International Standard 4751

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Copper and copper alloys — Determination of tin content — Spectrometric method

Cuivre et alliages de cuivre — Dosage de l'étain — Méthode spectrométrique

First edition — 1984-08-01

UDC 669.3 : 543.42 : 546.811 **Ref. No. ISO 4751-1984 (E)**

Descriptors: copper, copper alloys, chemical analysis, determination of content, tin, spectrophotometric analysis.

Price based on 3 pages

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 4751 was developed by Technical Committee ISO/TC 26, *Copper and copper alloys*, and was circulated to the member bodies in August 1982.

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

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The member bodies of the following countries expressed disapproval of the document on technical grounds:

Australia
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Copper and copper alloys — Determination of tin content — Spectrometric method

1 Scope and field of application

This International Standard specifies a spectrometric method for the determination of tin in copper and copper alloys.

The method is applicable to the determination of tin contents between 0,005 and 0,5 % (m/m) in all types of copper and copper alloys listed in International Standards.

A method applicable to the determination of tin contents below 0,005 % (m/m) is in preparation.

2 Principle

Extraction into methyl isobutyl ketone and spectrometric determination of the yellow tin-quercetin complex.

3 Reagents

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

3.1 Hydrochloric acid, ϱ 1,19 g/ml.

3.2 Methyl isobutyl ketone.

3.3 Ammonia solution, ϱ 0,91 g/ml.

3.4 Hydrogen peroxide, 30 % (m/m).

3.5 Hydrochloric acid, solution, diluted 1 + 1.

Dilute 50 ml of the hydrochloric acid (3.1) with 50 ml of water.

3.6 Sulfuric acid, solution, diluted 1 + 19.

Dilute 50 ml of sulfuric acid, ϱ 1,84 g/ml, to 1 000 ml with water.

3.7 Thiourea, 60 g/l solution.

Dissolve 15 g of thiourea (NH_2CSNH_2) in water and dilute to 250 ml.

3.8 Ascorbic acid, 20 g/l solution.

Dissolve 1 g of ascorbic acid in water and dilute to 50 ml.

Use a freshly prepared solution.

3.9 Quercetin, 1 g/l acid solution in ethanol.

Dissolve 500 mg of quercetin in 300 ml of 95 % (V/V) ethanol in a 500 ml one-mark volumetric flask. The dissolution takes some hours. Add 25 ml of the hydrochloric acid (3.1), dilute to the mark with the same ethanol, and mix. Filter off any residue.

3.10 Tin, standard solution, corresponding to 0,5 g of Sn per litre.

Dissolve 500 mg of pure tin in 100 ml of the hydrochloric acid (3.1) in a 1 000 ml one-mark volumetric flask. Cool, dilute to the mark with water, and mix.

1 ml of this standard solution contains 0,5 mg of Sn.

3.11 Tin, standard solution corresponding to 0,050 g of Sn per litre.

Transfer 10,0 ml of the standard tin solution (3.10) to a 100 ml one-mark volumetric flask. Add 20 ml of the hydrochloric acid (3.1). Dilute to the mark with water and mix.

1 ml of this standard solution contains 50 µg of Sn.

3.12 Copper, 1 g/l base solution.

Dissolve 5,0 g of electrolytic copper (free of tin) in 100 ml of the hydrochloric acid (3.1). Add 5 ml portions of the hydrogen peroxide (3.4) until the copper is completely dissolved, then add 200 ml of water and boil to destroy the excess hydrogen peroxide. Cool and transfer to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 Spectrometer.

5 Procedure

5.1 According to the expected tin content of the sample, weigh, to the nearest 0,001 g, a test portion according to the table. Transfer the test portion to a 250 ml conical flask.

Table

Expected tin content of sample % (m/m)	Mass of test portion g	Predilution: Aliquot portion taken ml	Predilution: Diluted to ml
0,005 to 0,010	2	No predilution	
0,01 to 0,02	1	No predilution	
0,02 to 0,04	1	100	200
0,04 to 0,08	1	50	200
0,08 to 0,16	1	25	200
0,16 to 0,30	0,5	25	200
0,30 to 0,50	0,4	20	200

5.2 Under cold running water, attack the test portion with 20 ml of the hydrochloric acid solution (3.5) and 10 ml of the hydrogen peroxide solution (3.4), in small portions. It is necessary to dissolve slowly to avoid loss of tin chloride. Complete the dissolution by heating gently. Add 10 ml of water and boil to destroy the excess hydrogen peroxide. Transfer the test solution to a 200 ml one-mark volumetric flask.

5.3 If according to the table no predilution is to be made, proceed as directed in 5.4. If a predilution is to be made, dilute to 200 ml with water and mix. Transfer the prescribed aliquot portion to a 200 ml one-mark volumetric flask.

5.4 Neutralize with the ammonia solution (3.3) just to the point where a precipitate appears and remains. Dissolve the precipitate with the hydrochloric acid (3.1) added drop by drop. Add 20 ml of water and 30,0 ml of the hydrochloric acid. Cool, dilute to the mark with water, and mix.

5.5 Introduce successively into a 125 ml separating funnel

- 25 ml of the thiourea solution (3.7);
- 5 ml of the ascorbic acid solution (3.8);
- 10,0 ml of the quercetin solution (3.9); mix;
- 10,0 ml of the sample solution, while mixing;
- 20,0 ml of the methyl isobutyl ketone (3.2).

5.6 Shake for 1 min and allow the layers to separate for 3 min. Discard the aqueous layer. Add 5 ml of the sulfuric acid solution (3.6), without mixing. Drain off the acid phase and add 20 ml of the sulfuric acid solution. Shake for 30 s. During shaking, avoid too close an intermixture of the phases; this will prolong the separation time. After 3 min, drain off and discard the acid phase and a little of the organic phase. Transfer the remainder of the organic phase through a small, dry, rapid filter paper into a dry, stoppered flask. After 10 min, measure the absorbance of the solution using the spectrometer (4.1), fitted with a 20 mm cell, against methyl isobutyl ketone at 440 nm.

5.7 Carry a blank test throughout the procedure and correct the results for the blank. Use the same mass of tin-free electrolytic copper as the test portion mass.

5.8 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 tin and of composition similar to the material to be analysed, and carrying out the procedure as specified in 5.1 to 5.7.

6 Preparation of calibration graph

Add 50 ml of the copper base solution (3.12) to each of seven 200 ml one-mark volumetric flasks. Add 10 ml of water and boil to destroy the excess hydrogen peroxide. Add increasing amounts of the standard tin solution (3.11), i.e. 0 — 0,5 — 1,0 — 2,0 — 3,0 — 4,0 — 5,0 ml, corresponding to 0 — 25 — 50 — 100 — 150 — 200 — 250 µg respectively of tin. Proceed as specified in 5.4, 5.5 and 5.6. Prepare the calibration graph from the measured values after correcting the results for the blank.

7 Expression of results

Read from the calibration graph the mass of tin, in micrograms, corresponding to the measured absorbance value (after correction for the blank) and calculate the tin content of the sample, as a percentage by mass, according to one of the following formulae:

— with no predilution:

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

$$= \frac{m_1 \times 10^{-4}}{m_0}$$

— with predilution:

$$\frac{m_1 \times 10^{-6} \times 200}{m_0 \times V} \times 100$$

$$= \frac{m_1 \times 2 \times 10^{-2}}{m_0 \times V}$$

where

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

m_1 is the mass, in micrograms, of tin found in the test solution;

200 is the diluted volume, in millilitres, of the aliquot portion taken for the determination (5.1);

V is the volume, in millilitres, of the aliquot portion taken for the determination (5.1).

8 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 operation not included in this International Standard or regarded as optional which might affect the results.

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