INTERNATIONAL STANDARD 3194

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Potassium hydroxide for industrial use — Determination of sulphur compounds — Method by reduction and titrimetry

Hydroxyde de potassium à usage industriel — Dosage des composés soufrés — Méthode par réduction et titrimétrie

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

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No Member Body expressed disapproval of the document.

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Potassium hydroxide for industrial use — Determination of sulphur compounds — Method by reduction and titrimetry

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reduction and titrimetric method for the determination of sulphur compounds in potassium hydroxide for industrial use.

The method is applicable to products of which the content of sulphur compounds, expressed as sulphate (SO_4), is equal to or greater than 5 mg/kg.

2 REFERENCE

ISO 2466, *Potassium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.*

3 PRINCIPLE

Reduction of the sulphur compounds to hydrogen sulphide by heating with a mixture of hydriodic acid and hypophosphorous acid.

Absorption of the hydrogen sulphide, entrained in a current of oxygen-free nitrogen, in a mixture of sodium hydroxide and acetone.

Titration of the sulphide with standard volumetric mercury(II) nitrate solution in the presence of dithizone as indicator.

4 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

4.1 Acetone.

4.2 Nitrogen, oxygen-free.

4.3 Sodium hydroxide, approximately 1 N solution.

4.4 Potassium permanganate and mercury(II) chloride solution, to purify the nitrogen (4.2).

Dissolve first 2 g of potassium permanganate ($KMnO_4$) and then 7 g of mercury(II) chloride ($HgCl_2$), in 100 ml of water and filter the solution.

4.5 Pyrogallol solution, to purify the nitrogen (4.2).

Dissolve 15 g of pyrogallol in 25 ml of water and add, while cooling, 150 ml of a 30 % (*m/m*) potassium hydroxide solution.

4.6 Reduction solution

Place in the flask (J) of the apparatus (5.3), shaking after each addition :

- 50 ml of hypophosphorous acid (H_3PO_2), ρ approximately 1,21 g/ml, about 50 % (*m/m*) solution;
- 100 ml of hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution;
- 120 ml of hydriodic acid, ρ approximately 1,97 g/ml, about 67 % (*m/m*) solution.

Assemble the flask and the reflux condenser (K) then, while passing a slow flow of the nitrogen (4.2), boil for 4 h.

Cool to room temperature under a flow of the nitrogen (4.2) and store the reagent away from direct sunlight in an amber glass bottle previously flushed with the nitrogen (4.2) and fitted with a ground glass stopper.

4.7 Sodium sulphate, 0,001 M standard reference solution.

Weigh, to the nearest 0,000 1 g, 0,142 0 g of anhydrous sodium sulphate, previously dried at 110 °C and cooled in a desiccator. Introduce into a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

1 ml of this solution corresponds to 96 μg of SO_4.

4.8 Mercury(II) nitrate, 0,05 M standard volumetric solution (= 0,1 N)

Dissolve 10,85 ± 0,01 g of mercury(II) oxide (HgO) in 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution, dilute to 1 000 ml with water and mix.

NOTE — The strength of the solution thus prepared is sufficiently exact for the small quantities of sulphur compounds to be determined and standardization is therefore unnecessary.

In most laboratories, however, a precisely 0,1 N standard volumetric solution, commonly used for the mercurimetric determination of chlorides, will be available.