SEMI C40-1110 (Reapproved 0618)
SPECIFICATION FOR POTASSIUM HYDROXIDE, 45% SOLUTION

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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.

2 Scope

2.1 The scope of this Document is a grade of 45% potassium hydroxide solution used in the semiconductor industry.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 None.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

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

5 Terminology

5.1 None.

6 Properties (for information only)

Table 1 Properties of Potassium Hydroxide, 45% Solution

Chemical Formula	KOH
CAS Number	1310-58-3
Density at 25°C	1.447 g/mL
Boiling Point	135°C

7 Requirements

7.1 The requirements for 45% potassium hydroxide solution for Grade 1 are listed in Table 2.

8 Grade 1 Procedures

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 (see SEMI C1), shake and allow to stand for five minutes. Add 0.15 mL of phenolphthalein indicator solution (see SEMI C1) and titrate with standardized 1 N hydrochloric acid. (Save this solution for the potassium carbonate test.)

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$$\%\text{Assay} = \frac{\text{mL} \times \text{N of HCl} \times 112.2}{\text{Weight of Sample (g)}} \quad (1)$$

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.

$$\%\ K_2CO_3 = \frac{\text{mL} \times \text{N of HCl} \times 138.2}{\text{Weight of Sample (g)}} \quad (2)$$

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 shall 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. 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 shall not exceed 5.0 mg.

8.5 *Stock Solution for ¶¶ 8.6–8.13* — Add 100 g of sample to 250 mL of carbon dioxide-free water and dilute with such water to 500 mL. 1 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) (see SEMI C1). 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 (see SEMI C1), dilute with water to 50, add 2 mL of Nessler reagent, and mix. Any color in the solution of the sample shall not exceed that in the standard.

8.7 *Chloride* — Dilute l0 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 (see SEMI C1). Any turbidity developed shall not exceed that produced by 0.02 mg of chloride (CI) 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 (see SEMI C1) and l mL of p-(methylamino)phenol sulfate reagent solution (see SEMI C1). Allow the solution to stand for 2 hours at room temperature. Any blue color developed shall 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 (see SEMI C1). Allow to stand for 10 minutes. Any turbidity developed shall 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 (see SEMI C1) to each solution and stir again. Any color developed in the sample shall not exceed that in the standard.

8.11 *Iron* — Neutralize 10 mL of the stock solution with hydrochloric acid, using a phenolphthalein indicator solution (see SEMI C1), add 2 mL of HCI in excess, and dilute with water to 50 mL. Add 30 to 50 mg of ammonium

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