BS 6075 : Part 9 : 1981
ISO 3697-1976
UDC 661.322.1 : 546.33—36 : 543.42.062 : [546.41+546.46]

British Standard Methods of

Sampling and test for sodium hydroxide for industrial use

Part 9. Determination of calcium and magnesium contents

[ISO title: Sodium hydroxide for industrial use — Determination of calcium and magnesium contents — Flame atomic absorption method]

Méthodes d'échantillonnage et d'essai du hydroxyde de sodium à usage industriel
Partie 9. Dosages du calcium et du magnésium

Probeentnahme- und Prüfmethoden für Natriumhydroxyd für industrielle Anwendung
Teil 9. Bestimmung der Kalzium- und Magnesiumgehalte

NOTE. It is recommended that this Part be read in conjunction with the information in the 'General introduction' published separately as BS 6075 : Part 0.

National foreword

This Part of BS 6075 is identical with ISO 3697 'Sodium hydroxide for industrial use — Determination of calcium and magnesium contents — Flame atomic absorption method', published in 1976 by the International Organization for Standardization (ISO).

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

The comma has been used throughout as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

Cross-reference

International standard	Corresponding British Standard
	BS 6075 Methods of sampling and test for sodium hydroxide for industrial use
ISO 3195-1975	Part 5 : 1981 Sampling and preparation of main test solution (Identical)

The standards listed in the annex are intended for information only. Corresponding British Standards are listed in BS 6075 : Part 0.

Additional information

Hydrochloric acid, ρ approximately 1.18 g/ml, about 36 % (*m/m*) solution, which is the corresponding reagent normally obtainable in the UK, is suitable for use in place of the solution specified in **4.1.**

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British Standards Institution

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.

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