INTERNATIONAL STANDARD ISO 3697

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Sodium hydroxide for industrial use — Determination of calcium and magnesium contents — Flame atomic absorption method

Hydroxyde de sodium à usage industriel — Dosages du calcium et du magnésium — Méthode par absorption atomique dans la flamme

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FOREWORD

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INTERNATIONAL STANDARD **ISO 3697-1976 (E)**

Sodium hydroxide for industrial use — Determination of calcium and magnesium contents — Flame atomic absorption method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a flame atomic absorption method for the determination of the calcium and magnesium contents of sodium hydroxide for industrial use.

The method is applicable to products having Ca and Mg contents greater than

- 2,5 mg/kg and 0,5 mg/kg respectively, if an acetylene-air flame is used;
- 0,6 mg/kg and 1,0 mg/kg respectively, if an acetylene-dinitrogen monoxide flame is used.

2 REFERENCE

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

3 PRINCIPLE

Acidification of a test portion with hydrochloric acid.

Aspiration of the solution into an acetylene-dinitrogen monoxide or acetylene-air flame, after addition of lanthanum ions in the latter case in order to suppress certain interferences.

Measurement of the absorption of the 422,7 nm and 285,2 nm lines emitted by calcium and magnesium hollow-cathode lamps.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only water doubly distilled in borosilicate glass apparatus with ground joints, or water of equivalent purity.

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution, or approximately 12 N.

4.2 Lanthanum chloride solution, corresponding to 5 g of lanthanum per litre.

NOTE — This solution is not required when an acetylene-dinitrogen monoxide flame is employed.

Prepare either of the following solutions :

4.2.1 Dissolve 5,9 g of lanthanum oxide (La_2O_3) in 15 ml of water and 15 ml of the hydrochloric acid solution (4.1). Dilute to the mark with water in a 1 000 ml one-mark volumetric flask and mix.

4.2.2 Dissolve 13,4 g of lanthanum chloride heptahydrate ($LaCl_3.7H_2O$) in water, dilute to the mark with water in a 1 000 ml one-mark volumetric flask and mix.

Either solution 4.2.1 or 4.2.2 shall pass the following test : 20,0 ml of the solution, diluted to the mark in a 100 ml one-mark volumetric flask, shall not give absorbances of the calcium and magnesium lines greater than those obtained with a standard aqueous solution containing 3 µg of Ca and 1 µg of Mg per 100 ml, when tested in accordance with 6.2.2 and 6.2.3.

In addition, the quantities of Ca and Mg corresponding to the measured absorbances shall be recorded so that account may be taken of these values in checking the purity of the sodium hydroxide (4.3).

4.3 Sodium hydroxide

This product, which is used in the preparation of the sodium chloride solution (4.4), should preferably not have Ca and Mg contents greater than 2,5 mg/kg and 0,5 mg/kg respectively. Check these contents as follows :

Note, from the calibration graphs (6.2.2 and 6.2.3) the absorbances obtained with the standard matching solution No. 0 (6.2.1). The Ca and Mg contents corresponding to these absorbances shall not exceed 2,5 µg and 0,5 µg, respectively, discounting quantities of Ca and Mg found in the check test on the lanthanum chloride solution (4.2) in the case where this solution is used.

4.4 Sodium chloride, 58,5 g/l acid solution.

Place 20,0 g of the sodium hydroxide (4.3) in a 600 ml polyethylene beaker. Dissolve, while cooling, in 100 ml of water, then acidify by adding carefully, while stirring 80 ml of the hydrochloric acid solution (4.1). Transfer quantitatively to 500 ml conical flask, boil for 5 min, allow to cool, transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

NOTE — If very pure sodium chloride is available, this solution (4.4) can be prepared as follows :

Place 29 g of sodium chloride in a 500 ml conical flask. Add 250 ml of water and 40 ml of the hydrochloric acid solution (4.1). Boil for 5 min, allow to cool, transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

4.5 Calcium, standard solution corresponding to 0,100 g of calcium per litre.[1]

Weigh, to the nearest 0,000 1 g, 0,249 7 g of calcium carbonate previously dried at about 250 °C for 2 h and cooled in a desiccator. Place in a 250 ml beaker and dissolve in a mixture of 10 ml of the hydrochloric acid solution (4.1) and 15 ml of water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,100 mg of Ca.

4.6 Calcium, standard solution corresponding to 10 mg of calcium per litre.

Transfer 20,0 ml of the standard calcium solution (4.5) to a 200 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 µg of Ca.

Prepare this solution just before use.

4.7 Magnesium, standard solution corresponding to 0,100 g of magnesium per litre.[1]

Weigh, to the nearest 0,000 1 g, 0,100 0 g of magnesium metal of high purity (minimum 99,95 %). Place in a 250 ml beaker and dissolve in a mixture of 10 ml of the hydrochloric acid solution (4.1) and 15 ml of water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,100 mg of Mg.

4.8 Magnesium, standard solution corresponding to 10 mg of magnesium per litre.

Transfer 20,0 ml of the standard magnesium solution (4.7) to a 200 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 µg of Mg.

Prepare this solution just before use.

5 APPARATUS

Ordinary laboratory apparatus, and

5.1 Atomic absorption spectrophotometer, fitted with a burner fed either with acetylene and dinitrogen monoxide or with acetylene and air.

5.2 Hollow-cathode calcium lamp.

5.3 Hollow-cathode magnesium lamp.

NOTE — All glassware and reagent bottles shall be either of borosilicate glass or of a quality not yielding calcium or magnesium.

6 PROCEDURE

6.1 Test portion

Take 25,0 ml of the solution A, prepared in accordance with ISO 3195 and containing 40 g of the test sample per 1 000 ml, stored in a polyethylene bottle.

6.2 Preparation of the calibration graphs

6.2.1 *Preparation of the standard matching solutions*

Into each of a series of five 100 ml one-mark volumetric flasks, place 25 ml of the acid sodium chloride solution (4.4) and, if using the acetylene-air flame, 20 ml of the lanthanum chloride solution (4.2).

Then add the volumes of the standard calcium solution (4.6) and standard magnesium solution (4.8) indicated in the following table :

Standard matching solution No.	Standard solutions		Corresponding masses	
	calcium (4.6)	magnesium (4.8)	Ca	Mg
	ml	ml	µg	µg
0*	0	0	0	0
1	1,0	0,2	10	2
2	5,0	1,0	50	10
3	10,0	2,0	100	20
4	20,0	4,0	200	40

* Blank test on reagents for calibration graphs.

Dilute to the mark and mix.

6.2.2 *Calibration graphs for calcium*

6.2.2.1 ADJUSTMENT OF THE APPARATUS

Install the hollow-cathode calcium lamp (5.2) in the apparatus (5.1) and leave it switched on for the time necessary to achieve stability. Adjust the lamp current, the attenuation and the slit, to suit the characteristics of the apparatus. Adjust the wavelength in the region of 422,7 nm in order to obtain the maximum absorbance. Adjust the acetylene and air or dinitrogen monoxide pressures according to the characteristics of the aspirator-burner.

6.2.2.2 SPECTROPHOTOMETER MEASUREMENTS

Aspirate the series of standard matching solutions (6.2.1) into the flame and measure the absorbance for each. Take care to keep the rate of aspiration constant throughout the preparation of the calibration graph.

NOTE — Pass water through the burner after each measurement.

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[1] The spectrophotometric standard reference solutions available commercially can also be used.

6.2.2.3 PLOTTING THE CALIBRATION GRAPH

Plot a graph having, for example, the numbers of micrograms of Ca contained in 100 ml of the standard matching solutions as abscissae and the corresponding values of the measured absorbances, reduced by the measured value for the standard matching solution No. 0, as ordinates.

6.2.3 *Calibration graph for magnesium*

Repeat the operations specified in 6.2.2.1, 6.2.2.2 and 6.2.2.3, after having fitted the hollow-cathode magnesium lamp (5.3) to the apparatus and having adjusted the wavelength to about 285,2 nm.

NOTE — The calibration graphs obtained should be linear as far as possible.

6.3 Determination

6.3.1 *Preparation of the test solution.*

Transfer the test portion (6.1) to a 100 ml conical flask, add 20 ml of water and, slowly while stirring, 4,0 ml of the hydrochloric acid solution (4.1). Boil gently for 5 min, cool and transfer quantitatively to a 100 ml one-mark volumetric flask. If using the acetylene-air flame, add 20,0 ml of the lanthanum chloride solution (4.2). Dilute to the mark and mix.

6.3.2 *Blank test*

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of reagents as used in the determination, but omitting the test portion.

6.3.3 *Spectrophotometric measurements*

Following the procedures specified in 6.2.2 and 6.2.3, determine, in order, the absorbances for the following solutions :

- the five standard matching solutions (6.2.1);
- the test solution (6.3.1);
- the blank test solution (6.3.2);
- the standard matching solution (6.2.1) for which the absorbance is closest to that of the test solution.

6.3.4 *Check test*

Verify the absence of interferences by repeating the operations specified in 6.3.1 with the addition of known quantities of the standard calcium and magnesium solutions (4.6 and 4.8). If interferences are detected, repeat the calibration using the standard additions method, starting with test solutions prepared as specified in 6.3.1. In this case, ensure that the operations are carried out in the linear part of the calibration graph.

7 EXPRESSION OF RESULTS

7.1 The calcium content, expressed as milligrams of Ca per kilogram, is given by the formula

$$m_1 \times \frac{1}{1\,000} \times \frac{A_1 - A_0}{A_2 - A_3} \times \frac{1\,000}{25} \times \frac{1\,000}{m_0} = \frac{40\, m_1\, (A_1 - A_0)}{m_0 \times (A_2 - A_3)}$$

where

A_0 is the absorbance of the blank test solution (6.3.2);

A_1 is the absorbance of the test solution (6.3.1);

A_2 is the mean of the measurements on the standard matching solution closest in absorbance to that of the test solution, carried out before and after the latter;

A_3 is the absorbance of the standard matching solution No. 0 (6.2.1);

m_0 is the mass, in grams, of the test sample used in the preparation of the main solution A;

m_1 is the mass, in micrograms, of Ca contained in the standard matching solution closest in absorbance to that of the test solution.

7.2 Calculate the magnesium content according to 7.1, using the relevant absorbances for magnesium.

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 in the International Standard to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO SODIUM HYDROXIDE FOR INDUSTRIAL USE

ISO 979 – Method of assay.

ISO 980 – Determination of carbonates content – Gas-volumetric method.

ISO 981 – Determination of chloride content – Mercurimetric method.

ISO 982 – Determination of sulphate content – Barium sulphate gravimetric method.

ISO 983 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 984 – Determination of silica content – Reduced silicomolybdic complex photometric method.

ISO 985 – Determination of silica content – Gravimetric method by precipitation of quinoline molybdosilicate.

ISO 986 – Determination of calcium – EDTA (*di*sodium salt) complexometric method.

ISO 3195 – Sampling – Test sample – Preparation of the main solution for carrying out certain determinations.

ISO 3196 – Determination of carbon dioxide content – Titrimetric method.

ISO 3197 – Determination of chloride content – Photometric method.

ISO 3198 – Determination of sulphur compounds – Method by reduction and titrimetry.

ISO 3697 – Determination of calcium and magnesium contents – Flame atomic absorption method.