INTERNATIONAL STANDARD 984

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Sodium hydroxide for industrial use — Determination of silica content — Reduced silico-molybdic complex photometric method

Hydroxyde de sodium à usage industriel — Dosage de la silice — Méthode photométrique au complexe silicomolybdique réduit

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 984 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in September 1973.

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

Austria	India	Spain
Belgium	Ireland	Switzerland
Bulgaria	Italy	Thailand
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
Egypt, Arab Rep. of	Poland	U.S.S.R.
France	Portugal	Yugoslovia
Germany	Romania	
Hungary	South Africa, Rep. of	

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 984-1969, of which it constitutes a technical revision.

Printed in Switzerland

Sodium hydroxide for industrial use — Determination of silica content — Reduced silico-molybdic complex photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reduced silico-molybdic complex photometric method for the determination of the silica content of sodium hydroxide for industrial use. The method is applicable to products having silica (SiO_2) contents exceeding 10 mg/kg.

2 REFERENCE

ISO 3195, *Sodium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.*[1)]

3 PRINCIPLE

Formation of the yellow oxidized silico-molybdic complex at pH 1,1 ± 0,2, in the presence of boric acid to suppress interference by fluorides.

Selective reduction of this complex with a mixture of amino-naphthol sulphonic acid (4-amino-3-hydroxynaphthalene-1-sulphonic 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 reagent 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

4.6.1 Dissolve 7 g of anhydrous sodium sulphite in 50 ml of water. Then add 1,5 g of 4-amino-3-hydroxynaphthalene-1-sulphonic acid and dissolve by trituration.

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

Mix the two solutions (4.6.1) and (4.6.2) and dilute to 1 000 ml. Filter, if necessary, store the solution in a cool place away from direct sunlight and renew it every 15 to 20 days.

4.7 Sodium chloride, 70 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 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 completely dissolved, 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.

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

Take 20,0 ml of the standard silica solution (4.8), transfer into 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 95 % (*V/V*) ethanol.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Spectrophotometer or

5.2 Photoelectric absorptiometer with 2 cm cells and 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, 13 ± 0,1 g of the solution A (see 4.3 of ISO 3195) stored in a silica-free vessel and containing 40 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

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents used for the determination (6.4) but replacing the 10,0 ml of the solution A by 10,0 ml of the sodium chloride solution (4.7).

6.3 Preparation of calibration curve

6.3.1 *Preparation of the standard colorimetric solution,* 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 polyethylene beakers, 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

Treat the contents of each beaker as follows.

Add 10,0 ml of the sodium chloride solution (4.7), dilute to 25 ml and add, swirling after each addition, 7,5 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, swirling 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), transfer quantitatively to a 100 ml one-mark volumetric flask, 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 suitable 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 numbers of milligrams of silica (SiO_2) contained in 100 ml of standard colorimetric solutions 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), already contained in a 100 ml polyethylene beaker, and neutralize with the hydrochloric acid solution (4.2). Add 10 ml of water and, swirling after each addition, 7,5 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, swirling 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), transfer quantitatively to a 100 ml one-mark volumetric flask, 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 specified 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 content, expressed as milligrams of silica (SiO_2) per kilogram, is given by the formula

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

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 solution A (see 4.3 of ISO 3195), determined by the procedure specified in 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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