INTERNATIONAL STANDARD 3119

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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.

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

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
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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.