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Crude sodium borates for industrial use — Determination of aluminium soluble in alkaline medium — EDTA titrimetric method

Borates de sodium bruts à usage industriel — Dosage de l'aluminium soluble en milieu alcalin — Méthode titrimétrique à l'EDTA

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3125 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in April 1973.

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

Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Bulgaria	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
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Crude sodium borates for industrial use — Determination of aluminium soluble in alkaline medium — EDTA titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an EDTA titrimetric method for the determination of aluminium soluble in alkaline medium in crude sodium borates for industrial use.

The method is applicable to products in which the content of aluminium soluble in alkaline medium, expressed as aluminium oxide (Al_2O_3), exceeds 100 mg/kg.

2 REFERENCE

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

3 PRINCIPLE

Formation of the complex between aluminium and EDTA in an aliquot part of solution A (see ISO 2217) prepared for the analysis of the soluble impurities in alkaline solution. Decomposition of the complex using sodium fluoride. Titration of the amount of liberated EDTA against a zinc chloride solution of known concentration in the presence of xylenol orange as indicator.

4 REAGENTS

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

4.1 Sodium fluoride, crystallized.

4.2 Ammonia solution, ρ approximately 0,983 g/ml, about 35 g/l or approximately 2 N.

4.3 Buffer solution, pH 5,5.

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

4.4 EDTA (*di*sodium salt), 0,01 M solution.

Dissolve 3,725 g, weighed to the nearest 0,001 g, of (ethylenedinitrilo)tetraacetic acid, *di*sodium salt, dihydrate, (EDTA *di*sodium salt) in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

4.5 Zinc chloride, 1 M standard volumetric solution.

Dissolve 65,37 g, weighed to the nearest 0,01 g, of granulated zinc in 600 ml of 6 N hydrochloric acid solution. Dissolution can be promoted by the addition of a small piece of platinum wire. Cool to ambient temperature, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution corresponds to 51 mg of aluminium oxide (Al_2O_3).

4.6 Zinc chloride, 0,01 M standard volumetric solution.

Place 10,0 ml of the standard volumetric zinc chloride solution (4.5) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution corresponds to 0,51 mg of aluminium oxide (Al_2O_3).

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

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

5 APPARATUS

Ordinary laboratory apparatus.

6 PROCEDURE

6.1 Test portion

Transfer 50,0 ml of solution A (see ISO 2217) to a 500 ml conical flask.

6.2 Blank test

Transfer 50,0 ml of solution B (see ISO 2217) to a 500 ml conical flask and proceed as specified in 6.3.

6.3 Determination

Add 20 ml of the EDTA (*di*sodium salt) solution (4.4) and 1 drop of the methyl orange solution (4.7) to the test portion (6.1) and neutralize with the ammonia solution (4.2) until the colour of the solution changes from red to orange-yellow. Immediately add 10 ml of the buffer solution (4.3), boil for 5 min and cool to ambient temperature in a cold water bath.

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Add 4 drops of the xylenol orange solution (4.8) and add the standard volumetric zinc chloride solution (4.6), drop by drop, until the colour changes from yellow to yellowish-brown.

Add 1 g of the sodium fluoride (4.1), boil for 5 min and cool to ambient temperature. Titrate with the standard volumetric zinc chloride solution (4.6) until the colour just changes from yellow to yellowish-brown.

7 EXPRESSION OF RESULTS

The content of aluminium soluble in alkaline medium, expressed in milligrams of aluminium oxide (Al_2O_3) per kilogram, is given by the formula

$$(V_1 - V_2) \times 0{,}51 \times \frac{500}{50} \times \frac{1\,000}{m_0} = 5\,100 \times \frac{V_1 - V_2}{m_0}$$

where

V_1 is the volume, in millilitres, of the standard volumetric zinc chloride solution (4.6) used for the titration of the test portion, after the addition of the sodium fluoride (4.1);

V_2 is the volume, in millilitres, of the standard volumetric zinc chloride solution (4.6) used for the titration in the blank test, after the addition of the sodium fluoride (4.1);

m_0 is the mass, in grams, of the test portion used for preparing solution A.

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.

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ANNEX

ISO PUBLICATIONS RELATING TO (A) BORIC ACID, (B) BORIC OXIDE, (C) *D*/SODIUM TETRABORATES, (D) SODIUM PERBORATES, AND (E) CRUDE SODIUM BORATES, FOR INDUSTRIAL USE

Applicability	
A	ISO 1914 – Determination of boric acid content – Volumetric method.
B	ISO 1915 – Determination of boric oxide content – Volumetric method.
C	ISO 1916 – Determination of sodium oxide and boric oxide contents and loss on ignition.
D	ISO 1917 – Determination of sodium oxide, boric oxide and available oxygen contents – Volumetric methods.
A B C E	ISO 1918 – Determination of sulphur compounds – Volumetric method.
A B C	ISO 2214 – Determination of manganese content – Formaldehyde oxime photometric method.
A B C	ISO 2215 – Determination of copper content – Zinc dibenzyldithiocarbamate photometric method.
E	ISO 2216 – Determination of sodium oxide and boric oxide contents – Volumetric method.
E	ISO 2217 – Determination of matter insoluble in alkaline medium and preparation of test solutions.
E	ISO 2218 – Determination of loss in mass after heating at 900 °C.
E	ISO 2760 – Determination of total aluminium content – Titrimetric method.
E	ISO 2761 – Determination of total titanium content – Photometric method.
D	ISO 3118 – Determination of particle size distribution by mechanical sieving.
A B C	ISO 3119 – Determination of chromium content – Diphenylcarbazide photometric method.
C E	ISO 3120 – Determination of water content – Gravimetric method.
A B C	ISO 3121 – Determination of chloride content – Mercurimetric method.
A B C D E	ISO 3122 – Determination of iron content – 2,2′-Bipyridyl photometric method.
D	ISO 3123 – Determination of rate of solution – Conductivity method.
E	ISO 3124 – Determination of iron soluble in alkaline medium – 2,2′-Bipyridyl photometric method.
E	ISO 3125 – Determination of aluminium soluble in alkaline medium – EDTA titrimetric method.
D	ISO 3424 – Determination of bulk density.

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