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INTERNATIONAL STANDARD ISO 1812

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Copper alloys — Determination of iron content — 1,10-Phenanthroline spectrophotometric method

Alliages de cuivre — Dosage du fer — Méthode spectrophotométrique à la phénanthroline-1,10

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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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 26, *Copper and copper alloys,* has reviewed ISO Recommendation R 1812-1971 and found it technically suitable for transformation. International Standard ISO 1812 therefore replaces ISO Recommendation R 1812-1971, to which it is technically identical.

ISO Recommendation R 1812 had been approved by the member bodies of the following countries :

Australia
Belgium
Brazil
Canada
Chile
Czechoslovakia
Egypt, Arab Rep. of
Finland
France
Germany
Greece
Hungary
India
Iran
Israel
Italy
Japan
Netherlands
New Zealand
Norway
Peru
Poland
South Africa, Rep. of
Spain
Sweden
Switzerland
Turkey
United Kingdom
U.S.A.
Yugoslavia

No member body had expressed disapproval of the Recommendation.

The member bodies of the following countries disapproved the transformation of the Recommendation into an International Standard :

Hungary
Italy

Printed in Switzerland

Copper alloys — Determination of iron content — 1,10-Phenanthroline spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a 1,10-phenanthroline spectrophotometric method for the determination of the iron content of copper alloys.

The method is applicable to the determination of iron contents up to 0,4 % (*m/m*) in any of the copper alloys listed in ISO publications.

2 REFERENCE

ISO/R 1811, *Chemical analysis of copper and copper alloys — Sampling of copper refinery shapes.*

3 PRINCIPLE

Extraction of the iron from a test portion as the iron(III)-chloro-complex with methyl isobutyl ketone, and spectrophotometric measurement of the iron(II)-1,10-phenanthroline complex at a wavelength corresponding to maximum absorption.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled or deionized water. For the determination of iron contents lower than 0,01 % (*m/m*), use double-distilled water.

4.1 Methyl isobutyl ketone.

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

4.3 Hydrochloric acid, 7 + 3 (*V* + *V*) solution.

Mix 70 ml of hydrochloric acid, ρ approximately 1,19 g/ml, with 30 ml of water.

4.4 Hydrochloric acid, 1 + 1 (*V* + *V*) solution.

Mix 50 ml of hydrochloric acid, ρ approximately 1,19 g/ml, with 50 ml of water.

4.5 Ascorbic acid, 10 g/l solution.

Dissolve 5 g of ascorbic acid in water and dilute to 500 ml.

This solution is stable for 3 or 4 days.

4.6 1,10-Phenanthroline, buffered solution.

Mix 1 g of 1,10-phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2 \cdot HCl \cdot H_2O$) with 215 ml of glacial acetic acid in a 500 ml one-mark volumetric flask and add, while cooling, 265 ml of ammonia solution, ρ 0,91 g/ml. This mixture should have a pH of 6,5 ± 0,1. Adjust, if necessary, by adding either ammonia solution or glacial acetic acid, then dilute to 500 ml.

This solution is stable.

4.7 Iron, stock solution (1 ml $\hat{=}$ 0,1 mg of Fe).

Dissolve 0,1 ± 0,01 g of high purity iron in 20 ml of hydrochloric acid, ρ 1,19 g/ml, and dilute to 1 l.

4.8 Iron, standard solution (1 ml $\hat{=}$ 10 μg of Fe).

Dilute 50 ml of the iron stock solution (4.7) to 500 ml.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Spectrophotometer, fitted with cells of optical path length 2 cm.

NOTE — All glassware shall be rinsed with hot hydrochloric acid solution (4.4) until the surface is free from iron.

6 SAMPLING

Sampling shall be carried out in accordance with the procedure given in ISO/R 1811.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, 5 g of the test sample.

7.2 Blank test

In parallel with the determination, carry out a blank test following the same procedure and using the same quantities of all the reagents as used in the determination but omitting the test portion.

7.3 Determination

7.3.1 Dissolve the test portion (7.1) in 40 ml of the hydrochloric acid solution (4.3) and 40 ml of the hydrogen peroxide solution (4.2) added in small portions, in a beaker. Cool until the violent reaction has ceased. When the test portion is completely dissolved, heat to boiling and continue boiling for approximately 2 min[1] to remove excess hydrogen peroxide and cool.

7.3.1.1 WITH IRON CONTENTS LESS THAN 0,004 % (*m/m*)

Transfer the solution to a 250 ml separating funnel and wash the beaker with the hydrochloric acid solution (4.4).

7.3.1.2 WITH IRON CONTENTS BETWEEN 0,003 AND 0,04 % (*m/m*)

Dilute the solution to 250 ml with the hydrochloric acid solution (4.4) and transfer 25,0 ml to the separating funnel.

7.3.1.3 WITH IRON CONTENTS BETWEEN 0,03 AND 0,4 % (*m/m*)

Dilute the solution to 500 ml with water, transfer 5,0 ml to the separating funnel and add 20 ml of the hydrochloric acid solution (4.4). Allow to stand until any turbidity disappears.

7.3.2 Add 20 ml of the methyl isobutyl ketone (4.1) to the separating funnel and shake for 15 s. Allow the phases to separate, discard the aqueous phase and wash the organic phase three times with 20 ml of the hydrochloric acid solution (4.4) until free of copper. If the phases separate only with difficulty, then the separation can be accelerated by adding about 2 ml of gasoline (boiling point range approximately 40 to 100 °C) to the emulsified phase mixture without any further shaking. Re-extract the iron from the organic phase by shaking vigorously for 20 s, with two successive 10 ml portions of the ascorbic acid solution (4.5).

7.3.3 Transfer the aqueous extracts to a 50 ml one-mark volumetric flask and mix with 5,0 ml of the buffered 1,10-phenanthroline solution (4.6). Dilute to the mark and within 30 min carry out the spectrophotometric measurements of the test and the blank test solutions after having adjusted the spectrophotometer (5.1) to zero absorbance against water at the wavelength of maximum absorption (usually 510 nm, but variations may occur).

7.4 Preparation of calibration curve

By means of a pipette, transfer amounts of the iron standard solution (4.8), from 0 to 20 ml corresponding to 0 to 200 µg of Fe, to a series of 50 ml volumetric flasks. To each add 20 ml of the ascorbic acid solution (4.5) and mix. Allow to stand for 1 min, then add 5 ml of the buffered 1,10-phenanthroline solution (4.6). Dilute to the mark and carry out the spectrophotometric measurements of the standard colorimetric solutions after having adjusted the spectrophotometer (5.1) to zero absorbance against water, as described in 7.3.3, using cells of optical path length 2 cm, and prepare a calibration curve.

8 EXPRESSION OF RESULTS

Convert the spectrophotometric readings of the test and the blank test solutions to micrograms of iron, by means of the calibration curve.

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

$m \times 0{,}000\,02$ [for contents less than 0,004 % (*m/m*)];

$m \times 0{,}000\,2$ [for contents between 0,003 and 0,04 % (*m/m*)];

$m \times 0{,}002$ [for contents between 0,03 and 0,4 % (*m/m*)].

where m is the mass, in micrograms, of iron found in the aliquot portion of the test solution taken, corrected for the blank.

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or in the document to which reference is made, or regarded as optional.

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[1] For dissolving silicon-containing alloys, especially silicon rich, such as copper-silicon alloy, add 50 drops of 40 % (*m/m*) hydrofluoric acid; in this case, use a beaker made of polytetrafluoroethylene or similar material, or a platinum dish. If dissolution is complete, the solution can be transferred to glassware in which all the following steps may be carried out.