles in Liquids'.

^{#2} Residue after Evaporation - since no new analytical data is available at this time the specification level remains at Grade 2 level.

^{#3} Water (H₂O) - since no new analytical data is available at this time the specification level remains at Grade 2 level.

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

SEMI C42-0699

SPECIFICATION FOR SODIUM HYDROXIDE PELLETS

This specification was technically approved by the Global Process Chemicals Committee and is 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 C1.22 in its entirety. Originally published in 1978.

1 Purpose

1.1 The purpose of this document is to standardize requirements for sodium hydroxide pellets 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 sodium hydroxide pellets 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 sodium hydroxide pellets 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)

6.1 Not applicable.

7 Requirements

7.1 The requirements for sodium hydroxide pellets for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

8.1 *Assay* — Weigh accurately 35 to 40 g of sample in a closed weighing bottle. Transfer to a 500 mL volumetric flask, dissolve with carbon dioxide-free water, cool to 25°C, dilute to the mark with carbon dioxide-free water, and mix thoroughly. Transfer 25 mL of this solution to a 500 mL glass-stoppered flask, dilute with 175 mL of carbon dioxide-free water, add 5 mL of barium chloride reagent solution, shake, and allow to stand for five minutes. Add 0.15 mL of phenolphthalein indicator solution and titrate with 1 N

hydrochloric acid. (Save this solution for the sodium carbonate test.)

$$\% \text{ Assay} = \frac{\text{mL} \times \text{N of HCl} \times 80.00}{\text{Weight of sample (g)}}$$

8.2 *Sodium Carbonate (Na_2CO_3)* — To the above titrated solution, add 0.15 mL of methyl orange indicator and continue the titration with 1 N hydrochloric acid, making sure that no insoluble carbonate remains. The additional hydrochloric acid is equivalent to that consumed by the sodium carbonate.

$$\% \text{ Na}_2\text{CO}_3 = \frac{\text{mL} \times \text{N of HCl} \times 106.00}{\text{Weight of sample (g)}}$$

8.3 *Ammonium Hydroxide Precipitate* — Weigh 10 g of sample and dissolve in about 100 mL of water. Cautiously add 15 mL of sulfuric acid to 15 mL of water, cool; then cautiously add the mixture to the solution of the sample, and evaporate to dense fumes. Cool, dissolve the residue in 130 mL of hot water, and add ammonium hydroxide until the solution is just basic to methyl red. Heat to boiling, filter, wash with hot water, and ignite. The weight of the residue should not exceed 2 mg.

8.4 *Insoluble Matter* — Dissolve 50 g of sample in 250 mL of carbon dioxide-free water in a 500 mL stoppered flask and cool to 25°C. The solution should be clear and colorless. Filter through a tared, medium-porosity filtering crucible and wash thoroughly with hot water. Dry at 105°C for 1 hour, cool, and weigh the residue. The result of the residue should not exceed 2.5 mg.

8.5 *Stock Solution for 8.6-8.11, 8.13-8.14* — Dissolve 50 g of sample in 250 mL of carbon dioxide-free water and dilute with such water to 500 mL. One mL contains 0.10 g of sample. Store in a plastic container.

8.6 *Nitrogen Compounds (as N)* — Dilute 20 mL of the Stock Solution with 50 mL of water in a flask connected through a spray trap to a condenser, the end of which dips beneath the surface of 10 mL of 0.1 N hydrochloric acid. For the standard, take 50 mL of water in a similar flask, and add 10 mL of the Stock Solution and 1 mL of the nitrogen standard solution. To each flask add 0.5 g of aluminum wire in small pieces, allow to stand for 1 hour, and slowly distill about 35

mL. To each distillate, add 2 mL of freshly boiled 10% sodium hydroxide reagent solution, dilute with water to 50 mL, and add 2 mL of Nessler reagent. Any color in the solution of the sample should not exceed that in the standard.

8.7 Chloride — Dilute 10 mL of the Stock Solution with water to 100 mL. Filter, if necessary, through a chloride-free filter. To 20 mL of this solution add 5 mL of water, 1 mL of nitric acid and 1 mL of silver nitrate reagent solution. Any turbidity developed should not exceed that produced by 0.01 mg of chloride ion (Cl⁻) in an equal volume of solution containing the quantities of reagents used in the test.

8.8 Phosphate — To 40 mL of Stock Solution, add 10 mL of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow to stand for 2 hours at room temperature. Any blue color developed should not exceed that produced by 0.02 mg of phosphate ion (PO₄³⁻) is treated as the sample.

8.9 Sulfate — To 20 mL of the Stock Solution, add 5 mL of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 20 mL of water and evaporate again to dryness. Dissolve the residue in 20 mL of water, filter if necessary and add 1 mL of dilute hydrochloric acid (1 + 19) and 1 mL of barium chloride reagent solution. Allow to stand for 10 minutes. Any turbidity developed should not exceed that produced when 0.04 mg of sulfate ion (SO₄²⁻) is treated as the sample.

8.10 Heavy Metals (as Ag) — To 60 mL of the Stock Solution, cautiously add 20 mL of nitric acid. For the standard, add 0.05 mg of silver ion (Ag) to 10 mL of the Stock Solution and cautiously add 20 mL of nitric acid. Evaporate both solutions to dryness over a low flame or an electric hot plate. Dissolve each residue in about 20 mL of water, filter if necessary through a chloride-free filter, and dilute with water to 25 mL. Adjust the pH of the standard and sample solutions to between 3 and 4 (using a pH meter) with 1 N acetic acid or ammonium hydroxide (10% NH₃). Dilute each solution with water to 40 mL and stir. Add 10 mL of freshly prepared hydrogen sulfide water to each solution and stir again. Any color developed in the sample should not exceed that in the standard.

8.11 Iron — Neutralize 10 mL of the Stock Solution with hydrochloric acid using phenolphthalein indicator solution, add 2 mL in excess, and dilute with water to 50 mL. Add 30 to 50 mg of ammonium peroxydisulfate crystals and 3 mL of ammonium thiocyanate reagent solution. Any red color developed should not exceed

that produced when 0.01 mg of iron ion (Fe) is treated as the sample.

8.12 Mercury — To each of two thoroughly clean 125 mL conical flasks add 20 mL of water and 1 mL of 4% potassium permanganate solution. To one add 5.5 g of sample. To the other add 0.5 g of sample and 0.5 mg of mercury ion (Hg). To each flask slowly add 18 mL of hydrochloric acid, with constant swirling. Heat to boiling, allow to cool, and dilute to 100 mL. Determine mercury in 10 mL aliquots by the cold vapor (flameless) atomic absorption method using 1 mL of 10% hydroxylamine hydrochloride reagent solution and 2 mL of 10% stannous chloride reagent solution for the reduction. The peak obtained with the sample solution should not be larger than that of the standard mercury solution.

8.13 Nickel — Dilute 20 mL of the Stock Solution to 50 mL with water and neutralize with hydrochloric acid using a pH meter. Dilute to 85 mL and add ammonium hydroxide to make the solution just basic (pH 8). Add 5 mL of bromine water, 5 mL of alcoholic 1% dimethylglyoxime solution in alcohol, and 5 mL of 10% sodium hydroxide reagent solution. Any red color developed should not exceed that produced when 0.02 mg of nickel ion (Ni) is treated as the sample.

8.14 Potassium — Dilute 10 mL of the Stock Solution with water to 100 mL to form the Sample Solution. To prepare the Standard Solution, add 0.2 mg of potassium ion (K) to 10 mL of the Stock Solution and dilute with water to 100 mL.

8.14.1 Using flame emission spectroscopy, observe the emission of the Standard Solution at the 767 nm potassium line. Observe the emission of the Sample Solution at the 767 nm potassium line and also at a wavelength of 750 nm. The difference between the intensities observed for the Sample Solution at 767 nm and 750 nm should not exceed the difference observed at 767 nm between the Sample Solution and Standard Solution (see SEMI C1, Section 3.7, Flame Emission Spectroscopy).

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 This section does not apply to this chemical.

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.

Table 1 Impurity Limits and Other Requirements for Sodium Hydroxide Pellets

Previous SEMI Reference #	C1.22-95
	Grade 1
	(Specification)
Assay (NaOH)	97.0% min
Sodium Carbonate (Na ₂ CO ₃)	0.5% max
Ammonium Hydroxide Precipitate	0.02% max
Insoluble Matter	50 ppm max
Nitrogen Compounds (as N)	10 ppm max
Chloride (Cl)	50 ppm max
Phosphate (PO ₄)	5 ppm max
Sulfate (SO ₄)	20 ppm max
Heavy Metals (as Ag)	10 ppm max
Iron (Fe)	10 ppm max
Mercury (Hg)	0.1 ppm max
Nickel (Ni)	10 ppm max
Potassium (K)	0.02% max

NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

The user's attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any

item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.

SEMI C43-0699

SPECIFICATION FOR SODIUM HYDROXIDE, 50% SOLUTION

This specification was technically approved by the Global Process Chemicals Committee and is 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.

1 Purpose

1.1 The purpose of this document is to standardize requirements for 50% sodium hydroxide solution 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 50% sodium hydroxide solution 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 50% sodium hydroxide solution 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)

6.1 Not applicable.

7 Requirements

7.1 The requirements for 50% sodium hydroxide solution for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

NOTE 1: Each laboratory is responsible for verifying the validity of the method within its own operation.

8.1 *Assay* — Accurately weigh 70 to 80 g of sample in a closed weighing bottle. Transfer to a 500 mL volumetric flask, dilute in carbon dioxide-free water, cool to 25°C, dilute to the mark with carbon dioxide-free water, and mix thoroughly. Transfer 25.0 mL of this solution to a 500 mL glass-stoppered flask, dilute with 175 mL of carbon dioxide-free water, add 5 mL of barium chloride reagent solution, shake and allow to

stand for five minutes. Add 0.15 mL of phenolphthalein indicator solution and titrate with standardized 1 N hydrochloric acid. (Save this solution for the sodium carbonate test.)

$$\% \text{ Assay} = \frac{\text{mL} \times \text{N of HCl} \times 80.00}{\text{Weight of Sample(g)}}$$

8.2 *Sodium Carbonate* — To the above titrated solution, add 0.15 mL of methyl orange indicator and continue the titration with 1 N hydrochloric acid, making sure that no insoluble carbonate remains. The additional hydrochloric acid is equivalent to that consumed by the sodium carbonate.

$$\% \text{ Na}_2\text{CO}_3 = \frac{\text{mL} \times \text{N of HCl} \times 106.0}{\text{Weight of Sample(g)}}$$

8.3 *Ammonium Hydroxide Precipitate* — Weigh 20 g of sample into 250 mL beaker and dissolve in about 100 mL of water.

8.3.1 Cautiously add 15 mL of sulfuric acid to 15 mL of water in a 100 mL glass beaker, cool; then cautiously add the mixture to the solution of the sample, and evaporate to dense fumes. Cool, dissolve the residue in 130 mL of hot water, and add ammonium hydroxide until the solution is just basic to methyl red.

8.3.2 Heat to boiling, filter, wash with hot water and ignite. The weight of the residue should not exceed 2 mg.

8.4 *Insoluble Matter* — Dilute 50 g of sample in 250 mL of carbon dioxide-free water in a 500 mL stoppered flask and cool to 25°C. The solution should be clear and colorless. Filter through a tared, medium porosity filtering crucible and wash thoroughly with hot water. Dry the crucible at 105°C for 1 hour, cool and weigh the residue. The weight of the residue should not exceed 2.5 mg.

8.5 *Stock Solution for Sections 8.6 through 8.13* — Add 50 g of sample to 250 mL of carbon dioxide-free water and dilute with such water to 500 mL. One mL contains 0.10 g of sample. Store in a plastic container.

8.6 *Nitrogen Compounds (as N)* — Dilute 30 mL of the stock solution with 50 mL of water in a flask connected through a spray trap to a condenser, the end of which dips beneath the surface of 10 mL of 0.1 N

hydrochloric acid. For the standard, take 50 mL of water in a similar flask, and add 10 mL of the stock solution and 1 mL of the nitrogen standard solution (0.010mg N/ml). To each flask add 0.5 g of aluminum wire in small pieces, and allow the solution to stand for 1 hour. Slowly distill to about 35 mL of solution. To each distillate add 2 mL of freshly boiled 10% sodium hydroxide reagent solution, dilute with water to 50 mL and add 2 mL of Nessler reagent. Any color in the solution of the sample should not exceed that in the standard.

8.7 *Chloride* — Dilute 10 mL of the stock solution with water to 100 mL. Filter, if necessary, through a chloride-free filter. To 25 mL of this solution, add 5 mL of water, 1 mL of nitric acid, and 1 mL of silver nitrate reagent solution. Any turbidity developed should not exceed that produced by 0.01 mg of chloride (Cl⁻) ion in an equal volume of solution containing the quantities of reagent used in the test

8.8 *Phosphate* — To 40 mL of the stock solution, add 10 mL of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow the solution to stand for 2 hours at room temperature. Any blue color developed should not exceed that produced when 0.02 mg of phosphate (PO₄³⁻) ion is treated as the sample.

8.9 *Sulfate* — To 40 mL of the stock solution, add 5 mL of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 20 mL of water and evaporate again to dryness. Dissolve the residue in 20 mL of water, filter if necessary, and add 1 mL of dilute hydrochloric acid (1+19) and 1 mL of barium chloride reagent solution. Allow the solution to stand for 10 minutes. Any turbidity developed should not exceed that produced when 0.04 mg of sulfate (SO₄²⁻) ion is treated as the sample.

8.10 *Heavy Metals (as Ag)* — To 60 mL of the stock solution cautiously add 20 mL of nitric acid. For the standard add 0.05 mg of silver ion (Ag) to 10 mL of the stock solution and cautiously add 20 mL of nitric acid. Evaporate both solutions to dryness over a low flame or on an electric hot plate. Dissolve each residue in about 20 mL of water, filter if necessary through a chloride-free filter, and dilute with water to 25 mL. Adjust the pH of the standard and sample solutions to between 3 and 4 (using a pH meter) with 1 N acetic acid or ammonium hydroxide (10% NH₃). Dilute each solution with water to 40 mL and stir. Add 10 mL of freshly prepared hydrogen sulfide water to each solution and stir again. Any color developed in the sample should not exceed that in the standard.

8.11 *Iron* — Neutralize 20 mL of the stock solution with hydrochloric acid, using a phenolphthalein indicator solution, add 2 mL of HCl in excess, and dilute with water to 50 mL. Add 30 to 50 mg of ammonium peroxydisulfate crystals and 3 mL of ammonium thiocyanate reagent solution. Any red color developed should not exceed that produced when 0.01 mg of iron (Fe) ion is treated as the sample.

8.12 *Nickel* — Dilute 40 mL of the stock solution to 50 mL with water and neutralize with hydrochloric acid using a pH meter. Dilute to 85 mL with water and add ammonium hydroxide to make the solution barely basic (pH 8). Add 5 mL of bromine water and 5 mL of alcoholic 1% dimethylglyoxime solution in alcohol, and 5 mL of 10% sodium hydroxide reagent solution. Any red color developed should not exceed that produced when 0.02 mg of nickel (Ni) ion is treated as the sample.

8.13 *Potassium* — Dilute 10 mL of the stock solution with water to 100 mL to form the sample solution. To prepare the standard solution, add 0.1 mg of potassium (K) ion to 10 mL of the stock solution and dilute with water to 100 mL.

8.13.1 Using flame emission spectroscopy, observe the emission of the standard solution at the 767 nm potassium line. Observe the emission of the sample solution at the 767 nm potassium line and also at a wavelength of 750 nm. The difference between the intensities observed for the Sample Solution at 767 nm and 750 nm should not exceed the difference observed at 767 nm between the sample solution and standard solution (see SEMI Cl, Section 3.7, Flame Emission Spectroscopy).

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 This section does not apply to this chemical.

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.

Table 1 Impurity Limits and Other Requirements for Sodium Hydroxide, 50% Solution

Previous SEMI Reference #	--
	Grade 1
	(Specification)
Assay (NaOH)	50.0 - 52.0%
Ammonium Hydroxide Precipitate	0.01% max
Insoluble Matter	50 ppm max
Nitrogen Compounds (as N)	5 ppm max
Chloride (Cl)	40 ppm max
Phosphate (PO ₄)	5 ppm max
Sodium Carbonate (Na ₂ CO ₃)	0.1% max
Sulfate (SO ₄)	10 ppm max
Heavy Metals (as Ag)	10 ppm max
Iron (Fe)	5 ppm max
Nickel (Ni)	5 ppm max
Potassium (K)	0.01% max

NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

The user's attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.



SEMI C44-0301

SPECIFICATIONS AND GUIDELINES FOR SULFURIC ACID

These specifications and guidelines 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 October 17, 1999. Initially available at www.semi.org February 2001; to be published March 2001. This document replaces SEMI C1.16, C7.8, C8.8, and C11.5 in their entirety. Originally published in 1978, 1990, 1992, and 1994 respectively; previously published October 2000.

1 Purpose

1.1 The purpose of this document is to standardize requirements for sulfuric acid 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 sulfuric acid 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 sulfuric acid used in the semiconductor industry.
- 2.2 The VLSI grade purity level is typically required by semiconductor devices with geometries of 0.8–1.2 microns.
- 2.3 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 limitations prior to use.

3 Limitations

- 3.1 None.

4 Referenced Standards

4.1 SEMI Standards

SEMI C1 — Specifications for Reagents

4.2 ASTM Standards¹

ASTM D5127 — Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

¹ American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555. Website: www.astm.org

5 Terminology

- 5.1 None.

6 Physical Property (for information only)

Density at 25°C	1.83 g/mL
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7 Requirements

7.1 The requirements for sulfuric acid for Grades 1 and 2, VLSI Grade, and Tiers B and C are listed in Table 1.

8 Grade 1 Procedures

NOTE 2: Each laboratory is responsible for verifying the validity of the method within its own operation.

8.1 *Assay* — Accurately weigh 1 mL of sample in a small glass-stoppered conical flask. Cautiously add 30 mL of water, cool, add 0.1 mL of methyl orange indicator solution, and titrate with standardized 1.0 N sodium hydroxide to a red to yellow color change.

$$\% \text{ Assay} = \frac{\text{mL} \times \text{N of NaOH} \times 4.904}{\text{Weight of sample (g)}}$$

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 *Chloride* — Place 40 mL of water in each of two beakers. To one, add carefully 27 mL (50 g) of sample and to the other, 27 mL (50 g) of chloride-free sulfuric acid and 0.005 mg of chloride ion (Cl). Cool to room temperature and add 1 mL of nitric acid and 1 mL of silver nitrate reagent solution. Mix well; if necessary, make the volume of each solution identical by adding water. After 10 minutes, measure the turbidity of each solution using a suitable nephelometer. The turbidity in the sample should be no greater than the standard.

NOTE 3: Prepare chloride-free sulfuric acid in a hood by gently fuming sulfuric acid in a crucible or dish for at least 30

minutes. Cool and transfer to a tightly-capped glass bottle for storage.

8.4 Nitrate — Prepare the following solutions:

Sample Solution A:	Cautiously add 27 mL (50 g) of sample to 1.0 mL of water, dilute to 50 mL with brucine sulfate reagent solution and mix.
Control Solution B:	Cautiously add 27 mL (50 g) of sample to 1.0 mL of the standard nitrate solution containing 0.01 mg of nitrate ion (NO_3^-) per mL, dilute to 50 mL with brucine sulfate reagent solution and mix.
Blank Solution C:	Use 50 mL of brucine sulfate reagent solution.

8.4.1 Heat the three solutions in a preheated (boiling) water bath for 10 minutes. Cool rapidly in an ice bath to room temperature. Set a photometer at 410 nm and, using 1-cm cells, adjust the instrument to read zero absorbance with Blank Solution C in the light path, then determine the absorbance of Sample Solution A. Adjust the instrument to read zero absorbance with Sample Solution A in the light path and determine the absorbance of Control Solution B. The absorbance of Sample Solution A should be no greater than that of Control Solution B.

8.5 Phosphate — Evaporate 11 mL (20 g) of sample to dryness in a platinum dish in a hood. Dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution and 1 mL p-(methylamino)phenol sulfate reagent solution. Allow to stand at room temperature for 2 hours. Any blue color produced should be no greater than that produced when 0.01 mg of phosphate ion (PO_4^{3-}) is treated as the sample.

8.6 Arsenic and Antimony (as As) — To 109 mL (200 g) of sample in a beaker, add 5 mL of nitric acid and evaporate to about 10 mL in a hood. Cool. Cautiously add 10 mL of water, and again evaporate to about 5 mL. Cool, and cautiously wash into a generator flask with water to make a volume of 35 mL. Proceed as described in General Method for Arsenic (and Antimony) under SEMI C1, Section 3.4.5, starting with the first sentence which begins: "Swirl the flask..." Any red color in the silver diethyldithiocarbamate solution from the sample should be no greater than that of the standard containing 0.001 mg of arsenic (As).

8.7 Trace Metal Analysis

8.7.1 Gold (Au) — Analyze by graphite atomic absorption using the manufacturer's recommended procedure. This technique has been shown to give satisfactory results using a 1:4 dilution and Zeeman background correction. Each laboratory must determine

the appropriate dilution and background correction for its instrument to meet the specification limit.

8.7.2 The following method has given satisfactory results in determining trace metal impurities at the value specified for each of the following trace metals: aluminum (Al), boron (B), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), and zinc (Zn). Alternate methods may be used as long as appropriate studies demonstrate recovery between 75–125% of a known sample spike for half of the value of each specified item.

8.7.3 Special Reagents

8.7.3.1 *Water* — The water used for all the dilution, calibration and standards should meet at a minimum the criteria for Type E1.1 in ASTM D5127 in regard to cation analysis.

8.7.3.2 *Indium Internal Standard* — Make up the indium internal standard solution to a concentration of 20 $\mu\text{g}/\text{mL}$ (ppm) from the appropriate concentrated indium standard solution.

8.7.3.3 *Nitric Acid, Ultrapure* — Use nitric acid specified for ultra low metal ion content.

8.7.3.4 *1% Nitric Acid Solution* — Dilute 10 mL of ultrapure nitric acid to 1 L using water meeting the criteria for Type E1.1 in ASTM D5127.

8.7.3.5 *2% Nitric Acid Solution* — Dilute 20 mL of ultrapure nitric acid to 1 L using water meeting the criteria for Type E1.1 in ASTM D5127.

8.7.3.6 *Hydrochloric Acid, Ultrapure* — Use hydrochloric acid specified for ultra low metal ion content.

8.7.3.7 *2% Hydrochloric Acid Solution* — Dilute 20 mL of ultra-pure hydrochloric acid to 1 L using water meeting the criteria for Type E1.1 in ASTM D5127.

8.7.4 Sample Preparation

8.7.4.1 *Boron* — In a clean environment, dilute 1.00 g sample with 15.0 g of Type E1.1 water and add 15 μL of the indium internal standard. Run a reagent blank.

8.7.4.2 *Tin* — In a clean environment, place 100 g of sulfuric acid in a quartz crucible. Slowly evaporate to dryness on a hot plate avoiding loss of sample by effervescence or spattering. Cool. Add 2 mL of high purity 12 M hydrochloric acid and 10 mL of water. Cover, and digest on the hot plate for 10 minutes. Cool. Transfer quantitatively to a 50 mL volumetric flask using water for rinsing and dilution to volume. Run a reagent blank.

8.7.5 All Other Elements — In a clean environment, place 100 g of sulfuric acid in a quartz crucible. Slowly evaporate on a hot plate avoiding loss of sample by effervescence or spattering until approximately 2 mL of liquid remains. Cool. Add carefully, 1 mL of high purity, 70% nitric acid. While maintaining volume, carefully warm several minutes to dissolve any residue. Cool. Transfer quantitatively to a 50 mL volumetric flask using 2% nitric acid and dilute to volume. Run a reagent blank.

8.7.6 Analysis — Using the prepared solutions and blanks, analyze boron by inductively coupled plasma mass spectrometry (ICP/MS). Using the acid sample and reagent blank, analyze group I elements by flame atomic absorption spectroscopy. Analyze all other elements by plasma emission spectroscopy.

NOTE 4: Analysis of dilute sulfuric acid can produce rapid corrosion of nickel cones commonly used in inductively coupled plasma mass spectrometry, platinum cones should be considered as alternative when performing this analysis.

9 Grade 2 Procedures

NOTE 5: Each laboratory is responsible for verifying the validity of the method within its own operation.

9.1 Non-Metal Impurities — See Section 8, which contains procedures for the following tests:

Assay

Color (APHA)

Chloride

Nitrate

Phosphate

9.2 Trace Metals Analysis

9.2.1 The following method has given satisfactory results in determining metal ion impurities at the values specified for each of the following metals: aluminum (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), bismuth (Bi), boron (B), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), gallium (Ga), germanium (Ge), gold (Au), iron (Fe), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), niobium (Nb), potassium (K), silver (Ag), sodium (Na), strontium (Sr), tantalum (Ta), tin (Sn), titanium (Ti), vanadium (V), zinc (Zn), and zirconium (Zr). Alternate methods may be used as long as appropriate studies demonstrate a recovery between 75–125% of a known sample spike for half of the value of each specified element.

9.2.2 Special Reagents

9.2.2.1 Nitric Acid, Ultrapure — Use nitric acid specified for low metal ion content.

9.2.2.2 1% Nitric Acid Solution — Dilute 10 mL of ultrapure nitric acid to 1 L using water meeting the criteria for Type E1.1 in ASTM D5127.

9.2.2.3 Water — The water used for all the dilution, calibration and standards should meet at a minimum the criteria for Type E1.1 in ASTM D5127 in regard to cation analysis.

9.2.2.4 Indium Internal Standard — Make up the indium internal standard solution to a concentration of 20 µg/mL (ppm) from the appropriate concentrated indium standard solution.

9.2.3 Sample Preparation

9.2.3.1 Chromium, Cobalt, Lithium, Manganese, Nickel, Titanium, Vanadium, and Zinc — In a clean environment, place 1.00 g of sample into a clean quartz dish. Slowly evaporate on a hot plate to dryness avoiding loss of sample by effervescence or spattering. Dissolve the residue with 5 mL of the 1% nitric acid solution by heating on a hot plate at low temperature for several minutes. Cool to room temperature, dilute to 15 mL with 1% nitric acid, add 15 µL of the indium internal standard, mix well. Run a reagent blank.

9.2.3.2 All Other Elements — In a clean environment, dilute 1.00 g sample with 15.0 g of Type E1.1 water and add 15 µL of the indium internal standard. Run a reagent blank.

9.2.4 Analysis

9.2.4.1 Using the prepared solutions and blanks, analyze sodium, potassium, calcium and iron by graphite furnace atomic absorption (GFAA) and the remaining elements by inductively coupled plasma mass spectrometry (ICP/MS). For calibration, the standards are made up with the 1% nitric acid solution and the indium internal standard such that the final indium concentration is 20 ng/g. For boron and tantalum, the standards for calibration must be matrix matched with equal amounts of sulfuric acid certified to have both elements below 1 µg/mL in the concentrated acid.

NOTE 6: Analysis of dilute sulfuric acid can produce rapid corrosion of nickel cones commonly used in inductively coupled plasma mass spectrometry, platinum cones should be considered as alternative when performing this analysis.

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 Specific procedures for this grade do not exist.
Refer to Sections 8 and 9 for available procedures.

14 Tier A Procedures

14.1 This section does not apply to this chemical.

15 Tier B Procedures

15.1 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 Chemical Reagent 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.

16 Tier C Procedures

16.1 Standardized test methods are being developed for all parameters at the purity levels indicated. The Process Chemicals Committee considers a test method to be valid 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.

17 Tier D Procedures

17.1 This section does not apply to this chemical.

Table 1 Impurity Limits and Other Requirements for Sulfuric Acid

Previous SEMI Reference #	C1.16-96	C7.8-94	C11.5-94	C8.8-92	
	Grade 1	Grade 2	VLSI Grade	Tier B	Tier C
	(Specification)	(Specification)	(Guideline)	(Guideline)	(Guideline)
Assay (H_2SO_4)	95.0–97.0%	95.0–97.0 %	95.0–97.0%	95.0–97.0%	95.0–97.0%
Color (APHA)	10 max	10 max	10 max	10 max	10 max
Residue after Ignition	--	--	3 ppm max	--	--
Chloride (Cl)	0.1 ppm max	100 ppb max	0.1 ppm max	50 ppb max	50 ppb max
Nitrate (NO_3)	0.2 ppm max	200 ppb max	0.2 ppm max	100 ppb max	100 ppb max
Phosphate (PO_4)	0.5 ppm max	500 ppb max	0.5 ppm max	100 ppb max	100 ppb max
Matters Reducing KMnO_4 (as O)	--	--	2 ppm max	--	--
Aluminum (Al)	0.2 ppm max	10 ppb max	0.02 ppm max	1 ppb max	100 ppt max
Ammonium (NH_4)	--	--	1 ppm max	--	--
Antimony (Sb)	--	5 ppb max	--	1 ppb max	100 ppt max
Arsenic (As)	--	10 ppb max	--	1 ppb max	100 ppt max
Arsenic and Antimony (as As)	0.005 ppm max	--	0.01 ppm max	--	--
Barium (Ba)	--	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Beryllium (Be)	--	10 ppb max	0.01 ppm max	1 ppb max	--
Bismuth (Bi)	--	10 ppb max	0.05 ppm max	1 ppb max	--
Boron (B)	0.02 ppm max	20 ppb max	0.02 ppm max	1 ppb max	100 ppt max
Cadmium (Cd)	--	10 ppb max	0.01 ppm max	1 ppb max	--
Calcium (Ca)	0.3 ppm max	10 ppb max	0.05 ppm max	1 ppb max	100 ppt max
Chromium (Cr)	0.2 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Cobalt (Co)	--	5 ppb max	0.01 ppm max	1 ppb max	--
Copper (Cu)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Gallium (Ga)	--	10 ppb max	0.01 ppm max	1 ppb max	--
Germanium (Ge)	--	10 ppb max	0.01 ppm max	1 ppb max	--
Gold (Au)	0.3 ppm max	5 ppb max	0.02 ppm max	1 ppb max	--
Indium (In)	--	--	0.01 ppm max	--	--
Iron (Fe)	0.2 ppm max	10 ppb max	0.05 ppm max	1 ppb max	100 ppt max
Lead (Pb)	0.3 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Lithium (Li)	--	10 ppb max	0.01 ppm max	1 ppb max	--

Previous SEMI Reference #	C1.16-96	C7.8-94	C11.5-94	C8.8-92	
	Grade 1	Grade 2	VLSI Grade	Tier B	Tier C
	(Specification)	(Specification)	(Guideline)	(Guideline)	(Guideline)
Magnesium (Mg)	0.3 ppm max	10 ppb max	0.05 ppm max	1 ppb max	100 ppt max
Manganese (Mn)	0.2 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Molybdenum (Mo)	--	10 ppb max	0.02 ppm max	1 ppb max	--
Nickel (Ni)	0.1 ppm max	10 ppb max	0.01 ppm max	1 ppb max	100 ppt max
Niobium (Nb)	--	10 ppb max	--	1 ppb max	--
Platinum (Pt)	--	--	0.05 ppm max	--	--
Potassium (K)	0.3 ppm max	10 ppb max	0.05 ppm max	1 ppb max	100 ppt max
Silicon (Si)	--	--	--	1 ppb max	--
Silver (Ag)	--	10 ppb max	0.01 ppm max	1 ppb max	--
Sodium (Na)	0.3 ppm max	10 ppb max	0.1 ppm max	1 ppb max	100 ppt max
Strontium (Sr)	--	10 ppb max	0.02 ppm max	1 ppb max	--
Tantalum (Ta)	--	10 ppb max	--	1 ppb max	--
Thallium (Tl)	--	10 ppb max	0.05 ppm max	1 ppb max	--
Tin (Sn)	0.2 ppm max	10 ppb max	0.05 ppm max	1 ppb max	100 ppt max
Titanium (Ti)	0.3 ppm max	10 ppb max	0.05 ppm max	1 ppb max	100 ppt max
Vanadium (V)	--	10 ppb max	0.01 ppm max	1 ppb max	--
Zinc (Zn)	0.2 ppm max	10 ppb max	0.05 ppm max	1 ppb max	100 ppt max
Zirconium (Zr)	--	10 ppb max	0.05 ppm max	1 ppb max	--
Particles in bottles (size, #/mL)	≥ 1.0 µm, 25 max	≥ 0.5 µm, 25 max	≥ 0.5 µm, 250 max	(See NOTE 1.)	(See NOTE 1.)

NOTE 1: Due to the limitations of current particle counters, particle size and number are to be agreed upon between supplier and user. See SEMI C1, Section 3.9 for particle counting methodology.

NOTICE: SEMI makes no warranties or representations as to the suitability of the standard set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

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SEMI C45-0301

SPECIFICATION AND GUIDELINE FOR TETRAETHYLOLTHOSILICATE (TEOS)

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 October 17, 1999. Initially available at www.semi.org February 2001; to be published March 2001. This document replaces SEMI C7.13 in its entirety. Originally published in 1991; previously published June 1999.

1 Purpose

1.1 The purpose of this document is to standardize requirements for tetraethylorthosilicate 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 tetraethylorthosilicate 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 tetraethylorthosilicate used in the semiconductor industry.

2.2 These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

3 Limitations

3.1 None.

4 Referenced Standards

4.1 SEMI Standards

SEMI C1 — Specifications for Reagents

4.2 ASTM Standards¹

ASTM D5127 — Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

5 Terminology

5.1 None.

¹ American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555. Website: www.astm.org

6 Physical Property (for information only)

6.1 Not applicable.

7 Requirements

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

8 Grade 1 Procedures

NOTE 2: Each laboratory is responsible for verifying the validity of the method within its own operation.

8.1 *Assay* — Analyze the sample by gas chromatography (see SEMI C1, Section 3.1, Guidelines for Assay by Wide Bore Column Gas Chromatography). The parameters cited have given satisfactory results.

8.1.1 *Column* — 30 meter x 530 micron fused silica capillary, coated with 5 micron film of DB-1 or equivalent (100% methyl silicone which has been surface bonded and cross linked).

8.1.2 *Column Temperature* — 60°C isothermal for 1 minute, then programmed to 200°C at 20°C/min.

Injector Temperature:	120°C
Detector Temperature:	260°C
Sample Size:	1 µL splitless
Carrier Gas:	Helium at 3 mL/min
Detector:	Thermal Conductivity
Retention Times (min)	
Water	2.0
Ethanol	2.5
TEOS	7.8

8.2 *Color* — Dilute 1.0 mL of platinum-cobalt stock solution (APHA No. 500) to 100 mL with water. Compare this standard (APHA No. 5) 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 *Water* — The cell is dried with a dry nitrogen gas stream for twenty minutes. Reagents are added and a blank (no sample) is run. Add 5 g of sample to the dry

Coulometric Karl Fischer water analyzer. Results are obtained in ppm by dividing the micrograms of water found in the sample by weight of TEOS in grams.

8.4 Trace Metals Analysis —The following method has given satisfactory results in determining trace metal impurities at the specified value for each of the following trace metals: aluminum (Al), arsenic (As), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), and zinc (Zn). Alternate methods may be used as long as appropriate studies demonstrate recovery between 75–125% of a known sample spike for half of the value of each specified item.

8.4.1 Special Reagents — All water used in the *Trace metals Analysis* and *Boron Analysis* should be a deionized water meeting the criteria for Type E1.1 in ASTM D5127.

8.4.1.1 Mixed Acid — 1% HF - 2% HNO₃ prepared by dilution of ultra pure acids with appropriate weights of deionized water.

8.4.1.2 Standards — Composite standard solutions containing 0, 1, 2, and 4 ppb multi-element standards are prepared by diluting with mixed acid of appropriate weights for ICP-MS. Single-element standards are prepared (Ca, Fe, K, Na) for GFAA.

8.4.2 Sample Preparation — In a clean environment, place 100 g of sample in a clean PFA Teflon closed bottle equipped with an inlet and outlet tube and evaporate under a nitrogen purge at approximately 110°C. When dry, close the inlet and the outlet tube and digest the residue at 100°C for 30 minutes with 7 mL of mixed acid solution. Cool and transfer the contents with deionized water. Dilute the transferred contents with deionized water to a final volume of 14 mL.

8.4.3 Analysis — Analyze the samples by graphite furnace atomic absorption (GFAA) for sodium, calcium, potassium, and iron. Analyze the sample for all other metallic elements by inductively coupled plasma mass spectrometry (ICP-MS).

8.5 Boron Analysis — The following method has given satisfactory results in the determination of trace levels of boron (B). Alternate methods may be used as long as appropriate studies demonstrate recovery between 75–125% of a known sample spike for half of the specified value.

8.5.1 Standard — Standards of 0, 2, and 4 ppm boron are prepared by dilution of an aqueous boron stock solution with deionized water meeting the criteria for Type E1.1 in ASTM D5127.

8.5.2 Sample Preparation — A 100 g sample of TEOS is extracted with 10 mL of deionized water, that meets the criteria for Type E1.1 in ASTM D5127, by vigorously shaking the solution for minimum of 10 minutes on a mechanical shaker. Allow the layers to separate and remove the aqueous portion to a separate container.

8.5.3 Analysis — The 10 mL water extract is analyzed for boron by inductively coupled plasma atomic emission spectroscopy (ICP-AES) at 249.68 nm.

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 Chemical Reagent 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.



Table 1 Impurity Limits and Other Requirements for Tetraethylorthosilicate

Previous SEMI Reference #	--	C7.13-95
	Grade 1	Tier A
	(Specification)	(Guideline)
Assay	99.99% min	99.99% min
Water (see NOTE 1)	20 ppm max	20 ppm max
Color APHA	5 max	5 max
Aluminum (Al)	1 ppb max	1 ppb max
Arsenic (As)	1 ppb max	1 ppb max
Barium (Ba)	--	1 ppb max
Bismuth (Bi)	--	1 ppb max
Boron (B)	10 ppb max	10 ppb max
Calcium (Ca)	1 ppb max	1 ppb max
Chromium (Cr)	1 ppb max	1 ppb max
Cobalt (Co)	--	1 ppb max
Copper (Cu)	1 ppb max	1 ppb max
Gallium (Ga)	--	1 ppb max
Gold (Au)	--	1 ppb max
Iron (Fe)	1 ppb max	1 ppb max
Lead (Pb)	1 ppb max	1 ppb max
Lithium (Li)	--	1 ppb max
Magnesium (Mg)	1 ppb max	1 ppb max
Manganese (Mn)	1 ppb max	1 ppb max
Mercury (Hg)	--	1 ppb max
Nickel (Ni)	1 ppb max	1 ppb max
Potassium (K)	1 ppb max	1 ppb max
Silver (Ag)	--	1 ppb max
Sodium (Na)	1 ppb max	1 ppb max
Strontium (Sr)	--	1 ppb max
Tin (Sn)	1 ppb max	1 ppb max
Titanium (Ti)	1 ppb max	1 ppb max
Zinc (Zn)	1 ppb max	1 ppb max
Particles in bottles: size, #/mL	≥ 0.3 µm, 50 max	≥ 0.3 µm, 50 max

NOTE 1: Chemical Property: Water reacts with TEOS to form Ethanol.

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

The user's attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.

SEMI C46-0699

GUIDELINE FOR 25% TETRAMETHYLMONIUM HYDROXIDE

This guideline was technically approved by the Global Process Chemicals Committee and is 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 C7.14 in its entirety. Originally published in 1991.

1 Purpose

1.1 The purpose of this document is to standardize requirements for 25% tetramethylammonium hydroxide 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 25% tetramethylammonium hydroxide 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 25% tetramethylammonium hydroxide used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

4.1 None.

5 Terminology

5.1 None.

6 Physical Property (for information only)

Not applicable.

7 Requirements

7.1 The requirements for 25% tetramethylammonium hydroxide for Tier A are listed in Table 1.

8 Grade 1 Procedures

8.1 This section does not apply to this chemical.

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.

Table 1 Impurity Limits and Other Requirements for 25% Tetramethylammonium Hydroxide

Previous SEMI Reference #	C7.14-91
	Tier A
	(Guideline)
Assay (wt %)	25.0 ± 0.1
Carbonate (as CO ₃ 2)	300 ppb max
Total Halides (as Cl ⁻¹)	5 ppb max
Color (APHA)	10 max
Aluminum (Al)	10 ppb max
Antimony (Sb)	10 ppb max
Arsenic (As)	10 ppb max
Barium (Ba)	10 ppb max
Beryllium (Be)	10 ppb max
Bismuth (Bi)	10 ppb max
Boron (B)	10 ppb max
Cadmium (Cd)	10 ppb max
Calcium (Ca)	10 ppb max
Chromium (Cr)	10 ppb max
Cobalt (Co)	10 ppb max
Copper (Cu)	10 ppb max
Gallium (Ga)	10 ppb max
Germanium (Ge)	10 ppb max
Gold (Au)	5 ppb max
Iron (Fe)	5 ppb max
Lead (Pb)	10 ppb max
Lithium (Li)	10 ppb max
Magnesium (Mg)	10 ppb max
Manganese (Mn)	10 ppb max
Molybdenum (Mo)	10 ppb max
Nickel (Ni)	10 ppb max
Potassium (K)	5 ppb max
Silver (Ag)	10 ppb max
Sodium (Na)	5 ppb max
Strontium (Sr)	10 ppb max
Thallium (Tl)	10 ppb max
Tin (Sn)	10 ppb max
Zinc (Zn)	10 ppb max
Zirconium (Zr)	10 ppb max
Particles in bottles: size, #/mL	≥0.5 µm, 50 max



NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

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SEMI C47-0699

GUIDELINE FOR TRANS 1,2 DICHLOROETHYLENE

This guideline was technically approved by the Global Process Chemicals Committee and is 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 C7.20 in its entirety. Originally published in 1995.

1 Purpose

1.1 The purpose of this document is to standardize requirements for trans 1,2 dichloroethylene 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 trans 1,2 dichloroethylene 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 trans 1,2 dichloroethylene used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

4.1 None.

5 Terminology

5.1 None.

6 Physical Property (for information only)

Density at 25°C	1.70 g/mL
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7 Requirements

7.1 The requirements for trans 1,2 dichloroethylene for Tier A are listed in Table 1.

8 Grade 1 Procedures

8.1 This section does not apply to this chemical.

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 Chemical Reagent 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.

Table 1 Impurity Limits and Other Requirements for Trans 1,2 Dichloroethylene

Previous SEMI Reference #	C7.20-95
	Tier A
	(Guideline)
Assay	99.9% min
Water (H ₂ O)	20 ppb max
Acidity	0.3 µeq/g max
Aluminum (Al)	10 ppb max
Antimony (Sb)	10 ppb max
Arsenic (As)	10 ppb max
Boron (B)	5 ppb max
Calcium (Ca)	10 ppb max
Chromium (Cr)	5 ppb max
Copper (Cu)	5 ppb max
Gold (Au)	5 ppb max
Iron (Fe)	10 ppb max
Lead (Pb)	5 ppb max
Magnesium (Mg)	5 ppb max
Manganese (Mn)	5 ppb max
Nickel (Ni)	5 ppb max
Potassium (K)	10 ppb max
Sodium (Na)	10 ppb max
Tin (Sn)	5 ppb max
Titanium (Ti)	5 ppb max
Zinc (Zn)	5 ppb max
Particles in bottles: size, #/mL	≥1.0 µm, 35 max

NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

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rights or copyrights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.

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:

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

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 (PO_4) 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.

**Table 1 Impurity Limits and Other Requirements for
1,1,1-Trichloroethane, Furnace Grade**

Previous SEMI Reference #	C1.26-92	C7.15-91
	Grade I	Tier A
	(Specification)	(Guideline)
Assay (CCl ₃ ,CH ₃)	99.9% min	99.95% min
Color (APHA)	10 max	5 max
Water (H ₂ O)	25 ppm max	15 ppm max
Acidity	0.3 µeg/g max	--
Acidity (as HCl)	--	10 ppm max
Phosphate (PO ₄)	0.01 ppm max	--
Residue after Evaporation	5 ppm max	--
Aluminum (Al)	0.005 ppm max	1 ppb max
Antimony (Sb)	0.01 ppm max	1 ppb max
Arsenic (As)	0.01 ppm max	2 ppb max
Barium (Ba)	0.01 ppm max	1 ppb max
Bismuth (Bi)	0.005 ppm max	1 ppb max
Boron (B)	0.001 ppm max	1 ppb max
Cadmium (Cd)	0.005 ppm max	1 ppb max
Calcium (Ca)	0.01 ppm max	1 ppb max
Chromium (Cr)	0.005 ppm max	1 ppb max
Cobalt (Co)	0.005 ppm max	1 ppb max
Copper (Cu)	0.005 ppm max	1 ppb max
Gallium (Ga)	0.01 ppm max	1 ppb max
Gold (Au)	0.005 ppm max	1 ppb max
Iron (Fe)	0.01 ppm max	1 ppb max
Lead (Pb)	0.005 ppm max	1 ppb max
Lithium (Li)	0.005 ppm max	1 ppb max
Magnesium (Mg)	0.005 ppm max	1 ppb max
Manganese (Mn)	0.005 ppm max	1 ppb max
Mercury (Hg)	0.005 ppm max	1 ppb max
Nickel (Ni)	0.01 ppm max	1 ppb max
Phosphorous (P)	--	1 ppb max
Potassium (K)	0.005 ppm max	1 ppb max
Silicon (Si)	0.01 ppm max	--
Silver (Ag)	0.005 ppm max	1 ppb max
Sodium (Na)	0.005 ppm max	1 ppb max
Strontium (Sr)	0.01 ppm max	1 ppb max
Tin (Sn)	0.005 ppm max	1 ppb max
Titanium (Ti)	0.01 ppm max	1 ppb max
Zinc (Zn)	0.005 ppm max	1 ppb max
Particles in bottles: size, #/mL	--	≥ 0.2 µm, 5 max ≥ 0.1 µm, 30 max

NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

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SEMI C49-0699

GUIDELINE FOR TRIMETHYLBORATE

This guideline was technically approved by the Global Process Chemicals Committee and is 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 C7.21 in its entirety. Originally published in 1995.

1 Purpose

1.1 The purpose of this document is to standardize requirements for trimethylborate 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 trimethylborate 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 trimethylborate used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

4.1 None.

5 Terminology

5.1 None.

6 Physical Property (for information only)

Density at 20°C	0.915 g/mL
Boiling Point	68.7°C

7 Requirements

7.1 The requirements for trimethylborate for Tier A are listed in Table 1.

8 Grade 1 Procedures

8.1 This section does not apply to this chemical.

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.

Table 1 Impurity Limits and Other Requirements for Trimethylborate

Previous SEMI Reference #	C7.21-95
	Tier A
	(Guideline)
Assay ($\text{B}(\text{CH}_3\text{O})_3$)	99.95% min
Water (H_2O)	25 ppm max
Aluminum (Al)	5 ppb max
Arsenic (As)	5 ppb max
Barium (Ba)	5 ppb max
Bismuth (Bi)	5 ppb max
Calcium (Ca)	10 ppb max
Chromium (Cr)	5 ppb max
Cobalt (Co)	5 ppb max
Copper (Cu)	5 ppb max
Gallium (Ga)	5 ppb max
Gold (Au)	5 ppb max
Iron (Fe)	5 ppb max
Lead (Pb)	5 ppb max
Lithium (Li)	5 ppb max
Magnesium (Mg)	5 ppb max
Manganese (Mn)	5 ppb max
Nickel (Ni)	5 ppb max
Potassium (K)	5 ppb max
Silver (Ag)	5 ppb max
Sodium (Na)	5 ppb max
Strontium (Sr)	5 ppb max
Tin (Sn)	5 ppb max
Zinc (Zn)	5 ppb max

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SEMI C50-0699

GUIDELINE FOR TRIMETHYLPHOSPHITE

This guideline was technically approved by the Global Process Chemicals Committee and is 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 C7.22 in its entirety. Originally published in 1995.

1 Purpose

1.1 The purpose of this document is to standardize requirements for trimethylphosphite 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 trimethylphosphite 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 trimethylphosphite used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

4.1 None.

5 Terminology

5.1 None.

6 Physical Property (for information only)

Density at 20°C	1.05 g/mL
Boiling Point	112°C

7 Requirements

7.1 The requirements for trimethylphosphite for Tier A are listed in Table 1.

8 Grade 1 Procedures

8.1 This section does not apply to this chemical.

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 supplier. 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.

Table 1 Impurity Limits and Other Requirements for Trimethylphosphite

Previous SEMI Reference #	C7.22-95
	Tier A (Guideline)
Assay ($\text{P}(\text{CH}_3\text{O})_3$)	99.0% min
Color (APHA)	10 max
Aluminum (Al)	5 ppb max
Arsenic (As)	10 ppb max
Barium (Ba)	5 ppb max
Bismuth (Bi)	5 ppb max
Calcium (Ca)	5 ppb max
Chromium (Cr)	5 ppb max
Cobalt (Co)	5 ppb max
Copper (Cu)	5 ppb max
Gallium (Ga)	5 ppb max
Gold (Au)	5 ppb max
Iron (Fe)	5 ppb max
Lead (Pb)	5 ppb max
Lithium (Li)	5 ppb max
Magnesium (Mg)	5 ppb max
Manganese (Mn)	5 ppb max
Mercury (Hg)	5 ppb max
Nickel (Ni)	5 ppb max
Potassium (K)	5 ppb max
Silver (Ag)	5 ppb max
Sodium (Na)	15 ppb max
Strontium (Sr)	5 ppb max
Tin (Sn)	5 ppb max
Titanium (Ti)	5 ppb max
Zinc (Zn)	10 ppb max

NOTICE: These standards do not purport to address safety issues, if any, associated with their use. It is the responsibility of the user of these standards to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

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SEMI C51-0699

SPECIFICATION FOR XYLENES

This specification was technically approved by the Global Process Chemicals Committee and is 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 C1.21 in its entirety. Originally published in 1978.

1 Purpose

1.1 The purpose of this document is to standardize requirements for xylenes 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 xylenes 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 xylenes used in the semiconductor industry.

2.2 This material is a mixture of ortho, meta, and para isomers and ethylbenzene. Xylene (o-, m-, and p- isomers and ethylbenzene) 99.0% min and Ethylbenzene 18% max.

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	0.86 g/mL
Boiling Range	136 - 144°C

7 Requirements

7.1 The requirements for xylenes for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

8.1 *Assay* — Assay the sample by gas chromatography (see SEMI C1, Section 3.1, Guidelines for Assay by Wide Bore Gas Chromatography). The parameters cited have given satisfactory results.

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:	175°C
Detector Temperature:	250°C
Sample Size:	0.2 µL splitless
Carrier Gas:	Helium at 3 mL/min
Detector:	Thermal Conductivity or Flame Ionization
Approximate Retention Times (min):	
Toulene	14.4
Ethylbenzene	17.0
p-xylene	16.9
m-xylene	17.2
o-xylene	17.8

8.1.1 Measure the areas under all peaks by any convenient means and calculate the concentration of all components in area percent (see SEMI C.1, Section 3.11, Guidelines for Assay by Wide Bore Column Gas Chromatography).

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 38 mL (33 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 should be required.

8.4 *Residue After Evaporation* — Evaporate 232 mL (200 g) of sample to dryness. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (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 23 mL (20 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.

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

8.6 *Chloride* — To a 100 mL separatory funnel, add 12 mL (10 g) of sample and 40 mL of water. Shake well for 30 seconds and allow the two layers to separate. Discard the sample (upper) layer. To a 20 mL portion of the water layer add 1 mL of nitric acid and 1 mL of silver nitrate reagent solution. Any turbidity produced should be no greater than that of a standard containing 0.015 mg of chloride ion (Cl⁻) in an equal volume of solution and the amounts of reagents used.

8.7 *Phosphate* — To 12 mL (10 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 and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow to stand at room temperature for 2 hours. Any blue color should be no greater than that produced when 0.01 mg of phosphate ion (PO₄³⁻) is treated as the sample.

8.8 *Arsenic and Antimony (as As)* — Evaporate 116 mL (100 g) of sample in a 250 mL beaker to a small volume in a hood. Add 50 mL of water and again evaporate to a small volume. Repeat the evaporation three times with water additions. 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 trioxide. Cool, cautiously add 10 mL of water, and again 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, Section 3.4.5, starting with the sentence which begins, "Swirl the flask..." Any red color in the silver diethyldithiocarbamate solution of the sample should be no greater than that of the standard containing 0.001 mg of arsenic (As).

8.9 *Trace Element Contents* — By a suitable emission spectrographic procedure, determine for each of the specified trace elements that its content is not greater than the stated specification limit (see SEMI C1, Section 3.5, Guidelines for Determination of Trace Elements by Emission Spectrography).

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 This section does not apply to this chemical.

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.

Table 1 Impurity Limits and Other Requirements for Xylenes

	Previous SEMI Reference #	C1.21-92
		Grade 1
		(Specification)
Color (APHA)	10 max	
Acidity	0.3 $\mu\text{eq/g}$ max	
Residue after Evaporation	5 ppm max	
Water (H_2O)	0.02% max	
Chloride (Cl)	3 ppm max	
Phosphate (PO_4)	1 ppm max	
Aluminum (Al)	0.1 ppm max	
Arsenic and Antimony (as As)	0.01 ppm max	
Boron (B)	0.1 ppm max	
Calcium (Ca)	0.1 ppm max	
Chromium (Cr)	0.1 ppm max	
Copper (Cu)	0.1 ppm max	
Gold (Au)	0.1 ppm max	
Iron (Fe)	0.1 ppm max	
Lead (Pb)	0.1 ppm max	
Magnesium (Mg)	0.1 ppm max	
Manganese (Mn)	0.1 ppm max	
Nickel (Ni)	0.1 ppm max	
Potassium (K)	0.1 ppm max	
Sodium (Na)	0.1 ppm max	
Tin (Sn)	0.1 ppm max	
Zinc (Zn)	0.1 ppm max	
Particles in bottles: size, #/mL	$\geq 1.0 \mu\text{m}$, 10 max	

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