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Boric acid, boric oxide and *di*sodium tetraborates for industrial use — Determination of chromium content — Diphenylcarbazide photometric method

Acide borique, oxyde borique et tétraborates disodiques à usage industriel — Dosage du chrome — Méthode photométrique à la diphénylcarbazide

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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 3119 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 :

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Bulgaria	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	United Kingdom
Germany	Poland	U.S.S.R.

No Member Body expressed disapproval of the document.

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Boric acid, boric oxide and *di*sodium tetraborates for industrial use — Determination of chromium content — Diphenylcarbazide photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a diphenylcarbazide photometric method for the determination of the chromium content of boric acid, boric oxide and *di*sodium tetraborates for industrial use.

2 PRINCIPLE

Fusion of a test portion with sodium carbonate followed by neutralization with sulphuric acid. Measurement of the absorbance, at a wavelength of about 540 nm, of the complex formed by chromium with diphenylcarbazide, sodium azide being added to destroy any colour due to manganese present in the sample.

3 REAGENTS

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

3.1 Ammonium persulphate.

3.2 Sodium carbonate, anhydrous.

3.3 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, diluted 1 + 1 by volume.

3.4 Phosphoric acid, ρ approximately 1,70 g/ml, about 85 % (*m/m*) solution, diluted 1 + 1 by volume.

3.5 Diphenylcarbazide, 2 g/l solution.

Dissolve 0,2 g of diphenylcarbazide — melting point in the range 175 ± 3 °C and molar absorptivity for the chromium complex (CrO_4) of about 42 000 — in 10 ml of glacial acetic acid (ρ 1,05 g/ml), approximately 17,4 N solution, and dilute with water to 100 ml.

Prepare this solution immediately before use.

3.6 Silver nitrate, 25 g/l solution.

3.7 Sodium azide, 50 g/l solution.

3.8 Chromium, standard solution, corresponding to 0,10 g of Cr per litre.

Dissolve 0,282 9 g of potassium dichromate, previously dried at 105 °C and cooled in a desiccator, in water, add 0,2 g of the sodium carbonate (3.2) and dilute to the mark in a 1 000 ml one-mark volumetric flask.

1 ml of this standard solution contains 100 μg of Cr.

3.9 Chromium, standard solution, corresponding to 0,001 0 g of Cr per litre.

Dilute 10,0 ml of the standard chromium solution (3.8) to the mark in a 1 000 ml one-mark volumetric flask.

1 ml of this standard solution contains 1 μg of Cr.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Platinum dish, about 100 ml capacity, with platinum lid.

4.2 Spectrophotometer, fitted with cells of 4 cm optical path length, or

4.3 Photoelectric absorptiometer, fitted with the same cells and with filters allowing a maximum transmission at about 540 nm.

4.4 pH meter.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,01 g, approximately 2 g of the test sample into the platinum dish (4.1).

5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used for the determination.

5.3 Preparation of calibration graph.

5.3.1 *Preparation of standard colorimetric solutions,* for photometric measurements with cells of 4 cm optical path length.

Into a series of six 100 ml one-mark volumetric flasks, transfer the volumes of the standard chromium solution (3.9) indicated in the following table and dilute each to about 50 ml with water.

Standard chromium solution (3.9)	Corresponding mass of chromium
ml	µg
0*	0
1,0	1
5,0	5
10,0	10
15,0	15
20,0	20

* Blank test on the reagents used for the preparation of the calibration graph.

To each flask, add 1 ml of the sulphuric acid solution (3.3), 1 ml of the phosphoric acid solution (3.4) and 2 ml of the diphenylcarbazide solution (3.5). Dilute to the mark, mix and allow to stand for 2 min.

5.3.2 *Photometric measurements*

Using the spectrophotometer (4.2), at a wavelength of about 540 nm, or the photoelectric absorptiometer (4.3), fitted with suitable filters, measure the absorbance of each solution, after having adjusted the instrument to zero absorbance against water. Deduct the absorbance of the blank test on the reagents used for the preparation of the calibration graph from those of the standard colorimetric solutions.

5.3.3 *Plotting of calibration graph*

Plot a graph having, for example, the chromium (Cr) contents in micrograms per 100 ml of standard colorimetric solution (5.3.1) as abscissae and the corresponding values of absorbance as ordinates.

5.4 Determination

5.4.1 *Preparation of the test solution*

Add 1 g of sodium carbonate (3.2) to the platinum dish (4.1) containing the test portion (5.1), mix, cover the dish with its lid, heat the contents carefully and maintain just at the fusion point until a clear melt is obtained. Cool and digest the fused melt well with 50 ml of hot water. Transfer the solution quantitatively to a beaker of convenient capacity.

Adjust the pH to between 6 and 7 by the addition of the sulphuric acid solution (3.3), using the pH meter (4.4).

Boil gently to remove carbon dioxide.

Cool the solution and add a further 1 ml of the sulphuric acid solution (3.3) and 1 ml of the phosphoric acid solution (3.4). Add 1 ml of the silver nitrate solution (3.6), followed by 1 g of the ammonium persulphate (3.1). Bring the solution slowly to the boil and boil gently until all the ammonium persulphate is decomposed and evolution of oxygen ceases.

Filter on a sintered glass crucible, porosity grade P16 (pore size index 10 to 16 µm), if a precipitate of silver chloride appears at this stage.

Transfer the beaker and its contents to a water-bath, and maintain at 70 ± 5 °C. If colour due to the presence of permanganate (from manganese present originally in the test portion) is noted at this stage, add the sodium azide solution (3.7), drop by drop, while gently swirling the beaker on the water-bath, until any colour is destroyed.

5.4.2 *Colour development*

Allow the solution to cool, transfer quantitatively to a 100 ml one-mark volumetric flask, add 2 ml of the diphenylcarbazide solution (3.5), dilute to the mark, mix and allow to stand for 2 min.

5.4.3 *Photometric measurements*

Carry out the photometric measurements on the test solution and on the blank test solution, as specified in 5.3.2, after having adjusted the instrument to zero absorbance against water.

6 EXPRESSION OF RESULTS

By reference to the calibration graph (5.3.3), determine the masses of chromium corresponding to the absorbances of the test solution and of the blank test solution.

The chromium content, expressed as milligrams of Cr 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 chromium found in the test solution;

m_2 is the mass, in micrograms, of chromium found in the blank test solution.

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.

ANNEX

ISO PUBLICATIONS RELATING TO (A) BORIC ACID, (B) BORIC OXIDE, (C) *di*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.