INTERNATIONAL STANDARD ISO 3124

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Crude sodium borates for industrial use — Determination of iron soluble in alkaline medium — 2,2′-Bipyridyl photometric method

Borates de sodium bruts à usage industriel — Dosage du fer soluble en milieu alcalin — Méthode photométrique au bipyridyle-2,2′

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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 3124 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	Spain
Belgium	Ireland	Switzerland
Bulgaria	Israel	Thailand
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Poland	U.S.S.R.
Germany	Romania	
Hungary	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

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Crude sodium borates for industrial use — Determination of iron soluble in alkaline medium — 2,2′-Bipyridyl photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a 2,2′-bipyridyl photometric method for the determination of the iron soluble in alkaline medium in crude sodium borates for industrial use.

The method is applicable to products in which the content of iron soluble in alkaline medium exceeds 2 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

Reduction of the trivalent iron with hydroxylammonium chloride, contained in an aliquot part of solution A (see ISO 2217) prepared for analysis of the soluble impurities in alkaline solution. Formation of the coloured complex between bivalent iron and 2,2′-bipyridyl in buffered system. Photometric measurement of the coloured complex at a wavelength of about 522 nm.

4 REAGENTS

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

4.1 Hydrochloric acid, approximately 2 N solution.

4.2 Hydroxylammonium chloride, 100 g/l solution.

4.3 Sodium hydroxide solution, ρ approximately 1,08 g/ml, about 7,5 % (*m/m*) solution or approximately 2 N.

4.4 Sodium acetate, anhydrous, 200 g/l solution.

4.5 2,2′-Bipyridyl, 5 g/l acid solution.

Dissolve 0,5 g of 2,2′-bipyridyl in 10 ml of hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution, or approximately 12 N solution, dilute to 100 ml and mix.

4.6 Iron, standard solution, corresponding to 1,0 g of Fe per litre.

Weigh, to the nearest 0,001 g, 7,022 g of ammonium iron(II) sulphate hexahydrate, dissolve in 100 ml of approximately 2 N sulphuric acid solution, dilute to 1 000 ml in a one-mark volumetric flask and mix.

1 ml of this standard solution contains 1,0 mg of Fe.

4.7 Iron, standard solution, corresponding to 0,010 g of Fe per litre.

Transfer 10,0 ml of the standard iron solution (4.6) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 μg of Fe.

Prepare this solution immediately before use.

4.8 Phenolphthalein, 10 g/l solution in 95 % (*V/V*) ethanol.

4.9 Indicator paper, pH range 4 to 6.

5 APPARATUS

Ordinary laboratory apparatus and

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

5.2 Photoelectric absorptiometer, fitted with the same cells and with filters allowing maximum transmission at about 522 nm.

6 PROCEDURE

6.1 Test portion

Transfer 50,0 ml of solution A (see ISO 2217) into a 250 ml beaker.

6.2 Blank test

Transfer 50,0 ml of solution B (see ISO 2217) into a 250 ml beaker and proceed as specified in 6.4.1 to 6.4.3.

6.3 Preparation of calibration graph

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

Into a series of seven 150 ml beakers containing 10 ml of the hydrochloric acid solution (4.1), transfer the volumes of the standard iron solution (4.7) indicated in the following table and dilute to about 60 ml.

Standard iron solution (4.7)	Corresponding mass of iron
ml	µg
0*	0
1,00	10,0
2,00	20,0
4,00	40,0
6,00	60,0
8,00	80,0
10,00	100,0

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

6.3.2 *Colour development*

Treat the contents of each beaker as follows :

Add 2 ml of the hydroxylammonium chloride solution (4.2).

After 2 to 3 min, add 10 ml of the sodium acetate solution (4.4) and 2 ml of the 2,2′-bipyridyl solution (4.5), mix and by adding drop by drop either the sodium acetate solution (4.4) or the hydrochloric acid solution (4.1), adjust the pH to between 4 and 6, using the indicator paper (4.9) externally.

Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark, mix and allow to stand for 5 min.

6.3.3 *Photometric measurements*

Using the spectrophotometer (5.1), at a wavelength of about 522 nm, or the photoelectric absorptiometer (5.2), 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.

6.3.4 *Plotting of the calibration graph*

Plot a graph having, for example, the iron (Fe) contents, in micrograms per 100 ml of standard colorimetric solutions (6.3.1), as abscissae and the corresponding net values of the absorbance as ordinates.

6.4 Determination

6.4.1 *Preparation of the test and blank solutions*

Add 1 drop of the phenolphthalein solution (4.8) to the test portion (6.1) and to the blank test solution (6.2) and neutralize using the sodium hydroxide solution (4.3). Add 10 ml of the hydrochloric acid solution (4.1).

6.4.2 *Colour development*

Proceed as 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, as specified in 6.3.3, after having adjusted the instrument to zero absorbance against water.

7 EXPRESSION OF RESULTS

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

The content of iron soluble in alkaline medium, expressed as milligrams of iron (Fe) per kilogram, is given by the formula :

$$(m_1 - m_2) \times \frac{500}{50} \times \frac{1\,000}{m_0} \times \frac{1}{1\,000}$$

$$= \frac{m_1 - m_2}{m_0} \times 10$$

where

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

m_1 is the mass, in micrograms, of iron found in the test solution;

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

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.

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.