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Crude sodium borates for industrial use — Determination of total and alkali-soluble calcium and magnesium contents — Flame atomic absorption spectrometric method

Borates de sodium bruts à usage industriel — Dosage du calcium et du magnésium total et du calcium et du magnésium solubles en milieu alcalin — Méthode par spectrométrie d'absorption atomique dans la flamme

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INTERNATIONAL STANDARD ISO 6918-1984 (E)

Crude sodium borates for industrial use — Determination of total and alkali-soluble calcium and magnesium contents — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the total and alkali-soluble calcium and magnesium contents of crude sodium borates for industrial use.

The method is applicable to products in which the calcium content, expressed as calcium, is not lower than 0,02 % (*m/m*) and not greater than 1,0 % (*m/m*) and in which the magnesium content, expressed as magnesium, is not lower than 0,01 % (*m/m*) and not greater than 0,2 % (*m/m*).

2 Reference

ISO 2217, *Crude sodium borates for industrial use — Determination of matter insoluble in alkaline medium and preparation of test solutions.*

3 Principle

Preparation of test solutions

a) by fusion of a test portion with sodium carbonate in the case of total calcium and magnesium contents, and

b) from an aliquot portion of "solution A" (see ISO 2217) for alkali-soluble calcium and magnesium contents.

Addition of lanthanum ions to suppress certain interferences and determination of the calcium and magnesium contents by flame atomic absorption spectrometry by aspirating the test solution into an acetylene/dinitrogen monoxide flame, using the calcium line at a wavelength of 422,7 nm and the magnesium line at a wavelength of 285,2 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Sodium carbonate, anhydrous.

4.2 Sodium chloride, 110 g/l solution.

4.3 Lanthanum chloride, solution corresponding to 100 g of lanthanum per litre.

Dissolve 268 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.

4.4 Hydrochloric acid, ϱ approximately 1,19 g/ml, approximately 37 % (*m/m*) solution, diluted 1 + 1 with water.

4.5 Calcium, standard solution corresponding to 1,00 g of Ca per litre.

Weigh, to the nearest 0,000 1 g, 1,249 g of calcium carbonate, previously dried at approximately 110 °C and cooled in a desiccator, into a 250 ml beaker. Cover with 50 ml of water and add 5,0 ml of the hydrochloric acid solution (4.4). When dissolution is complete, transfer the contents of the beaker quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1,00 mg of Ca.

4.6 Magnesium, standard solution corresponding to 1,00 g of Mg per litre.

Weigh, to the nearest 0,001 g, 1,00 g of magnesium (metal turnings used for the Grignard reaction are suitable) into a 250 ml beaker. Cover with 50 ml of water and add carefully 50 ml of hydrochloric acid solution (4.4). When dissolution is complete, transfer the contents of the beaker quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1,00 mg of Mg.

4.7 Combined calcium and magnesium, standard solution corresponding to 0,10 g of Ca per litre and 0,02 g of Mg per litre.

Transfer, by means of a pipette, 50,0 ml of the standard calcium solution (4.5) and 10,0 ml of the standard magnesium solution (4.6) into a 500 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 0,10 mg of Ca and 0,02 mg of Mg.

Prepare this solution on the day of use.

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5 Apparatus

Ordinary laboratory apparatus and

5.1 Platinum crucible, with lid.

5.2 Atomic absorption spectrometer, fitted with a suitable burner fed with dinitrogen monoxide and acetylene.

WARNING — Comply rigorously with the manufacturer's instructions for igniting and extinguishing the acetylene/dinitrogen monoxide flame.

5.3 Calcium hollow-cathode lamp.

5.4 Magnesium hollow-cathode lamp.

5.5 Test sieve, of nominal aperture size 250 µm, complying with ISO 565.

6 Procedure

6.1 Test portion

6.1.1 Total calcium and magnesium contents

After thorough mixing to ensure homogeneity, take a representative portion of the test sample and grind it so that it passes entirely through the test sieve (5.5). Weigh, to the nearest 0,001 g, approximately 0,1 g of the ground and sieved material into the platinum crucible (5.1).

6.1.2 Alkali-soluble calcium and magnesium contents

Prepare solution A as described in ISO 2217 and transfer, by means of a pipette, 5,0 ml of this solution into a 100 ml one-mark volumetric flask.

6.2 Blank test

Carry out a blank test at the same time as the determination, using the same quantities of all the reagents used in the determination but

a) omitting the test portion (6.1.1) for the determination of total calcium and magnesium contents;

b) using 5,0 ml of "solution B", prepared as described in ISO 2217, instead of the test portion (6.1.2) for the determination of alkali-soluble calcium and magnesium contents.

6.3 Preparation of the calibration graph

6.3.1 Preparation of the calibration solutions

Into a series of seven 100 ml one-mark volumetric flasks add from a burette the quantities of the standard combined calcium and magnesium solution (4.7) indicated in table 1.

Table 1 — Calibration solutions

Standard solution (4.7)	Corresponding mass of metal	
	Ca	Mg
ml	mg	mg
0[1)]	0,0	0,0
1,0	0,10	0,02
2,0	0,20	0,04
4,0	0,40	0,08
6,0	0,60	0,12
8,0	0,80	0,16
10,0	1,00	0,20

1) Reagent blank for the calibration graph.

Add to each flask 10,0 ml of the sodium chloride solution (4.2), 2,0 ml of the lanthanum chloride solution (4.3) and 5,0 ml of the hydrochloric acid solution (4.4). Dilute to the mark with water and mix.

6.3.2 Spectrometric measurements

Together with the spectrometric measurements on the appropriate test solution (6.4.1) and blank test solution (6.2), carry out measurements on the corresponding calibration solutions (6.3.1) using the procedure specified in 6.4.2.

6.3.3 Plotting the calibration graphs

Plot graphs having, for example, the masses, in milligrams, of Ca or Mg in 100 ml of the calibration solutions (6.3.1) as abscissae and the corresponding values of absorbance, less the value for the reagent blank for the calibration graph (6.3.1), as ordinates. The calibration graphs should be linear over most of the range.

6.4 Determination

6.4.1 Preparation of the test solutions

6.4.1.1 Total calcium and magnesium contents

Add 1 ± 0,1 g of the anhydrous sodium carbonate (4.1) to the test portion (6.1.1) in the platinum crucible (5.1) and mix thoroughly with a microspatula. Heat the mixture carefully until a clear melt is obtained and continue heating for 1 h, maintaining the temperature at just above the fusion point of the melt. Cool, place the crucible on its side in a 150 ml beaker and add 20 ml of water. Warm gently to loosen the melt and then add cautiously 9,0 ml of the hydrochloric acid solution (4.4). Continue to simmer gently until the melt has completely dissolved. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, add 2,0 ml of the lanthanum chloride solution (4.3), dilute to the mark with water and mix.

6.4.1.2 Alkali-soluble calcium and magnesium contents

Add 9,0 ml of the sodium chloride solution (4.2), 5,0 ml of the hydrochloric acid solution (4.4) and 2,0 ml of the lanthanum chloride solution (4.3) to the 100 ml one-mark volumetric flask containing the test portion (6.1.2). Dilute to the mark with water and mix.

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6.4.2 Spectrometric measurement

Mount the appropriate hollow-cathode lamp (5.3 or 5.4) in the spectrometer and switch on. When stabilization is reached, adjust to the wavelength of maximum absorption, i.e. approximately 422,7 nm for calcium or 285,2 nm for magnesium. Adjust the sensitivity and the slit aperture according to the characteristics of the apparatus. Adjust the gas pressures according to the characteristics of the burner so as to obtain an oxidizing flame.

Aspirate the appropriate calibration solutions (6.3.1), the test solution (6.4.1.1 or 6.4.1.2) and the appropriate blank test solution (6.2) in turn into the flame and measure the absorbance of each.

Ensure that the rate of aspiration into the flame remains constant throughout the measurements.

Aspirate water through the burner after each measurement.

6.4.3 Check test for interferences

Check for interferences by repeating, as appropriate, the operations specified in 6.4.1.1, or 6.4.1.2, with the addition of known quantities of the combined calcium and magnesium standard solution (4.7). If interferences are detected, repeat the calibration using the standard additions method, starting with the appropriate test solution (6.4.1.1 or 6.4.1.2). In this case, ensure that the operations are carried out in the linear part of the calibration graph.

7 Expression of results

Deduct the value of the absorbance of the appropriate blank test solution (6.2) from that of the corresponding test solution (6.4.1) and, by means of the appropriate calibration graph, determine the corrected masses of calcium or magnesium in the test solution.

The absorbance for the sample shall be on the linear part of the calibration graph. If necessary, the size of the test portion shall be adjusted and the measurement repeated.

Calculate the calcium and magnesium contents, total and alkali-soluble, expressed as percentages by mass using the formulae given in table 2.

Table 2 — Formulae for calcium and magnesium contents

	Calcium content [% (*m*/*m*)] expressed as		Magnesium content [% (*m*/*m*)] expressed as	
	Ca	CaO	Mg	MgO
Total content	$\frac{m_2}{10\ m_1}$	$\frac{m_2}{m_1} \times \frac{56{,}08}{400{,}8}$	$\frac{m_3}{10\ m_1}$	$\frac{m_3}{m_1} \times \frac{40{,}31}{243{,}1}$
Alkali-soluble content	$\frac{10\ m_2}{m_1}$	$\frac{m_2}{m_1} \times \frac{5\ 608}{400{,}8}$	$\frac{10\ m_3}{m_1}$	$\frac{m_3}{m_1} \times \frac{4\ 031}{243{,}1}$

In the formulae in table 2:

m_1 is, as appropriate,

— either the mass, in grams, of the test portion (6.1.1) for the determination of total calcium and magnesium contents,

— or the mass, in grams, of the test sample used for the preparation of "solution A" (see ISO 2217) from which is taken the test portion (6.1.2) for the determination of alkali-soluble calcium and magnesium contents;

m_2 is the mass, in milligrams, of Ca in the test solution;

m_3 is the mass, in milligrams, of Mg in the test solution.

8 Test report

The test report shall include the following particulars:

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

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

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Annex

ISO publications relating to crude sodium borates for industrial use

ISO 1918 — Determination of sulphur compounds — Volumetric method.

ISO 2216 — Determination of sodium oxide and boric oxide contents — Volumetric method.

ISO 2217 — Determination of matter insoluble in alkaline medium and preparation of test solutions.

ISO 2218 — Determination of loss in mass after heating at 900 °C.

ISO 2760 — Determination of total aluminium content — Titrimetric method.

ISO 2761 — Determination of total titanium content — Photometric method.

ISO 3120 — Determination of water content — Gravimetric method.

ISO 3122 — Determination of iron content — 2,2'-Bipyridyl photometric method.

ISO 3124 — Determination of iron soluble in alkaline medium — 2,2'-Bipirydyl photometric method.

ISO 3125 — Determination of aluminium soluble in alkaline medium — EDTA titrimetric method.

ISO 5933 — Determination of total nickel content of boric acid, boric oxide and *di*sodium tetraborates and the alkali-soluble nickel content of crude sodium borates — Furil α-dioxime photometric method.

ISO 5934 — Determination of alkali soluble copper and manganese contents — Zinc bis(dibenzyldithiocarbamate) and formaldehyde oxime photometric methods.

ISO 5935 — Determination of total and alkali-soluble silica contents — Molybdosilicate spectrometric method.

ISO 5936 — Determination of carbonate content — Gravimetric method.

ISO 6918 — Determination of total and alkali-soluble calcium and magnesium contents — Flame atomic absorption spectrometric method.

ISO 6920 — Determination of total and alkali-soluble calcium and magnesium contents — Titrimetric method.

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