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Crude sodium borates for industrial use — Determination of total aluminium content — Titrimetric method

Borates de sodium bruts à usage industriel — Dosage de l'aluminium total — Méthode titrimétrique

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

Austria	Israel	Spain
Belgium	Italy	Sweden
Czechoslovakia	Netherlands	Switzerland
Egypt, Arab Rep. of	New Zealand	Thailand
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Hungary	Romania	U.S.S.R.
India	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

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Crude sodium borates for industrial use — Determination of total aluminium content — Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a titrimetric method for the determination of the total aluminium content of crude sodium borates for industrial use, for material containing up to 1 % aluminium.

The method, as described, is not applicable in the presence of zirconium or titanium. More than 0,006 % of iron will interfere in the determination.

2 PRINCIPLE

Fusion of a test portion with sodium carbonate and extraction of the molten mass with hydrochloric acid. Formation of a complex by the addition of the *di*sodium salt of ethylenediaminetetra-acetic acid (EDTA). Decomposition of the complex by sodium fluoride, followed by titration of the liberated EDTA with a standard volumetric solution of zinc chloride in the presence of xylenol orange as indicator.

3 REAGENTS

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

3.1 Sodium carbonate, anhydrous.

3.2 Sodium fluoride, crystalline.

3.3 Hydrochloric acid, approximately 6 N solution.

3.4 Ammonia, 1 to 2 N solution.

3.5 Ammonium acetate buffer solution, pH 5,5.

Dissolve 50 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in water, add 2,5 ml of glacial acetic acid (approximately 17 N) and dilute to 1 000 ml.

3.6 Sodium ethylenediamine-*N,N,N',N'*-tetra-acetate (EDTA), 0,01 M solution.

Weigh, to the nearest 0,001 g, 3,725 g of EDTA, dissolve it in water, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

3.7 Zinc, 1 M standard volumetric solution.

Remove the oxide film from a sample of pure zinc weighing not less than about 70 g, by washing it with an approximately 2 M solution of hydrochloric acid. Rinse the cleaned zinc with methanol and dry it by washing with diethyl ether. After the ether has evaporated, immediately weigh, to the nearest 0,01 g, 65,37 g of this zinc and dissolve it in 600 ml of the hydrochloric acid solution (3.3). Cool the solution and transfer it quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

NOTE — Solution of the zinc is facilitated by the introduction of a small piece of platinum wire.

1 ml of this standard volumetric solution corresponds to 51 mg of Al_2O_3.

3.8 Zinc, 0,01 M standard volumetric solution.

Transfer 10,0 ml of the standard volumetric zinc solution (3.7) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard volumetric solution corresponds to 0,51 mg of Al_2O_3.

3.9 Methyl orange, 0,5 g/l solution.

3.10 Xylenol orange, 1 g/l solution, filtered if necessary.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Platinum crucible, with platinum lid.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the test sample, into the platinum crucible (4.1).

5.2 Determination

Add 2 g of the sodium carbonate (3.1) to the platinum crucible (4.1) containing the test portion (5.1), mix, cover the crucible with its lid, heat the contents cautiously and maintain just at the point of fusion until a clear mix is

obtained. Cool and transfer the melt, using 50 ml of hot water, to a suitable vessel. After digestion, add 1 drop of the methyl orange solution (3.9) and add the hydrochloric acid solution (3.3) until the colour just changes from orange-yellow to red; then add 1 drop of this acid in excess. Boil gently to remove the carbon dioxide. Cool, add 20 ml of the EDTA solution (3.6), 1 more drop of the methyl orange solution (3.9) and the ammonia solution (3.4) drop by drop until the colour changes from red to orange-yellow.

Add immediately 10 ml of the ammonium acetate buffer solution (3.5). Heat the solution to boiling and allow to boil for 5 min, then cool the solution to ambient temperature with the aid of a cold water bath. Add 4 drops of the xylenol orange solution (3.10) and titrate the excess EDTA with the standard volumetric zinc solution (3.8) until the colour changes from yellow to brownish-yellow. Add 1 g of the sodium fluoride (3.2), heat the solution and allow it to boil for 5 min, then cool it to ambient temperature. Titrate the liberated EDTA with the standard volumetric zinc solution (3.8) until the same colour change (from yellow to brownish-yellow) takes place. Note only the volume (V) of the zinc solution used in this last titration.

6 EXPRESSION OF RESULTS

The aluminium content, expressed as aluminium oxide (Al_2O_3), is given, as a percentage by mass, by the formula

$$V \times \frac{0{,}51}{1\,000} \times \frac{100}{m} = \frac{0{,}051 \times V}{m}$$

where

V is the volume, in millilitres, of the standard volumetric zinc solution (3.8) used for the titration;

m is the mass, in grams, of the test portion.

7 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 regarded as optional.