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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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International Standard ISO 1918 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

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

| | | |
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No Member Body expressed disapproval of the document.





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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,** $\rho$ 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, $\rho$ 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.