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Zinc alloys — Determination of copper content — Electrolytic method

Alliages de zinc — 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 now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 18 has reviewed ISO Recommendation R 1976 and found it technically suitable for transformation. International Standard ISO 1976 therefore replaces ISO Recommendation R 1976-1971 to which it is technically identical.

ISO Recommendation R 1976 was approved by the Member Bodies of the following countries :

Australia	Germany	South Africa, Rep. of
Belgium	Greece	Sweden
Canada	India	Thailand
Chile	Italy	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.A.
France	Norway	U.S.S.R.

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1976 into an International Standard.

Zinc 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 zinc alloys.

The method is applicable to zinc alloy Zn Al4 Cu1 defined in ISO/R 301.

It is suitable for the determination of copper contents between 0,5 and 3,5 %.

2 REFERENCES

ISO/R 301, *Zinc alloy ingots.*

ISO 3752, *Zinc alloy ingots — Selection and preparation of samples for chemical analysis.*¹⁾

3 PRINCIPLE

Determination of copper by electrolysis in nitric sulphuric acid medium.

4 REAGENTS

During the analysis, use only reagents of analytical reagent grade and distilled or demineralized water.

4.1 Nitric acid, ρ 1,4 g/ml.

4.2 Ammonia solution, ρ 0,91 g/ml.

4.3 Sulphuric acid diluted 1 + 1 (approximately 18 N solution).

4.4 Ethanol, approximately 95 % (V/V) (concentration of the azeotrope water-ethanol).

5 APPARATUS

5.1 Electrolyser.

5.2 Stirrer (mechanical or magnetic stirrer or rotating anode).

5.3 Gauze electrodes, made of platinum or platinum hardened by alloying with a metal of the same group.

6 SAMPLING

Sampling shall be carried out in accordance with the requirements of ISO 3752.

7 PROCEDURE

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

7.2 Introduce the test portion into a 400 ml beaker.

7.3 Add 20 ml of water. Cover the beaker with a watch-glass and attack with 20 ml of the nitric acid (4.1), added carefully and in small portions. Cool in water if the reaction is too violent. When the dissolution is complete, boil to expel nitrous fumes.

7.4 Dilute with water to 200 ml. Heat to gentle boiling. Neutralize, while stirring constantly, with the ammonia solution (4.2) added drop by drop until there is a slight permanent precipitate of aluminium hydroxide.

7.5 Add 2 ml of the nitric acid (4.1) and 4 ml of the sulphuric acid solution (4.3). Dilute with water to 300 ml approximately. Cool to room temperature.

7.6 Weigh, to the nearest 0,000 1 g, the cathode (5.3) previously cleaned in concentrated nitric acid, rinsed in water and in ethanol and dried between 105 and 110 °C for 3 to 5 min.

7.7 Insert the electrodes (5.3) in the electrolyser (5.1) and place the beaker in position. Add water until the electrodes are completely immersed. Cover with a suitable split cover-glass.

7.8 Electrolyse, at a current density of about 2 A/dm², while stirring.

1) At present at the stage of draft.

7.9 After about 30 min, rinse the split cover-glass and the walls of the beaker with a jet of water and continue the electrolysis until the deposition of copper is complete, as indicated by the absence of a pink coloration on the freshly immersed surface of the stem of the cathode.

7.10 Reduce the current density to 0,5 A/dm², remove the beaker and replace it immediately by another one of equal or larger dimensions filled with acidulated water (approximately 1 % nitric acid (4.1)). After a few seconds of immersion, repeat the operation with a third beaker filled with water.

7.11 Remove the beaker and switch off the current. Remove the cathode and rinse it carefully with water. Dip it successively in two beakers containing ethanol (4.4).

7.12 Dry in an oven between 105 and 110 °C for 3 to 5 min. Allow to cool.

7.13 Weigh the copper-plated cathode to the nearest 0,000 1 g.

8 EXPRESSION OF RESULTS

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

$$(m_2 - m_1) \times 20$$

where

m_1 is the mass, in grams, of the cathode before electrolysis;

m_2 is the mass, in grams, of the cathode after electrolysis.

9 TEST REPORT

The test report shall mention the method used and the results obtained. It shall also mention all operational details not provided for in this International Standard, or any optional details, as well as any circumstances which could have influenced the results.

The test report shall include all details required for complete identification of the sample.

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