INTERNATIONAL STANDARD 995

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Potassium hydroxide for industrial use — Determination of silica content — Reduced molybdosilicate photometric method

Hydroxyde de potassium à usage industriel — Dosage de la silice — Méthode photométrique au molybdosilicate réduit

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 995 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in July 1973.

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

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INTERNATIONAL STANDARD ISO 995-1975 (E)

Potassium hydroxide for industrial use — Determination of silica content — Reduced molybdosilicate photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reduced molybdosilicate photometric method for the determination of the silica content of potassium hydroxide for industrial use. The method is applicable to products having silica (SiO_2) contents exceeding 10 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

Formation of the yellow oxidized molybdosilicate at pH 1,1 ± 0,2, in the presence of boric acid to eliminate interference by fluorides.

Selective reduction of the molybdosilicate with a mixture of aminonaphtholsulphonic acid, (4-amino-3-hydroxy-1-naphthalenesulphonic acid), sodium metabisulphite and sodium sulphite, in the presence of oxalic acid and in a strongly acid medium so as to suppress interference by phosphates.

Photometric measurement of the blue-coloured complex at a wavelength of about 795 nm.

4 REAGENTS

During the analysis use only reagents of recognized analytical reagent grade, and only demineralized water. Store all the reagents in polyethylene bottles.

4.1 Sulphuric acid, approximately 9 N solution.

4.2 Hydrochloric acid, approximately 2 N solution.

4.3 Boric acid, saturated solution (about 48 g/l).

4.4 Oxalic acid, 100 g/l solution.

4.5 Sodium molybdate dihydrate [$Na_2MoO_4.2H_2O$], 140 g/l solution.

Dissolve 35 g of this product in 200 ml of water at about 50 °C in a polyethylene beaker. Cool to room temperature, transfer to a 250 ml one-mark volumetric flask, dilute to the mark and mix. Transfer to a polyethylene bottle.

If necessary, filter the solution before use.

4.6 Reducing solution

a) Dissolve 7 g of anhydrous sodium sulphite in 50 ml of water. Then add 1,5 g of aminonaphtholsulphonic acid (4-amino-3-hydroxy-1-naphthalenesulphonic acid) and dissolve by trituration.

b) Dissolve 90 g of anhydrous sodium metabisulphite in 900 ml of water.

Mix the two solutions a) and b) and dilute to 1 000 ml. Filter, if necessary, and store the solution in a cool place away from direct sunlight. This mixture should not be kept longer than 15 to 20 days.

4.7 Potassium chloride, 60 g/l solution.

4.8 Silica, standard solution, corresponding to 0,500 g of SiO_2 per litre.

In a platinum crucible, weigh to the nearest 0,001 g :

— either 0,500 g of silica (SiO_2) produced from pure silicic acid (H_2SiO_3) calcined at 1 000 °C to constant mass and cooled in a desiccator;

— or 0,500 g of pure quartz, finely ground and previously calcined for 1 h at 1 000 °C and cooled in a desiccator.

Add 5 g of anhydrous sodium carbonate to the crucible. Mix well, preferably with a platinum spatula, and fuse carefully. Allow to cool, add warm water, heat moderately until complete dissolution, cool, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Transfer immediately to a polyethylene bottle.

1 ml of this standard solution contains 0,500 mg of SiO_2.

4.9 Silica, standard solution, corresponding to 10 mg of SiO_2 per litre.

Take 20,0 ml of the silica standard solution (4.8), transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,010 mg of SiO_2.

Prepare this standard solution at the time of use.

4.10 Phenolphthalein, 10 g/l solution in ethanol 95 % (*V*/*V*).

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Spectrophotometer or

5.2 Photoelectric absorptiometer, with 2 cm cells, fitted with suitable filters ensuring a maximum transmission at about 795 nm.

NOTE — If such filters are not available, operate at about 680 nm, with 4 cm cells.

6 PROCEDURE

6.1 Test portion

6.1.1 Weigh, to the nearest 0,01 g, in a 100 ml polyethylene beaker, 10,5 ± 0,1 g of the solution A (see 4.3 of ISO 2466), stored in a silica-free vessel and containing 50 g of the test sample in 1 000 ml. During this operation, prevent the solution from coming into contact with glass.

6.1.2 In addition, determine the mass, to the nearest 0,01 g, of 10,0 ml of the solution A, taken with the aid of a pipette or a burette, to enable the mass of the test portion (6.1.1) to be converted to a volume when the results are calculated.

6.2 Blank test

At the same time as the determination carry out a blank test, following the same procedure and using the same quantities of all the reagents as in the determination (6.4), but replacing the 10,0 ml of the solution A by 10,0 ml of the potassium chloride solution (4.7).

6.3 Preparation of calibration curve

6.3.1 *Preparation of the standard matching solutions,* for photometric measurements with 2 cm cells at a wavelength of about 795 nm or with 4 cm cells for photometric measurements at about 680 nm.

Into a series of four 100 ml one-mark volumetric flasks, introduce the volumes of the standard silica solution (4.9) shown in the following table.

Standard silica solution (4.9)	Corresponding mass of SiO_2
ml	mg
0*	0
2,0	0,02
5,0	0,05
10,0	0,10

* Compensation solution.

Add 10,0 ml of the potassium chloride solution (4.7), dilute to 25 ml and add, shaking after each addition, 8,0 ml of the hydrochloric acid solution (4.2), 20 ml of the boric acid solution (4.3) and 10 ml of the sodium molybdate solution (4.5).

The pH of the solution should now be about 1,1. Wait 10 min and then add, shaking after each addition, 5 ml of the oxalic acid solution (4.4) and 20 ml of the sulphuric acid solution (4.1). Allow to stand for 2 min, then add 2 ml of the reducing solution (4.6), dilute to the mark and mix.

6.3.2 *Photometric measurements*

After at least 10 min, but not more than 40 min, carry out the photometric measurements using the spectrophotometer (5.1) at a wavelength of about 795 nm or the photoelectric absorptiometer (5.2), fitted with appropriate filters, after having adjusted the instrument to zero absorbance against the compensation solution.

6.3.3 *Preparation of the calibration chart*

Plot a graph having, for example, the content, in milligrams, of silica (SiO_2) contained in 100 ml of standard matching solution on the abscissa and the corresponding values of absorbance on the ordinate.

6.4 Determination

6.4.1 *Colour development*

Add 1 drop of the phenolphthalein solution (4.10) to the test portion (6.1) and neutralize with the hydrochloric acid solution (4.2). Transfer quantitatively to a 100 ml one-mark volumetric flask, rinsing with 10 ml of water, and add, shaking after each addition, 8,0 ml of the hydrochloric acid solution (4.2), 20 ml of the boric acid solution (4.3) and 10 ml of the sodium molybdate solution (4.5). Wait for 10 min, then add, stirring after each addition, 5 ml of the oxalic acid solution (4.4) and 20 ml of the sulphuric acid solution (4.1). Allow to stand for 2 min, then add 2 ml of the reducing solution (4.6), dilute to the mark and mix.

6.4.2 *Photometric measurement*

After at least 10 min, but not more than 40 min, carry out the photometric measurement according to the procedure described in 6.3.2, after having adjusted the instrument to zero absorbance against the blank test solution (6.2).

7 EXPRESSION OF RESULTS

By means of the calibration curve (6.3.3), determine the quantity of SiO_2 corresponding to the value of the photometric measurement.

The silica (SiO_2) content is given, in milligrams per kilogram, by the formula

$$m_1 \times \frac{1\,000}{m_2 \times 10/m_3} \times \frac{1\,000}{m_0} = \frac{m_1\,m_3}{m_0\,m_2} \times 10^5$$

where

m_0 is the mass, in grams, of the test sample used to prepare solution A;

m_1 is the mass, in milligrams, of SiO_2 found by the determination;

m_2 is the mass, in grams, of the test portion (6.1.1);

m_3 is the mass, in grams, of 10,0 ml of the solution A (see 6.1.2).

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or the International Standard to which reference is made, or regarded as optional.

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