
International Standard



6920

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Crude sodium borates for industrial use — Determination of total and alkali-soluble calcium and magnesium contents — Titrimetric 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 titrimétrique

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Foreword

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Crude sodium borates for industrial use — Determination of total and alkali-soluble calcium and magnesium contents — Titrimetric method

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the total and alkali-soluble calcium and magnesium contents of crude sodium borates for industrial use. Modified procedures are described for special cases for the determination of total calcium content to eliminate interference by silica, iron or aluminium.

The method is applicable to products in which the calcium content, expressed as calcium, is not lower than 0,01 % (*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 References

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

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

3 Principle

Preparation of test solutions

- by fusion of a test portion with sodium carbonate in the case of total calcium and magnesium contents, and
- from an aliquot portion of solution A (see ISO 2217) for alkali-soluble calcium and magnesium contents.

Determination of the calcium content by titration of an aliquot portion of the test solution with ethyleneglycol-bis-(2-aminoethyl)-*N, N, N', N'* tetra-acetic acid, *d*/sodium salt (EGTA) solution using 2-carboxy-2'-hydroxy-5' sulphoformazyl-benzene (Zincon) as indicator.

Determination of the combined magnesium and calcium contents by titration of a further aliquot portion of the test solution with ethylenediaminetetra-acetic acid, *d*/sodium salt (EDTA) solution, using Mordant black 11/potassium chloride as indicator, after addition of triethanolamine solution and buffer solution.

Calculation of the magnesium content by difference.

4 Reagents

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

- Sodium carbonate**, anhydrous.
- Hydrochloric acid**, approximately ρ 1,19 g/ml, about 37 % (*m/m*) solution, diluted 1 → 2 with water.
- Triethanolamine**, 300 g/l solution.
- Sodium hydroxide**, approximately 400 g/l solution.
- Sodium hydroxide**, approximately 80 g/l solution.
- Magnesium chloride hexahydrate** ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), approximately 20 g/l solution.
- Buffer solution**, pH 10.

Dissolve 67,5 g of ammonium chloride in 250 ml of water, add 570 ml of ammonia solution (ρ approximately 0,88 g/ml) and dilute with water to 1 litre.

- Zinc chloride**, standard reference solution corresponding to 1 g of zinc oxide per litre.

Calcine zinc oxide in a porcelain crucible for 2 h in a muffle furnace maintained at 550 ± 25 °C and then cool in a desiccator. Weigh, to the nearest 0,001 g, 1,0 g of the dried zinc oxide and dissolve in a mixture of 30 ml of water and 40 ml of the hydrochloric acid (4.2). Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

- EDTA**, standard volumetric solution, $c(\text{EDTA}) = 0,01$ mol/l.

4.9.1 Preparation of the solution

Dissolve 3,725 g of ethylenediamine tetra-acetic acid, *d*/sodium salt (EDTA), in a little water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1,00 ml of solution, $c(\text{EDTA}) = 0,010$ mol/l, corresponds to 0,403 mg of MgO or 0,243 mg of Mg.