International Standard 7266

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Copper and copper alloys — Determination of sulfur content — Combustion titrimetric method

Cuivre et alliages de cuivre — Dosage du soufre — Méthode titrimétrique après combustion

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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.

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It has been approved by the member bodies of the following countries :

Australia	Italy	Sweden
Belgium	Japan	Switzerland
Canada	Korea, Dem. P. Rep. of	Thailand
Czechoslovakia	Mexico	Turkey
Finland	Netherlands	USA
France	Poland	USSR
Germany, F.R.	Romania	Venezuela
Hungary	South Africa, Rep. of	
Iran	Spain	

No member body expressed disapproval of the document.

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Copper and copper alloys — Determination of sulfur content — Combustion titrimetric method

1 Scope and field of application

This International Standard specifies a combustion titrimetric method for the determination of the sulfur content of copper and copper alloys.

The method is applicable to contents of sulfur greater than 0,010 % (m/m) in all types of copper and copper alloys listed in International Standards.

2 Principle

Combustion of a test portion in oxygen at 1 250 °C. Absorption of combustion gases in dilute hydrogen peroxide solution. Titration of the sulfuric acid formed with sodium borate in the presence of a mixed methyl red-methylene blue indicator solution.

3 Reagents

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

3.1 Hydrogen peroxide, approximately 3 g/l solution.

Dilute 10 ml of hydrogen peroxide, 30 % (m/m) to 1 000 ml with water.

3.2 Sulfuric acid, solution, c (H_2SO_4) ≈ 0,002 5 mol/l

Dilute 14 ml of sulfuric acid (ϱ 1,84 g/l) to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml.

3.3 Sodium borate, standard solution.

Dissolve 1,189 5 g of sodium borate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) in water and dilute to the mark in a 1 000 ml one-mark volumetric flask.

1 ml of this standard solution is equivalent to 100 µg of S.

3.4 Mixed indicator

Dissolve 0,120 g of methyl red and 0,080 g of methylene blue in 100 ml of ethanol.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Burette, 25 ml, with 0,05 ml graduations.

4.2 Combustion apparatus (see figure 1), consisting of the following :

4.2.1 Oxygen bottle with pressure regulator and flowmeter (D). The oxygen must be sulfur-free.

4.2.2 Purging tubes (A_1 and A_2). A_1 is packed with asbestos treated with sodium hydroxide. The bottom part of A_2 is filled to three-quarters of its height with anhydrous magnesium perchlorate; the top part is filled with phosphorus(V) oxide. The two substances are separated by a plug of glass wool.

4.2.3 Two-way valve (R), with 3 to 4 mm tubing, such that oxygen can flow into the combustion tube T (4.2.6) and the combustion gases can flow into the bubbler tube B (4.3.2).

4.2.4 Mercury check valve (S), with an equilibrium flask and a safety tube. The level of mercury is adjusted, by means of the equilibrium flask, so that a seal is made when, with valve R (4.2.3) open, the gas flows from the combustion tube at a rate of 2,5 l/min. When valve R is opened, an overpressure is created, and the mercury seal operates until normal pressure is established.

4.2.5 Pressure-release container (V).

4.2.6 Combustion tube (T), made of non-porous refractory material, in which the combustion boat containing the test portion is placed (see 7.2).

4.2.7 Combustion boats, previously calcined at 1 250 °C in a stream of oxygen (see 7.3).

4.2.8 Tube furnace (F), capable of maintaining the heated portion of the combustion tube T (4.2.6) at 1 250 °C, with a metal cooling head for the combustion tube (see figure 3).

4.2.9 Glass outlet tube, of the same diameter as the combustion tube T (4.2.6), connected to the combustion tube by a rubber sleeve (b).