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Sulphur for industrial use — Determination of acidity — Titrimetric method

Soufre à usage industriel — Détermination de l'acidité — Méthode titrimétrique

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FOREWORD

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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 3704 was drawn up by Technical Committee ISO/TC 47, Chemistry, and was circulated to the Member Bodies in January 1975.

It has been approved by the Member Bodies of the following countries:

India

Austria Belgium Brazil Bulgaria Czechoslovakia France

Ireland Israel Poland Portugal Romania Spain Switzerland Turkey

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United Kingdom U.S.S.R.

Germany South Africa, Rep. of

No Member Body expressed disapproval of the document.

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Sulphur for industrial use — Determination of acidity — Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a titrimetric method for the determination of the acidity of sulphur for industrial use.

The method is applicable to products having acidities, expressed as H_2SO_4 , equal to or greater than 0,01 % (m/m).

2 PRINCIPLE

Extraction of acidic substances with a mixture of water and propan-2-ol. Titration of the extract with standard volumetric sodium hydroxide solution in the presence of phenolphthalein as indicator.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water, freshly boiled and cooled, or water of equivalent purity.

- **3.1** Propan-2-ol, previously boiled, cooled, and neutralized in the presence of the phenolphthalein solution (3.3).
- **3.2 Sodium hydroxide,** 0,1 N standard volumetric solution, freshly prepared and carbonate-free.
- 3.3 Phenolphthalein, 10 g/l ethanolic solution.

Dissolve 1 g of phenolphthalein in 60 ml of 95 % (V/V) ethanol and dilute to 100 ml with water.

4 APPARATUS

Ordinary laboratory apparatus.

5 SAMPLING AND PREPARATION OF TEST SAMPLE

Follow the procedure specified in the appropriate International Standard.¹⁾

Prepare a test sample from the laboratory sample by grinding a sufficient quantity of the undried sample until it passes through a sieve of nominal aperture 250 μ m.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,1 g, about 25 g of the test sample (clause 5) in a 250 ml conical flask fitted with a ground glass stopper.

6.2 Determination

To the conical flask containing the test portion (6.1), add 25 ml of the propan-2-ol (3.1), stopper the flask and shake until the sulphur is completely wetted. Then add 50 ml of water, stopper the flask, and shake again for 2 min. Allow to stand for 20 min, shaking occasionally.

Titrate with the standard volumetric sodium hydroxide solution (3.2), in the presence of a few drops of the phenolphthalein solution (3.3), to the first permanent pink colour.

7 EXPRESSION OF RESULTS

The acidity, expressed as a percentage by mass of sulphuric acid (H_2SO_4) , is given by the formula

$$V \times \frac{1}{10} \times 0,049 \times \frac{100}{m} = \frac{0,49 \times V}{m}$$

where

V is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.2) used in the determination;

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

0,049 is the mass, in grams, of sulphuric acid (H_2SO_4) corresponding to 1 ml of exactly 1 N sodium hydroxide solution.

NOTE — If the standard volumetric solution used does not have exactly the concentration given in the list of reagents, an appropriate correction shall be applied.

¹⁾ Under study.

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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.

ANNEX

ISO PUBLICATIONS RELATING TO SULPHUR FOR INDUSTRIAL USE

ISO 2866 — Determination of total carbon content — Titrimetric method.

ISO 3425 — Determination of ash content at 850-900 $^{\circ}$ C and of residue at 200 $^{\circ}$ C.

ISO 3426 - Determination of loss in mass at 80 °C.

ISO 3704 — Determination of acidity — Titrimetric method.

ISO 3705 — Determination of arsenic content — Silver diethyldithiocarbamate photometric method.

ISO 5793 - Determination of chloride content - Photometric method.