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Sampling and test for sodium hydroxide for industrial use — Part 1 — Determination of silica content

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Foreword

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Draft Uganda Standards adopted by the Technical Committee are widely circulated to stakeholders and the general public for comments. The committee reviews the comments before recommending the draft standards for approval and declaration as Uganda Standards by the National Standards Council.

The committee responsible for this document is Technical Committee UNBS/TC ###, *[name of committee]*, Subcommittee SC ##, *[name of subcommittee]*.

This second/third/… edition cancels and replaces the first/second/… edition (US nnn-n:yyyy), which has been technically revised.

US nnn consists of the following parts, under the general title *Introductory element — Main element*:

- *— Part n: Part title*
- *— Part [n+1]: Part title*
- *— Part [n+2]: Part title*

Sampling and test for sodium hydroxide for industrial use — Part 1— Determination of silica content

1 Scope

This Draft Uganda 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 Normative references

The following referenced documents referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3195, Sodium hydroxide for industrial use - Sampling -- Test sample - Preparation of the main solution for carrying out certain determinations

ISO 3696:1987 Water for analytical laboratory use -- Specification and test methods

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. Water complying with the requirements for ISO 3696:1987 shall be used. 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 [$Na_2Mo0_4.2H_20$]

140 g/l solution.

Dissolve 35 g of this reagent in 200 ml of water at about 50 ºC in a polyethylene beaker. Cool to room temperature, transfer to a 250ml 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 sulphite metabisulphite in 900 ml of water.

Mix the two solutions (4.6.1) and (4.6.2) and dilute to 1000 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.

4.9 Silica, standard solution corresponding to 10 mg of SiO2 per litre.

Take 20,0ml 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 Spectrophotometar 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 10.0ml of the solution A by 10.0ml 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 100ml polyethylene beakers, introduce the volumes of the standard silica solution (4.9) shown in the following table:

Standard silica solution(4.9), ml	Corresponding mass of SiO_2, 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 10,0ml 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 Spectro-photometer (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.

Note: For some grades of sodium hydroxide, it may be necessary to adjust the range of calibration graph to cover the expected silica content.

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 100ml polyethylene beaker, and neutralize with the hydrochloric acid solution (4.2). Add 10 ml of water and, swirling after each addition, 7.5ml 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 2ml of the reducing solution (4.6), transfer quantitatively to a 100ml 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 ($Si0_2$) per kilogram, is given by the formula

$$m_1 x \frac{1000}{m_2 \, x \, 10 / m_3} \, x \frac{1000}{m_0} = \frac{m_1 m_3}{m_0 m_2} \, x 10^5$$

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;

Bibliography

[1] BS 6075: Part 4: 1981 Sampling and test for sodium hydroxide for industrial use Part 4: Determination of silica content

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ICS 71.100.35

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