INTERNATIONAL STANDARD ISO 1916

G-92-07

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

Disodium tetraborates for industrial use — Determination of sodium oxide and boric oxide contents and loss on ignition

First edition — 1972-05-15

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 1916 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in July 1970 by the Member Bodies of the following countries:

- Australia
- Belgium
- Chile
- Czechoslovakia
- Egypt, Arab Rep. of
- France
- Germany
- Greece
- Hungary
- India
- Israel
- Japan
- Netherlands
- New Zealand
- Poland
- Portugal
- Romania
- South Africa, Rep. of
- Spain
- Switzerland
- Thailand
- Turkey
- United Kingdom
- U.S.S.R.

No Member Body expressed disapproval of the document.

Printed in Switzerland

Disodium tetraborates for industrial use — Determination of sodium oxide and boric oxide contents and loss on ignition

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods for the determination of the sodium oxide and boric oxide contents and of the loss on ignition of disodium tetraborates for industrial use, in their various states of hydration.

2 PRINCIPLE

Determination of the sodium oxide content by addition of an excess of a solution of hydrochloric acid and back titration with a standard volumetric solution of sodium hydroxide, using screened methyl red as indicator.

Subsequent titration of the boric oxide with the standard volumetric solution of sodium hydroxide in the presence of mannitol or sorbitol, using phenolphthalein as indicator.

Determination of the loss on ignition at 900 °C, expressed conventionally as the water content.

3 REAGENTS

Distilled water or water of equivalent purity, free from carbon dioxide, shall be used in the test.

3.1 Mannitol, neutral, or alternatively **sorbitol**, neutral.

These products shall satisfy the following condition :

5.0 g, dissolved in 50 ml of carbon dioxide-free water, requires for neutralization not more than 0.3 ml of 0.02 N sodium hydroxide using phenolphthalein solution as indicator.

3.2 Hydrochloric acid, 0.5 N standard volumetric solution.

3.3 Sodium hydroxide, 0.5 N standard volumetric solution, free from carbonate.

3.4 Screened methyl red, indicator solution.

Dissolve 0.01 g of methyl red and 0.01 g of bromocresol green in 95 % (V/V) ethanol and dilute to 100 ml with the same ethanol.

3.5 Phenolphthalein, 10 g/l ethanolic solution.

Dissolve 1 g of phenolphthalein in 95 % (V/V) ethanol, dilute to 100 ml with the same ethanol and add 0.02 N sodium hydroxide solution until the first appearance of a pink colour.

4 APPARATUS

4.1 Platinum crucible, 35 mm diameter and 40 mm deep, with lid.

4.2 Electric furnace, capable of being controlled at 900 ± 50 °C.

5 SAMPLING

Follow the principles described in ISO ....[1].

6 PROCEDURE

6.1 Test portion

For the determinations indicated, the mass of the laboratory sample to be taken and the precision of the weighing are given in the following Table :

TABLE — Mass of the test portion and precision of the weighing

Presumed state of hydration	Determination of sodium oxide and boric oxide		Determination of loss on ignition	
molecules of water	mass of test portion g	precision of weighing g	mass of test portion g	precision of weighing g
0 (anhydrous)	0.5	± 0.000 5	2.0	± 0.000 2
5	1.0	± 0.000 5	1.0	± 0.000 5
10	1.0	± 0.000 5	1.0	± 0.000 5

6.2 Determination of sodium oxide content

Dissolve the test portion (6.1) in about 70 ml of water by heating. Cool the solution to ambient temperature and add 0.4 ml of the screened methyl red indicator solution (3.4). Add 25.0 ml of the hydrochloric acid solution (3.2). Titrate with the sodium hydroxide solution (3.3) until the solution is just yellow. Retain this solution for the determination of boric oxide content (see 6.3).

Add 25.0 ml of the hydrochloric acid solution (3.2) to about 50 ml of water. Add 0.4 ml of the screened methyl red indicator solution (3.4) and titrate with the sodium hydroxide solution (3.3) until the solution is just yellow.

6.3 Determination of boric oxide content

To the solution retained from the determination of sodium oxide content (see 6.2), add approximately 15 g of the mannitol or sorbitol (3.1) and 0.4 ml of the phenolphthalein solution (3.5). Titrate the solution with the sodium hydroxide solution (3.3) to a distinct pink colour.

NOTE — The solution turns red on the addition of the mannitol or sorbitol; on titration with the sodium hydroxide solution the colour of the solution changes to yellow and subsequently to the phenolphthalein pink colour at the end point.

To ensure that the correct titration end point is obtained, the following standard colour matching solution may be used for comparison with the solution being titrated.

Mix

- 50 ml of 3.81 g/l solution of disodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$),
- 100 ml of water,
- 1.0 ml of the hydrochloric acid solution (3.2),
- 0.4 ml of the screened methyl red indicator solution (3.4),
- 0.4 ml of the phenolphthalein solution (3.5).

Equal volumes of this solution and of the titrand shall be compared in similar beakers.

6.4 Determination of loss on ignition

6.4.1 *Anhydrous salt*

Weigh the test portion as given in the Table into the tared platinum crucible (4.1), without its lid. Transfer the crucible to the electric furnace (4.2) and maintain it at 900 ± 50 °C for 15 min. Remove the crucible from the furnace, allow it to cool in a desiccator and weigh it again.

6.4.2 *Hydrated salt*

Weigh the test portion as given in the Table into the tared platinum crucible (4.1) with its lid. Adjust the flame of a Bunsen burner so that it is 50 mm long and clamp the burner in a nearly horizontal position with the flame angled slightly downwards. Cover the crucible and place it so that the Bunsen flame plays on its lid and heat in this manner for 20 min. Then use the burner to heat the crucible in the normal manner from below, for a further 10 min.

Transfer the covered crucible to the electric furnace (4.2) and maintain it at 900 ± 50 °C for 15 min. Remove the crucible from the furnace, allow it to cool in a desiccator and weigh it again with its lid.

7 EXPRESSION OF RESULTS

7.1 Sodium oxide content (Na_2O) is given, as a percentage by mass, by the formula :

$$\frac{V_2 - V_1}{m_1} \times 1.549$$

where

V_1 is the volume, in millilitres, of the sodium hydroxide solution (3.3) used in the titration of the excess of the hydrochloric acid solution (3.2);

V_2 is the volume, in millilitres, of the sodium hydroxide solution (3.3) used in the titration of the 25 ml of the hydrochloric acid solution (3.2);

m_1 is the mass, in grams, of the test portion (6.1).

7.2 Boric oxide content (B_2O_3) is given, as a percentage by mass, by the formula :

$$\frac{V_3}{m_1} \times 1.741$$

where

V_3 is the volume, in millilitres, of the sodium hydroxide solution (3.3) used in the titration, after addition of the mannitol or sorbitol (3.1);

m_1 is the mass, in grams, of the test portion (6.1).

7.3 Loss on ignition (expressed conventionally as water content) is given, as a percentage by mass, by the formula :

$$\frac{m_2 - m_3}{m_2} \times 100$$

where

m_2 is the mass, in grams, of the test portion (6.1);

m_3 is the mass, in grams, of the test portion after ignition.

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 regarded as optional.