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Wrought and cast copper alloys — Determination of copper content — Electrolytic method

Alliages de cuivre corroyés et moulés — Dosage du cuivre — Méthode électrolytique

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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 1554-1971 and found it technically suitable for transformation. International Standard ISO 1554 therefore replaces ISO Recommendation R 1554-1971, to which it is technically identical.

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ISO Recommendation R 1554 had been approved by the member bodies of the following countries :

- Australia
- Belgium
- Canada
- Czechoslovakia
- Egypt, Arab Rep. of
- Finland
- France
- Germany
- Hungary
- India
- Iran
- Israel
- Italy
- Netherlands
- New Zealand
- Norway
- Peru
- Poland
- South Africa, Rep. of
- Spain
- Sweden
- Switzerland
- Thailand
- 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

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Wrought and cast copper alloys — Determination of copper content — Electrolytic method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an electrolytic method for the determination of the copper content of wrought and cast copper-zinc alloys, copper-aluminium alloys and copper-nickel-zinc alloys, as listed in the relevant ISO publications.

2 REFERENCES

ISO 1553, *Unalloyed copper containing not less than 99,90 % of copper — Determination of copper content — Electrolytic method.*

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

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3 PRINCIPLE

Electrolytic determination of the copper content of a fluoroboric-nitric acid solution of a test portion.

4 REAGENTS

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

4.1 Boric acid (H_3BO_3), 40 g/l solution.

4.2 Hydrofluoric acid, 38 to 40 % (m/m) or 48 % (m/m).

4.3 Nitric acid, diluted 1 + 1 $(V + V)$ (ρ approximately 1,2 g/ml).

4.4 Ammonia solution, ρ approximately 0,91 g/ml.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Current source.

For preference, use a 6 V accumulator. If a rectifier is to be used, an additional buffer battery is recommended.

5.2 Electrolysis equipment, the electrodes of which meet the following requirements :

5.2.1 Platinum cathode, Winkler type[1], made preferably from gauze containing approximately 400 meshes per square centimetre (50 meshes per linear inch), woven from wire of diameter approximately 0,20 mm. The cathode shall be stiffened by doubling the gauze for about 3 mm at the top and the bottom of the cylinder or by reinforcing the gauze at the top and bottom with a platinum band or ring. The diameter of the cylinder shall be 30 to 50 mm and the height 40 to 60 mm. The stem shall be made from a platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium, having a diameter of approximately 1,30 mm, flattened and welded the entire length of the gauze. The overall height of the cathode shall be approximately 130 mm. The cathode shall be sandblasted.

5.2.2 Spiral anode, made of platinum alloy wire of minimum diameter 1 mm, formed into a spiral of seven turns, having a height of approximately 50 mm and a diameter of 12 mm, the overall height being approximately 130 mm. The spiral section shall be sandblasted.

In the case of an analysis of alloys containing lead, gauze anodes shall be used.

5.3 Electric oven, capable of being controlled at 110 °C.

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 0,000 1 g, 2,5 g of the test sample, preferably in the form of fine drillings.

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[1] Platinum cathodes formed from plain or perforated sheets may also be used.

7.2 Determination

7.2.1 Transfer the test portion (7.1) to a 400 ml tall-form beaker, then add 15 ml of the boric acid solution (4.1), 2 ml of the hydrofluoric acid (4.2) and 30 ml of the nitric acid solution (4.3), cover with a watch glass and allow to stand for a few minutes until the reaction has nearly ceased.

7.2.2 Wash the watch glass and the walls of the beaker, heat the uncovered beaker at a temperature of about 90 °C and maintain the solution at this temperature for about 1 h to completely expel the oxides of nitrogen. Cool to ambient temperature and dilute the solution with 50 ml of cold water. Neutralize with the ammonia solution (4.4) until a precipitate appears, and reacidify with the nitric acid solution until the precipitate is dissolved. Add 20 ml of the nitric acid solution in excess and dilute the solution with water to 300 ml.

7.2.3 Insert the electrodes (5.2.1 and 5.2.2) in the solution and cover the beaker with two halves of a watch glass, one of which has two indentations through which the electrode stems may pass. Electrolyse, without stirring, at a current density of about 0,6 A per square decimetre of cathode surface. When the solution becomes colourless, reduce the current density to about 0,3 A/dm^2 and wash the watch glasses, electrode stems, and walls of the beaker. Continue the electrolysis until the deposition of the copper is complete, as indicated by failure to plate on an unplated surface of the electrode stem when the level of the solution is raised.

7.2.4 Without switching off the current, quickly replace the beaker by another of the same size containing approximately 350 ml of distilled water. Continue the electrolysis for 15 min. Remove the cathode and dip it in ethanol or methanol. Dry in the oven (5.3), maintained at 110 °C, for 3 to 5 min and cool. Determine the mass of the deposit of metallic copper.

7.2.5 Verify the absence of copper in the electrolyte by a colorimetric test (see annex B of ISO 1553).

8 EXPRESSION OF RESULTS

8.1 Method of calculation

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

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

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in grams, of copper deposited.

8.2 Repeatability

The determination shall be carried out twice, and the results obtained shall agree within 0,07 % (m/m) of copper.

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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 documents to which reference is made, or regarded as optional.