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**Crude sodium borates for industrial use —  
Determination of sodium oxide and boric oxide contents —  
Volumetric method**

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## FOREWORD

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It was approved in August 1971 by the Member Bodies of the following countries :

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# Crude sodium borates for industrial use — Determination of sodium oxide and boric oxide contents — Volumetric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of the sodium oxide and boric oxide contents of crude sodium borates for industrial use.

## 2 PRINCIPLE

Determination of the sodium oxide content by treatment of an aqueous extract with an excess of hydrochloric acid solution, followed by back titration with standard volumetric sodium hydroxide solution in the presence of methyl red as indicator. Subsequent titration of the boric oxide with standard volumetric sodium hydroxide solution in the presence of mannitol or sorbitol and of phenolphthalein as indicator.

## 3 REAGENTS

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

### 3.1 Sodium chloride

3.2 Mannitol, neutral, or alternatively, sorbitol, neutral.

These products shall satisfy the following test requirement :

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 solution, phenolphthalein solution (3.6) being used as indicator.

3.3 Hydrochloric acid, approximately 0.5 N solution.

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

3.5 Methyl red, 0.1 g/l ethanolic solution.

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

3.6 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

Ordinary laboratory apparatus.

## 5 PROCEDURE

### 5.1 Test portion

Weigh, to the nearest 0.000 5 g, about 5 g of the test sample, previously ground to pass a sieve of 250  $\mu\text{m}$  aperture, and transfer to a beaker.

### 5.2 Preparation of test solution

Add to the beaker about 150 ml of water, boil the solution gently for 5 min, then transfer the beaker and contents to a boiling water bath, and digest for 15 min, stirring occasionally. Transfer the contents of the beaker quantitatively to a 250 ml one-mark volumetric flask, add 5 g of the sodium chloride (3.1), swirl to dissolve, dilute with water to almost 250 ml and allow to cool to room temperature. Dilute to the mark with water, mix and leave the flask undisturbed until the insoluble matter has settled. Carefully decant the clear supernatant liquid through a dry medium grade filter paper, collecting the filtrate in a dry beaker. As far as possible, avoid transferring the insoluble matter, as this will greatly retard filtration.

### 5.3 Determination of sodium oxide content

By means of a pipette, transfer 50.0 ml of the filtrate (5.2) into a beaker, dilute to about 100 ml with water and add 0.2 ml of the methyl red solution (3.5). Using a pipette, add 25.0 ml of the hydrochloric acid solution (3.3) and simmer gently for 5 min with a clock glass covering the beaker. Allow to cool and titrate the solution with the sodium hydroxide solution (3.4) to the methyl red end point. Retain this solution for the determination of boric oxide content (5.4).

At the same time, determine the equivalence of the solutions (3.3 and 3.4) as follows :

From a pipette add 25.0 ml of the hydrochloric acid solution (3.3) to about 50 ml of water.

Add 0.2 ml of the methyl red solution (3.5) and titrate with the sodium hydroxide solution (3.4).

#### 5.4 Determination of boric oxide content

To the test solution from the determination of sodium oxide content (5.3), add approximately 15 g of mannitol or of sorbitol (3.2) and 0.4 ml of the phenolphthalein solution (3.6). Titrate the solution with the standard volumetric sodium hydroxide solution (3.4) to a distinct phenolphthalein 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 compared with the solution being titrated.

Mix :

50 ml of a 3.81 g/l solution of disodium tetraborate decahydrate ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ );

100 ml of distilled water, free from carbon dioxide;

1.0 ml of the standard volumetric hydrochloric acid solution (3.3);

0.2 ml of the methyl red solution (3.5);

0.4 ml of the phenolphthalein solution (3.6).

An equal volume of this solution should be compared with the titrand in a similar beaker.

## 6 EXPRESSION OF RESULTS

### 6.1 Sodium oxide content

Sodium oxide content, expressed as  $\text{Na}_2\text{O}$ , is given, as a percentage by mass, by the formula :

$$\frac{V_1 - V_2}{m} \times 7.75$$

where

$V_1$  is the volume, in millilitres, of the standard

volumetric sodium hydroxide solution (3.3) used in the titration of 25.0 ml of the hydrochloric acid solution (3.3);

$V_2$  is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.4) used in the titration of the excess hydrochloric acid solution (3.3);

$m$  is the mass, in grams, of the test portion.

### 6.2 Boric oxide content

Boric oxide content, expressed as  $\text{B}_2\text{O}_3$ , is given, as a percentage by mass, by the formula :

$$\frac{V_3}{m} \times 8.70$$

where

$V_3$  is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.4) used, after addition of mannitol or sorbitol (3.2);

$m$  is the mass, in grams, of the test portion.

## 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.