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Copper and copper alloys — Determination of antimony content — Rhodamine B spectrometric method

Cuivre et alliages de cuivre — Dosage de l'antimoine — Méthode spectrométrique à la rhodamine B .

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Foreword

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

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INTERNATIONAL STANDARD ISO 5956-1984 (E)

Copper and copper alloys — Determination of antimony content — Rhodamine B spectrometric method

1 Scope and field of application

This International Standard specifies a Rhodamine B spectrometric method for the determination of the antimony content of copper and copper alloys.

The method is applicable to antimony contents between 0,001 and 0,1 % (*m/m*) in all types of copper and copper alloys listed in International Standards. The range of application may be extended by appropriate modification of the mass of the test portion, the extraction volume and the cell path length. Specific proposals to cover lower and higher concentrations are in preparation.

2 Principle

Extraction of pentavalent antimony into isopropyl ether and spectrometric determination of the chloroantimonate-Rhodamine B complex.

3 Reagents

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

3.1 Isopropyl ether.

3.2 Hydrochloric acid, ϱ 1,19 g/ml.

3.3 Hydrogen peroxide, 30 % (*m/m*).

3.4 Hydrochloric acid, solution, diluted 7 + 3.

Mix 700 ml of the hydrochloric acid (3.2) with 300 ml of water.

3.5 Cerium(IV) sulfate, solution.

Dissolve 4 g of cerium(IV) sulfate tetrahydrate [$Ce(SO_4)_2.4H_2O$] and dilute to 100 ml with 0,5 mol/l sulfuric acid solution.

3.6 Hydroxylammonium chloride, solution.

Dissolve 1 g of hydroxylammonium chloride ($NH_2OH.HCl$) in water and dilute to 100 ml.

Use a freshly prepared solution only.

3.7 Hydrochloric acid, 1 mol/l solution.

Dilute 83 ml of the hydrochloric acid (3.2) to 1 000 ml with water.

3.8 Rhodamine B, solution.

Dissolve 0,01 g of Rhodamine B and dilute to 100 ml with the hydrochloric acid solution (3.7).

Filter the solution before use.

3.9 Antimony, standard solution corresponding to 1,000 g of Sb per litre.

Dissolve 0,274 3 g of potassium antimonyl tartrate hemihydrate [$K(SbO)C_4H_4O_6 \cdot 0,5H_2O$] and make up to volume with the hydrochloric acid solution (3.4) in a 100 ml one-mark volumetric flask.

1 ml of this standard solution contains 1,000 mg of Sb.

3.10 Antimony, standard solution corresponding to 100 mg of Sb per litre.

Dilute 10 ml of the antimony standard solution (3.9) to the mark with the hydrochloric acid solution (3.4) in a 100 ml one-mark volumetric flask.

1 ml of this standard solution contains 100 µg of Sb.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Refrigeration cupboard.

4.2 Spectrometer.

5 Procedure

5.1 Depending on the expected antimony content of the sample, weigh a test portion according to the table and transfer it to a 250 ml conical flask.

Table

Expected Sb content	Mass of test portion, m_0	Volume of HCl (3.2), V_1	Volume of test solution to be taken V_0
% (*m*/*m*)	g	ml	ml
0,001 to 0,004	2	10	5
0,005 to 0,02	1	15	2
0,03 to 0,1	0,5	15	1

5.2 Dissolve the test portion (5.1) in 15 ml of the hydrochloric acid (3.4) and add 5 to 10 ml of the hydrogen peroxide solution (3.3), in small portions. Cool until the violent reaction has ceased. When the test portion is completely dissolved, heat the solution to boiling for several minutes to decompose the excess hydrogen peroxide. Cool to room temperature. Transfer the test solution to a 200 ml one-mark volumetric flask. Dilute to the mark with the hydrochloric acid solution (3.4) and mix.

5.3 According to the table, transfer to a separating funnel, V_1 ml of the hydrochloric acid (3.2) previously cooled to 5 °C or below in the refrigeration cupboard (4.1), and V_0 ml of the test solution.

5.4 Carry out the following operations without interruption.

Add 6 drops of the cerium(IV) sulfate (3.5) and mix. After 2 min, add 3 drops of the hydroxylammonium chloride solution (3.6) and mix again. Add 10 ml of the isopropyl ether (3.1) at a maximum temperature of 20 °C. Shake thoroughly for 30 s. Add 75 ml of water previously cooled to 5 °C or below and shake for 15 s. Allow to stand for 5 min.

Discard the aqueous layer and wash the organic layer twice with 3 ml of the hydrochloric acid solution (3.7). Add 20 ml of the Rhodamine B solution (3.8), shake for 30 s and allow to stand for 2 min. Drain the aqueous layer together with a small amount of the organic layer. Collect the remaining organic layer in a small, dry, stoppered flask. Swirl the flask to collect water droplets on the bottom. Transfer the organic phase to a 1 cm cell and immediately measure the absorbance against water at about 550 nm.

5.5 Carry a blank test through all steps. Correct the result for the blank.

5.6 Check test

Make a preliminary check of the apparatus by preparing a solution of standard material or a synthetic sample containing a known amount of antimony and of composition similar to the material to be analysed, and carrying out the procedure as specified in 5.1 to 5.4.

5.7 Preparation of calibration curve

To a series of 250 ml conical flasks, transfer 1 g of pure copper (antimony-free) and 0 to 6 ml of the standard antimony solution (3.9). Treat the samples according to 5.2. Using 15 ml of the hydrochloric acid (3.2) and 1 ml of the standard matching solutions, proceed to 5.3 and 5.4. The solutions for spectrometric measurements thus prepared will contain 0 to 6 µg of Sb.

6 Expression of results

Calculate the antimony content, as a percentage by mass, by the formula

$$\frac{m_1 \times 100}{V_0 \times m_0 \times 10^6} \times 100$$

$$= \frac{m_1}{V_0 \times m_0 \times 100}$$

where

m_0 is the mass, in grams, of the test portion (5.1);

m_1 is the mass, in micrograms, of antimony determined in the volume V_0;

V_0 is the volume, in millilitres, of the test solution taken (see 5.3).

7 Test report

The test report shall include the following particulars:

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operations not included in this International Standard or regarded as optional which might affect the results.