INTERNATIONAL STANDARD 3122

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

Boric acid, boric oxide, *di*sodium tetraborates, sodium perborates and crude sodium borates for industrial use — Determination of iron content — 2,2′-Bipyridyl photometric method

Acide borique, oxyde borique, tétraborates disodiques, perborates de sodium et borates de sodium bruts à usage industriel — Dosage du fer — Méthode photométrique au bipyridyle-2,2′

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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 3122 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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Boric acid, boric oxide, *di*sodium tetraborates, sodium perborates and crude sodium borates for industrial use — Determination of iron content — 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 content of boric acid, boric oxide, *di*sodium tetraborates, sodium perborates and crude sodium borates for industrial use.

2 PRINCIPLE

Reduction of the trivalent iron by hydroxylammonium chloride, suppression of interference from tin, if necessary, with oxalic acid followed by the 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.

3 REAGENTS

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

3.1 Sodium carbonate, anhydrous (for crude sodium borates test only).

3.2 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution or approximately 12 N.

3.3 Hydrochloric acid, approximately 2 N solution.

3.4 Oxalic acid, 5 g/l solution.

3.5 Hydroxylammonium chloride, 100 g/l solution.

3.6 Potassium iodide, 50 g/l solution (for sodium perborates test only).

3.7 Sodium acetate, anhydrous, 300 g/l solution.

3.8 Sodium sulphite, 50 g/l solution (for sodium perborates test only).

3.9 2,2′-Bipyridyl, 5 g/l acid solution

Dissolve 0,5 g of 2,2′-bipyridyl in 10 ml of the hydrochloric acid (3.2), dilute to 100 ml and mix.

3.10 Iron, standard solution, corresponding to 1,00 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.

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

Transfer 10,0 ml of the standard iron solution (3.10) 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.

3.12 Indicator paper, pH range 4 to 6.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Platinum crucible, 35 mm diameter, 40 mm deep, with platinum lid (for crude sodium borates test only).

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 maximum transmission at about 522 nm.

5 PROCEDURE

5.1 Test portion

5.1.1 *Boric acid, boric oxide and* disodium *tetraborates*

Weigh, to the nearest 0,01 g, 4,0 g of the test sample.

5.1.2 *Sodium perborates*

Weigh, to the nearest 0,01 g, an amount of the test sample that corresponds to 4,0 g of $NaBO_2.H_2O_2.3H_2O$.

5.1.3 *Crude sodium borates*

Weigh, to the nearest 0,001 g, 0,5 g of the test sample directly into the platinum crucible (4.1).

NOTE — For test portions containing 100 to 500 μg of iron, see note to clause 6.

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 in the determination.

5.3 Preparation of calibration graph

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

Into a series of six 150 ml beakers containing 10 ml of the hydrochloric acid (3.3), transfer the volumes of the standard iron solution (3.11) indicated in the following table and dilute to about 60 ml.

Standard iron solution (3.11)	Corresponding mass of iron
ml	μg
0*	0
2,0	20
4,0	40
6,0	60
8,0	80
10,0	100

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

5.3.2 *Colour development*

Treat the contents of each beaker as follows :

Add 2 ml of the hydroxylammonium chloride solution (3.5) and bring to the boil on a hot-plate.

Add 1 ml of the oxalic acid solution (3.4), continue to boil gently for 3 min, then cool to between 50 and 60 °C. This step may be omitted if tin is known to be absent.

Add 10 ml of the sodium acetate solution (3.7) and cool to 20 °C.

Add 2 ml of the 2,2'-bipyridyl solution (3.9), mix and, by adding drop by drop either the sodium acetate solution (3.7) or the hydrochloric acid solution (3.3), adjust the pH value to between 4 and 6, using the indicator paper (3.12) externally.

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

If the solution is not perfectly clear, filter it through an acid-washed, iron-free, dry filter paper, or through any other suitable iron-free filtering medium, into a dry receiver.

5.3.3 *Photometric measurements*

Using the spectrophotometer (4.2), at a wavelength of about 522 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.

5.3.4 *Plotting of the calibration graph*

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

5.4 Determination

5.4.1 *Preparation of test solutions*

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

Transfer the test portion (5.1.1) 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 (3.2), and evaporate just to dryness.

Add 10 ml of the hydrochloric acid solution (3.3), and dilute to about 60 ml.

5.4.1.2 Sodium perborates

Transfer the test portion (5.1.2) to a 250 ml beaker and add 20 ml of water and 1 ml of the potassium iodide solution (3.6).

Heat the solution gently until the evolution of gas ceases (about 5 min is normally adequate). Add 20 ml of the hydrochloric acid solution (3.2), place on a boiling water bath and evaporate just to dryness.

Add 10 ml of the hydrochloric acid solution (3.3), dilute to about 60 ml and add 0,2 ml of the sodium sulphite solution (3.8).

5.4.1.3 Crude sodium borates

Add 1 g of the sodium carbonate (3.1) to the platinum crucible containing the test portion (5.1.3), mix and cover the crucible with its lid. Carefully heat the contents, maintain just at the fusion point until a clear melt is obtained and then cool to ambient temperature.

Mix 25 ml of the hydrochloric acid solution (3.2) with 50 ml of water. Cautiously using this mixture to digest the melt, transfer quantitatively the contents of the crucible to a 250 ml beaker.

Evaporate the solution to dryness on a boiling water bath.

Add 100 ml of the hydrochloric acid solution (3.3) and 100 ml of water, warm to 30 to 40 °C to dissolve the solids and transfer quantitatively to a 500 ml one-mark volumetric flask. Cool to 20 °C, dilute to the mark and mix.

Transfer 50,0 ml of this diluted solution to a 150 ml beaker containing 10 ml of water.

NOTE – In the preparation of these solutions, carefully controlled evaporation to low bulk may be carried out on a hot-plate before transferring to the boiling water bath.

5.4.2 *Colour development*

Proceed as specified in 5.3.2.

5.4.3 *Photometric measurements*

Carry out the photometric measurements on the test solution and on the blank test solution, as specified in 5.3.3, after having adjusted the instrument to zero absorbance against water.

6 EXPRESSION OF RESULTS

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

The iron (Fe) content, expressed in milligrams per kilogram, is given by the following formulae :

a) **boric acid, boric oxide, *di*sodium tetraborates and sodium perborates**

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

b) **crude sodium borates**

$$\frac{10 \times (m_1 - m_2)}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

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 corresponding blank test solution.

NOTE – If the iron content is found to be greater than 100 µg Fe, the approximate iron content can be obtained by the following procedure :

Reserve the blank test solution after measuring its absorbance against water. Take a suitable volume of the coloured test solution and dilute it to 100 ml with the reserved blank test solution. Measure the absorbance of this diluted solution against water.

Calculate the result using the formula :

$$\frac{100 \times (m_3 - m_2)}{V \times m_0}$$

or, in the case of crude sodium borates :

$$\frac{1\,000 \times (m_3 - m_2)}{V \times m_0}$$

where

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

m_3 is the mass, in micrograms, of iron found in the diluted test solution;

V is the volume, in millilitres, of the normal coloured test solution taken for dilution to 100 ml with the blank test solution.

This result can be used to calculate the mass of test portion that should be used if it is necessary to arrive at a more accurate result which is within the scope of the method.

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.

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