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Boric acid, boric oxide and *di*sodium tetraborates for industrial use — Determination of cobalt content — 2-nitroso-1-naphthol photometric method

Acide borique, oxyde borique et tétraborates disodiques à usage industriel — Dosage du cobalt — Méthode photométrique au nitroso-2 naphtol-1

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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 5932 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
Austria
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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).

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Boric acid, boric oxide and *di*sodium tetraborates for industrial use — Determination of cobalt content — 2-nitroso-1-naphthol photometric method

1 Scope and field of application

This International Standard specifies a 2-nitroso-1-naphthol photometric method for the determination of the cobalt content of boric acid, boric oxide and *di*sodium tetraborates for industrial use.

The method is applicable to products having cobalt contents equal to or greater than 0,1 mg/kg.

2 Principle

Dissolution of a test portion, formation of the coloured cobalt 2-nitroso-1-naphthol complex, extraction with chloroform and photometric measurement at a wavelength of about 530 nm.

3 Reagents

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

3.1 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.

3.2 Hydrochloric acid, approximately 73 g/l solution.

3.3 Sodium hydroxide, approximately 80 g/l solution.

3.4 Hydrogen peroxide, 30 g/l solution.

3.5 *Tri*sodium citrate dihydrate ($C_6H_5Na_3O_7.2H_2O$),200 g/l solution.

3.6 2-nitroso-1-naphthol, 10 g/l solution in glacial acetic acid.

Dissolve 1 g of 2-nitroso-1-naphthol in glacial acetic acid, transfer to a 100 ml one-mark volumetric flask, add 1 g of activated carbon, dilute to the mark with the glacial acetic acid and mix.

Shake the solution before use and filter off the required amount.

Store the solution in a refrigerator, but discard after 2 to 3 weeks as there is a tendency for colour to develop in the extracted chloroform solution even in the absence of cobalt.

3.7 Cobalt, standard solution corresponding to 0,200 g of cobalt (Co) per litre.

Weigh, to the nearest 0,000 1 g, 0,200 g of pure cobalt and dissolve in the 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 0,20 mg of Co.

NOTE — The standard cobalt solution may also be prepared by dissolving a cobalt salt in approximatively 3,6 g/l hydrochloric acid solution. If necessary, determine the exact cobalt concentration of this solution by, for example, titration with EDTA solution.

3.8 Cobalt, standard solution corresponding to 0,002 g of cobalt (Co) per litre.

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

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

Prepare this solution at the time of use.

4 Apparatus

Ordinary laboratory apparatus and

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

4.2 Photoelectric absorptiometer, fitted with similar cells.

4.3 pH-meter, fitted with a glass measurement electrode and a calomel reference electrode.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0,01 g, a mass of test portion not exceeding that indicated in table 1, and containing not more than 4 µg of cobalt.

Table 1

Product	Mass of test portion g
Boric acid (H_3BO_3)	3
Boric oxide (B_2O_3)	1,5
*Di*sodium tetraborate ($Na_2B_4O_7$)	2
*Di*sodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$)	4

5.2 Blank test

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.

5.3 Preparation of the calibration graph

5.3.1 Preparation of standard colorimetric solutions

Transfer the quantities of the standard cobalt solution (3.8) indicated in table 2 into a series of five 150 ml beakers. Add 10 ml of the hydrochloric acid solution (3.2), dilute to 30 ml with water and add 10 ml of the *tri*sodium citrate solution (3.5).

Table 2

Standard cobalt solution (3.8)	Corresponding mass of cobalt
ml	µg
0*	0
0,5	1,0
1,0	2,0
1,5	3,0
2,0	4,0

* Compensation solution.

5.3.2 Colour development

Cautiously add sufficient of the sodium hydroxide solution (3.3) to adjust the pH, as measured by the pH meter (4.3), to 3,5. Then add 10 ml of the hydrogen peroxide solution (3.4) and mix. Allow to stand for 2 min.

Add 2 ml of the 2-nitroso-1-naphthol solution (3.6), mix, and allow to stand for 30 min at room temperature. Transfer quantitatively to a 100 ml separating funnel and add 10 ml of the chloroform (3.1). Shake the funnel vigorously for 2 min, releasing any pressure build-up and allow the layers to separate for 5 min.

Transfer the chloroform layer to a 50 ml separating funnel. Add 10 ml of the hydrochloric acid solution (3.2) and shake for 1 min. Allow the layers to separate for 5 min.

Transfer the chloroform layer to another 50 ml separating funnel. Add 10 ml of the sodium hydroxide solution (3.3) and shake for 1 min. Allow the layers to separate for 10 min. Run the chloroform layer through a dry filter paper into a 10 ml one-mark volumetric flask and dilute to the mark with more of the chloroform (3.1) which has been passed through the filter.

5.3.3 Photometric measurements

Using the spectrophotometer (4.1) at a wavelength of about 530 nm or the photoelectric absorptiometer (4.2) fitted with a suitable filter, carry out the photometric measurements on the chloroform extracts, after having adjusted the instrument to zero absorbance against the chloroform (3.1).

5.3.4 Plotting the graph

Deduct the absorbance of the compensation solution from those of the standard colorimetric solutions (see 5.3.1). Plot a graph having, for example, the masses, in micrograms, of cobalt in the standard colorimetric solutions as abscissae and the corresponding values of absorbance as ordinates.

5.4 Determination

5.4.1 Preparation of test solution

5.4.1.1 Boric acid and boric oxide

Transfer the test portion (5.1) quantitatively to a 150 ml beaker and add 10 ml of the hydrochloric acid solution (3.2) and 20 ml of water. Dissolve by heating, but without boiling. Add 10 ml of the *tri*sodium citrate solution (3.5) and cool the solution to ambient temperature.

5.4.1.2 *Di*sodium tetraborates

Transfer the test portion (5.1) quantitatively to a 150 ml beaker and add 20 ml of the hydrochloric acid solution (3.2) and 10 ml of water. Dissolve by heating but without boiling. Add 10 ml of the *tri*sodium citrate solution (3.5) and cool the solution to ambient temperature.

5.4.2 Colour development

Develop the colour following the procedure specified in 5.3.2.

5.4.3 Photometric measurement

Carry out the photometric measurement on the test solution (5.4.1) and on the blank test solution (5.2) following the procedure specified in 5.3.3.

6 Expression of results

By reference to the calibration graph (5.3.4), determine the masses of cobalt corresponding to the absorbances of the test solution and of the blank test solution.

The cobalt content, expressed in milligrams of Co 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 (5.1);

m_1 is the mass, in micrograms, of cobalt found in the test solution (5.4.1);

m_2 is the mass, in micrograms, of cobalt found in the blank test solution (5.2).

7 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 regarded as optional.

Annex

ISO publications relating to (A) boric acid, (B) boric oxide, and (C) *di*sodium tetraborates, 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	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.
A B C	ISO 3119 — Determination of chromium content — Diphenylcarbazide photometric method.
A B C	ISO 3121 — Determination of chloride content — Mercurimetric method.
A B C	ISO 3122 — Determination of iron content — 2,2′-Bipyridyl photometric method.
A B C	ISO 5932 — Determination of cobalt content — 2-nitroso-1-naphthol photometric method.
A B C	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.