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Boric acid, boric oxide, disodium tetraborates and crude sodium borates for industrial use — Determination of sulphur compounds — Volumetric method

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FOREWORD

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

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

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Boric acid, boric oxide, disodium tetraborates and crude sodium borates for industrial use — Determination of sulphur compounds — Volumetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of sulphur compounds in boric acid, boric oxide, *di*sodium tetraborates and crude sodium borates for industrial use.

2 PRINCIPLE

Reduction of sulphur compounds to hydrogen sulphide by heating with a mixture of hydriodic acid and hypophosphorous acid. Absorption of the evolved hydrogen sulphide in a mixture of sodium hydroxide and acetone, followed by titration with mercuric acetate standard volumetric solution using dithizone as indicator.

3 REAGENTS

Distilled water or water of equivalent purity shall be used in the test.

3.1 Acetone.

3.2 Nitrogen, oxygen free.

3.3 Hydrochloric acid, ρ 1,19 g/ml approximately 38 % (*m*/*m*) solution.

3.4 Sodium hydroxide, approximately N solution.

3.5 Reducing solution.

In a 500 ml round-bottomed flask, fitted with a ground glass joint, mix

- 50 ml of hypophosphorous acid, 50 % (*m*/*m*),
- 100 ml of the hydrochloric acid solution (3.3),
- 120 ml of hydriodic acid, ρ 1,9 g/ml approximately.

Attach a reflux condenser and insert a glass tube down the condenser to permit the introduction of a stream of nitrogen (3.2) into the mixture. Boil under reflux for 4 h, allowing a continuous stream of nitrogen to pass through the mixture. Allow the mixture to cool while the nitrogen is still flowing. Store in a dark coloured glass bottle.

3.6 Mercury(II) acetate, 0,001 M standard volumetric solution.

Dissolve 0,318 7 g of mercury (II) acetate in water and dilute to 1 000 ml.

3.7 Dithizone, 1 g/l solution in acetone.

Dissolve 0,1 g of dithizone in 100 ml of acetone (3.1).

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Apparatus as illustrated in the Figure, fitted with ground glass joints[1] and comprising the following components :

A) **Flask,** round-bottomed, with one central neck and one angled side neck;

B) **Condenser,** Liebig;

C) **Receiver;**

D) **Microburette,** with lateral stopcock, capacity 2 ml, graduated in 0,01 ml.

5 PROCEDURE

5.1 Test portion

5.1.1 *Boric acid, boric oxide and disodium tetraborates*

Weigh, to the nearest 0,001 g, 1,0 g of the laboratory sample.

5.1.2 *Crude sodium borates*

Weigh, to the nearest 0,001 g, 0,5 g of the finely ground laboratory sample.

5.2 Determination

Transfer the test portion (5.1) into the flask (A), and add 1 ml of water. Add 5 ml of the sodium hydroxide solution (3.4) and 5 ml of acetone (3.1) to the receiver (C). With the exception of the boiling water bath, assemble the apparatus as shown in the Figure. Add, through the side neck of flask (A), 10 ml of the reducing solution (3.5),

1) See ISO/R 383.

immediately replace the side arm and adjust the nitrogen flow to about 15 ml/min. Place the boiling water bath in position and proceed to heat the flask (A). Add to the receiver (C) 0,1 ml of the dithizone solution (3.7) and, from the microburette (D), 1 drop of the mercury (II) acetate standard volumetric solution (3.6). The contents of the receiver (C) will be coloured red. The first evolution of hydrogen sulphide will be indicated by the contents of the receiver (C) changing from red to yellow. When this occurs, continue to heat the flask for a further 20 min.

With the stream of nitrogen (3.2) still flowing, titrate the contents of the receiver with the mercury (II) acetate standard volumetric solution (3.6) until a permanent red colour is obtained.

5.3 Blank test

Add 1 ml of water to the flask (A) and proceed according to the instructions given in 5.2.

6 EXPRESSION OF RESULTS

Content of sulphur compounds, expressed as sulphate (SO_4), is given, in milligrams per kilogram, by the formula :

$$\frac{V_1 - V_2}{m} \times 96$$

where

V_1 is the volume, in millilitres, of the mercury (II) acetate standard volumetric solution (3.6) used in the titration;

V_2 is the volume, in millilitres, of the mercury (II) acetate standard volumetric solution (3.6) used in the blank test;

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.

Dimensions in millimetres

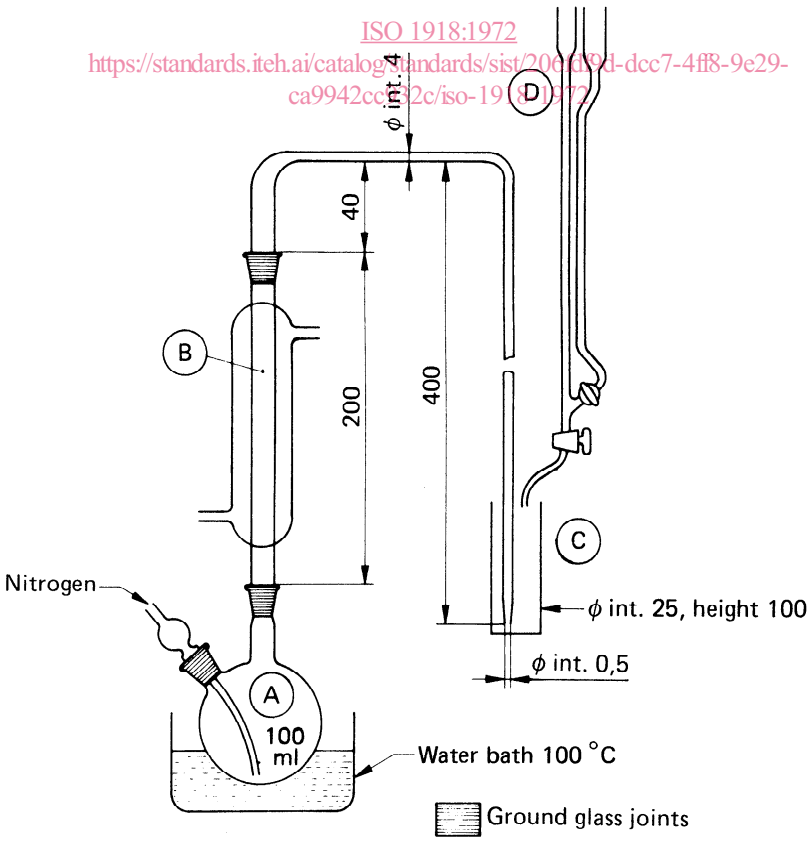

FIGURE — **Apparatus (4.1)**