International Standard 4748

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

Copper alloys — Determination of iron content — Na_2EDTA titrimetric method

Alliages de cuivre — Dosage du fer — Méthode titrimétrique au Na_2EDTA

First edition — 1984-04-15

UDC 669.3. : 543.24 : 546.72 **Ref. No. ISO 4748-1984 (E)**

Descriptors: copper, copper alloys, chemical analysis, determination of content, iron, volumetric analysis.

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (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 authorized 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 4748 was developed by Technical Committee ISO/TC 26, *Copper and copper alloys*, and was circulated to the member bodies in September 1982.

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

Belgium	Iran	Spain
Brazil	Italy	Sweden
Canada	Japan	Switzerland
China	Korea, Dem. P. Rep. of	Turkey
Czechoslovakia	Korea, Rep. of	USA
Egypt, Arab Rep. of	Norway	USSR
France	Poland	Venezuela
Germany, F.R.	Romania	

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

Australia
South Africa, Rep. of

Printed in Switzerland

Copper alloys — Determination of iron content — Na_2EDTA titrimetric method

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the iron content of copper alloys.

The method is applicable to contents of iron in all types of copper alloys listed in International Standards.

2 Principle

Separation of iron from copper by extraction as the iron(III)-chloro complex, followed by chelation with excess EDTA at pH 4,5 and back-titration with a standard volumetric zinc solution.

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

3.3 Methyl isobutyl ketone.

3.4 Ethanol.

3.5 Hydrochloric acid, diluted 1 + 1.

Dilute 100 ml of the hydrochloric acid (3.1) with 100 ml of water.

3.6 Lithium chloride, solution.

Dissolve 275 g of lithium chloride (LiCl) in water and dilute to 1 000 ml.

3.7 Ammonium fluoride, solution.

Dissolve 37 g of ammonium fluoride (NH_4F) in water and dilute to 1 000 ml.

3.8 Thiourea, solution.

Dissolve 100 g of thiourea (H_2NCSNH_2) in water and dilute to 1 000 ml.

3.9 Hexamethylenetetramine, solution.

Dissolve 200 g of hexamethylenetetramine in water and dilute to 1 000 ml.

3.10 *Di*sodiumethylenediaminetetraacetate dihydrate (Na_2EDTA), 0,05 mol/l standard volumetric solution.

Dissolve 18,61 g of Na_2EDTA in water, dilute to the mark in a 1 000 ml one-mark volumetric flask, and mix. Standardize the solution by taking a known amount of the iron(III) solution (3.12) and, omitting only the extraction step, titrating as in clause 5.

3.11 Zinc, 0,05 mol/l standard volumetric solution.

Dissolve 3,269 g of high purity zinc metal with 25 ml of nitric acid (1 + 1). Expel nitrous oxides by boiling. Cool and adjust to pH 4 to 5 by addition of the hexamethylenetetramine solution (3.9). Dilute to the mark with water in a 1 000 ml one-mark volumetric flask and mix.

3.12 Iron(III), 0,05 mol/l solution.

Dissolve 3,992 g of iron(III) oxide (Fe_2O_3) with 40 ml of the hydrochloric acid solution (3.5). Dilute to the mark with water in a 1 000 ml one-mark volumetric flask and mix.

3.13 Xylenol orange.

Grind 1 g of xylenol orange with 100 g of potassium nitrate.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 pH meter.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0,001 g, 1,000 g of the sample into a 250 ml tall-form beaker.

5.2 Determination

In a cooling bath, dissolve the test portion with 10 ml of the hydrochloric acid (3.1) and 10 ml of the hydrochloric acid solution (3.5), followed by the addition of 10 ml of the hydrogen peroxide solution (3.2) in several small portions. When dissolution is complete, destroy any excess peroxide by boiling gently for 5 min.

Cool the solution to room temperature and transfer to a 150 ml separating funnel. Rinse the beaker with the hydrochloric acid solution (3.5) and add the washings to the separating funnel. The total volume shall not exceed 50 ml.

Add 30 ml of the methyl isobutyl ketone (3.3) and shake vigorously for 1 min. Allow the phases to separate and drain the lower aqueous phase into a second separating funnel. Add 20 ml of the methyl isobutyl ketone (3.3) to the aqueous phase and shake slowly for 1 min. Allow the phases to separate and discard the lower aqueous phase.

Combine the two organic phases and wash by shaking with 20 ml of the lithium chloride solution (3.6) for 30 s. Discard the lower aqueous phase. Repeat the washing with a second portion of 20 ml of the lithium chloride solution.

Discard the aqueous layer and rinse the purified ketone phase with 50 ml of water into a 400 ml beaker. Add 2 ml of the hydrochloric acid solution (3.5) and stir well. To achieve a single-phase system, dilute with 100 ml of the ethanol (3.4) and add, while stiring, as much ammonium fluoride solution (3.7) as is necessary to decolorize the solution.[1)]

Add 5 ml of the thiourea solution (3.8) and 20,00 ml of the Na_2EDTA solution (3.10)[2)]. Adjust the pH of the solution to 4,5 ± 0,1 by the addition of hexamethylenetetramine solution (3.9). Add a spatula-tip (about 0,1 g) of xylenol orange (3.13) and titrate with the zinc solution (3.11) until the indicator turns sharply from yellow to red.

The titration shall be carried out drop by drop and slowly near the end-point.

5.3 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 iron and of composition similar to the material to be analysed, and carrying out the procedure as specified in 5.1 and 5.2.

6 Expression of results

6.1 Calculation

The iron content, expressed as a percentage by mass, is given by the formula

$$\frac{(V_1 \times F - V_2) \times 0{,}002\,793 \times 100}{m} = \frac{(V_1 \times F - V_2) \times 0{,}279\,3}{m}$$

where

V_1 is the volume, in millilitres, of the Na_2EDTA solution (3.10) added;

V_2 is the volume, in millilitres, of the zinc solution (3.11) required for the determination (5.2);

F is the factor of the Na_2EDTA solution, i.e., the concentration of the Na_2EDTA solution established by standardization divided by the nominal concentration, 0,05 mol/l;

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

0,002 793 is the mass, in grams, of iron corresponding to 1 ml of exactly 0,05 mol/l Na_2EDTA solution.

The result should be expressed to two decimal places.

6.2 Repeatability and reproducibility

Comparative tests carried out on two samples by 7 laboratories gave the following statistical data:

Characteristic		Sample 1	Sample 2
Average, % (m/m)		1,92	4,54
Standard deviation	of repeatability, σ_r	0,013	0,019
	of reproductibility, σ_R	0,024	0,038

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

---

1) For alloys with a high tin content, stannic acid may precipitate on the addition of ethanol, but will redissolve on the addition of ammonium fluoride solution. Tin does not affect the determination.

2) The volume of Na_2EDTA solution is sufficient for samples containing up to 5 % (m/m) iron. For samples containing over 5 % (m/m) iron, the volume of Na_2EDTA solution must be increased.