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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Boric acid, boric oxide, *di*sodium tetraborates and crude sodium borates for industrial use — 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

ERRATUM

Page 1

4 Reagents

Delete "**4.3 Ammonia solution**", and substitute "**4.3 Ammonium hydroxide**".

International Standard 5933

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Boric acid, boric oxide, *di*sodium tetraborates and crude sodium borates for industrial use — 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

Acide borique, oxyde borique, tétraborates disodiques et borates de sodium bruts à usage industriel — Dosage du nickel total dans l'acide borique, l'oxyde borique et les tétraborates disodiques, et du nickel soluble en milieu alcalin dans les borates de sodium bruts — Méthode photométrique à la furil α-dioxime

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ISO 5933-1980 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 5933 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in October 1978.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Poland
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Israel	Switzerland
Bulgaria	Italy	Thailand
China	Korea, Rep. of	United Kingdom
Czechoslovakia	Mexico	USSR
Egypt, Arab Rep. of	Netherlands	Yugoslavia
France	Philippines	

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Boric acid, boric oxide, *di*sodium tetraborates and crude sodium borates for industrial use — 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

1 Scope and field of application

This International Standard specifies a furil α-dioxime photometric method for the determination of the total nickel content of boric acid, boric oxide and *di*sodium tetraborates for industrial use and for determination of the alkali-soluble nickel content of crude sodium borates for industrial use.

The method is applicable to products in which the nickel content being determined exceeds 0,1 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

Dissolution of a test portion, formation of the coloured nickel furil α-dioxime complex, extraction with chloroform and photometric measurement at a wavelength of about 435 nm.

4 Reagents

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

4.1 Chloroform, redistilled.

WARNING — Chloroform is toxic. Avoid breathing the vapour and contact with skin. Carry out all operations involving its use in a fume cupboard.

4.2 Hydrochloric acid, ϱ approximately 1,19 g/ml, about 38 % (m/m) solution.

4.3 Ammonia solution, ϱ approximately 0,91 g/ml, about 25 % (m/m) solution.

4.4 Sulphuric acid, approximately 4,9 g/l solution.

4.5 Sodium hydroxide, approximately 40 g/l solution.

4.6 Hydrogen peroxide, 50 g/l solution.

4.7 *Tri*sodium citrate dihydrate ($C_6H_5Na_3O_7.2H_2O$), 100 g/l solution.

4.8 2,2'-Furil (Z, E)-dioxime (Furil α-dioxime), 10 g/l ethanolic solution.

Dissolve 1 g of furil α-dioxime in 60 to 70 ml of ethanol at approximately 70 °C. Cool to ambient temperature and dilute to 100 ml with the same ethanol.

4.9 Nickel, standard solution corresponding to 0,100 g of nickel (Ni) per litre.

Weigh to the nearest 0,000 1 g, 0,100 g of pure nickel and dissolve in a minimum quantity of approximately 500 g/l nitric acid solution. Heat the solution on a hot plate until the fumes evolved are no longer brown, cool and add about 100 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 0,10 mg of Ni.

4.10 Nickel, standard solution corresponding to 0,002 g of nickel (Ni) per litre.

Transfer 5,0 ml of the standard nickel solution (4.9) to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

1 ml of this standard solution contains 2 µg of Ni.

4.11 Phenolphthalein, 5 g/l solution in 95 % (V/V) ethanol.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Spectrophotometer, fitted with cells having optical path lengths of 4 or 5 cm and of capacity 10 to 12 ml, or

5.2 Photoelectric absorptiometer, fitted with similar cells.

6 Procedure

6.1 Test portion

6.1.1 Boric acid, boric oxide and *di*sodium tetraborates

Weigh, to the nearest 0,01 g, 2,0 g of the test sample.

6.1.2 Crude sodium borates

Transfer 100,0 ml of Solution A (see ISO 2217) to a 250 ml beaker.

6.2 Blank tests

6.2.1 Boric acid, boric oxide and *di*sodium tetraborates

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

6.2.2 Crude sodium borates

Transfer 100,0 ml of Solution B (see ISO 2217) to a 250 ml beaker and carry out a blank test, at the same time as the determination, using the same procedure and the same quantities of all the reagents used in the determination, but omitting the test portion.

6.3 Preparation of the calibration graph

6.3.1 Preparation of standard colorimetric solutions

Into a series of five 100 ml separating funnels, each containing 1 ml of the hydrochloric acid solution (4.2), place the quantities of the standard nickel solution (4.10) indicated in the following table.

Standard nickel solution (4.10)	Corresponding mass of nickel
ml	µg
0*	0
0,5	1,0
1,0	2,0
1,5	3,0
2,0	4,0

* Compensation solution

Dilute each solution to 50 ml with water and add 0,5 ml of the hydrogen peroxide solution (4.6).

6.3.2 Colour development

Taking each separating funnel in turn, add to its contents 5 ml of the *tri*sodium citrate solution (4.7) and 0,1 ml of the phenolphthalein solution (4.11). Cautiously add the sodium hydroxide solution (4.5), with continuous mixing, until the indicator colour turns pink and then add the sulphuric acid solution (4.4), drop by drop, until the pink colour is discharged.

Add, mixing after each addition, 1 ml of the furil α-dioxime solution (4.8), 5 ml of the ammonia solution (4.3) and 15 ml of the chloroform (4.1). Shake the funnel vigorously for 2 min, releasing any pressure build-up, and allow the layers to separate for 3 min. Transfer the chloroform layer to a second 100 ml separating funnel containing 50 ml of the sulphuric acid solution (4.4). Shake the funnel vigorously for 2 min and allow the layers to separate for 3 min. Fill one of the cells with the chloroform layer by filtering it through a dry acid-washed grade filter paper.

6.3.3 Photometric measurement

Using the spectrophotometer (5.1) at a wavelength of approximately 435 nm, or the photoelectric absorptiometer (5.2) fitted with a suitable filter, carry out the photometric measurements after having adjusted the instrument to zero absorbance against the chloroform (4.1).

6.3.4 Plotting the graph

Deduct the absorbance of the compensation solution from those of the standard colorimetric solutions (see 6.3.1). Plot a graph having, for example, the masses, in micrograms, of nickel in the standard colorimetric solutions as abscissae and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 Preparation of test solution

6.4.1.1 Boric acid, boric oxide and *di*sodium tetraborates

Transfer the test portion (6.1.1) quantitatively to a 150 ml beaker, add 20 ml of water and place on a boiling water bath. After 5 min, add 20 ml of the hydrochloric acid solution (4.2) and evaporate just to dryness. Add 1 ml of the hydrochloric acid solution (4.2) and 30 ml of water to the residue. Stir to dissolve the contents of the beaker and transfer the solution quantitatively to a 100 ml separating funnel. Dilute to 50 ml with water and add 0,5 ml of the hydrogen peroxide solution (4.6).

6.4.1.2 Crude sodium borates

Place the beaker containing the test portion (6.1.2) on a hot plate and evaporate down to a volume of 45 to 50 ml. Cool, transfer the solution quantitatively to a 100 ml separating funnel and add 0,5 ml of the hydrogen peroxide solution (4.6).

6.4.2 Colour development

Develop the colour following the procedure specified in 6.3.2.

6.4.3 Photometric measurements

Carry out the photometric measurements on the test solution and on the blank test solution following the procedure specified in 6.3.3.

7 Expression of results

By reference to the calibration graph (6.3.4), determine the masses of nickel corresponding to the absorbances of the test solution and of the blank test solution.

7.1 Boric acid, boric oxide and *di*sodium tetraborates

The total nickel content, expressed in milligrams of Ni per kilogram, is given by the formula

$$\frac{m_1 - m_2}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (6.1.1);

m_1 is the mass, in micrograms, of nickel found in the test solution (6.4.1.1);

m_2 is the mass, in micrograms, of nickel found in the blank test solution (6.2.1).

7.2 Crude sodium borates

The alkali-soluble nickel content, expressed in milligrams of Ni per kilogram, is given by the formula

$$(m_3 - m_4) \times \frac{5}{m_5}$$

where

m_3 is the mass, in micrograms, of nickel found in the test solution (6.4.1.2);

m_4 is the mass, in micrograms, of nickel found in the blank test solution (6.2.2);

m_5 is the mass, in grams, of the test portion used for preparing 500 ml of Solution A (see ISO 2217).

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 usual features noted during the determination;

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

Annex

ISO publications relating to (A) boric acid, (B) boric oxide, (C) *di*sodium tetraborates, and (D) 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.
A B C D	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.
D	ISO 2216 — Determination of sodium oxide and boric oxide contents — Volumetric method.
D	ISO 2217 — Determination of matter insoluble in alkaline medium and preparation of test solutions.
D	ISO 2218 — Determination of loss in mass after heating at 900 °C.
D	ISO 2760 — Determination of total aluminium content — Titrimetric method.
D	ISO 2761 — Determination of total titanium content — Photometric method.
A B C	ISO 3119 — Determination of chromium content — Diphenylcarbazide photometric method.
D	ISO 3120 — Determination of water content — Gravimetric method.
A B C	ISO 3121 — Determination of chloride content — Mercurimetric method.
A B C D	ISO 3122 — Determination of iron content — 2,2'-Bipyridyl photometric method.
D	ISO 3124 — Determination of iron soluble in alkaline medium — 2,2'-Bipyridyl photometric method.
D	ISO 3125 — Determination of aluminium soluble in alkaline medium — EDTA titrimetric method.
A B C	ISO 5932 — Determination of cobalt content — 2-nitroso-1-naphthol photometric method.
A B C D	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.
D	ISO 5934 — Determination of alkali soluble copper and manganese contents — Zinc bis(dibenzyldithiocarbamate) and formaldehyde oxime photometric methods.
D	ISO 5935 — Determination of total silica content and content of silica soluble in alkaline medium — Photometric method.
D	ISO 5936 — Determination of carbonate content — Gravimetric method.