SEMI C39-1105
SPECIFICATION FOR POTASSIUM HYDROXIDE PELLETS

This Standard was technically approved by the Process Chemicals Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on September 8, 2005. Available at www.semi.org in November 2005; originally published in 1978; previously published June 1999.

NOTICE: This Document was completely rewritten in 2005.

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.

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 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.)

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

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.

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

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 Through 8.11, ¶¶8.13 and 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.

NOTE 1: The nitrogen standard solution in the previous section should be as described in SEMI C1.

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 (PO_4) 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 (SO_4) 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 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 *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.

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