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Sulphur for industrial use — Determination of total carbon content — Titrimetric method

Soufre à usage industriel — Dosage du carbone total — Méthode titrimétrique

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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 2866 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1972.

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

Australia	Ireland	South Africa, Rep. of
Belgium	Israel	Spain
Czechoslovakia	Italy	Sweden
Egypt, Arab Rep. of	Mexico	Switzerland
France	Netherlands	Thailand
Germany	New Zealand	Turkey
Hungary	Portugal	United Kingdom
India	Romania	U.S.S.R.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

Sulphur for industrial use – Determination of total carbon content – Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the total carbon content of sulphur for industrial use.

Two alternative methods are given for the final titration :

- a potentiometric method;
- a visual method.

2 PRINCIPLE

Combustion of the sulphur in a current of oxygen.

Oxidation and fixation of the sulphur dioxide and trioxide gases in chromic and sulphuric acid solutions.

Absorption of the carbon dioxide formed in a solution of barium hydroxide, followed by either potentiometric or visual titration in the presence of phenolphthalein and screened methyl orange as indicator.

3 REAGENTS

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

3.1 Pure oxygen (free from CO_2), in cylinders fitted with pressure-reducing valves.

3.2 Pure nitrogen (free from CO_2), in cylinders fitted with pressure-reducing valves.

3.3 Chromium(VI) oxide (CrO_3), 500 g/l solution.

3.4 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (m/m) solution.

3.5 Soda-asbestos.

3.6 Platinized asbestos, containing 5 to 10 % of platinum.

3.7 Hydrogen peroxide, approximately 60 g/l solution (20 volumes).

3.8 Barium hydroxide, freshly prepared 0,05 N solution, containing a few drops of the phenolphthalein solution (3.12).

This solution shall be protected from atmospheric carbon dioxide by means of a trap containing soda-asbestos.

3.9 Hydrochloric acid, 0,05 N standard volumetric solution.

3.10 Sodium hydroxide, 0,05 N standard volumetric solution.

3.11 Screened methyl orange indicator.

Dissolve 0,25 g of methyl orange and 0,15 g of xylene cyanol FF in 50 ml of 95 % (V/V) ethanol.

3.12 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 and

4.1 Combustion and absorption apparatus, giving complete and controlled combustion. An example of a suitable apparatus is shown in figure 1. This apparatus is fitted with spherical ground glass joints, lubricated with sulphuric acid, and comprises :

4.1.1 Mercury lute, fitted with an inner tube dipping 1 cm below the surface of the mercury (A).

4.1.2 Three U-tubes, with side arms and ground stoppers, of diameter 15 mm and height 150 mm (B_1 , B_2 , B_3).

4.1.3 Flow meter, suitable for oxygen flow measurements of between 20 and 200 ml/min (C).

4.1.4 Combustion tube, transparent silica, 60 cm long, external diameter 15 mm, reduced at one end over a length of 15 mm to an external diameter of 4 mm (D).

4.1.5 Furnace, capable of being maintained at 800 to 900 °C during the combustion (E).

4.1.6 Furnace, capable of being maintained at 400 to 450 °C during the combustion (F).