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ISO

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ISO RECOMMENDATION

R 1553

CHEMICAL ANALYSIS OF COPPER AND COPPER ALLOYS

ELECTROLYTIC DETERMINATION OF COPPER IN UNALLOYED COPPER CONTAINING NOT LESS THAN 99.90 % OF COPPER

1st EDITION

August 1971

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 1553, *Chemical analysis of copper and copper alloys – Electrolytic determination of copper in unalloyed copper containing not less than 99.90 %*, was drawn up by Technical Committee ISO/TC 26, *Copper and copper alloys*, the Secretariat of which is held by the Deutscher Normenausschuss (DNA).

Work on this question led to the adoption of Draft ISO Recommendation No. 1553, which was circulated to all the ISO Member Bodies for enquiry in March 1968.

The Draft was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1553 August 1971

CHEMICAL ANALYSIS OF COPPER AND COPPER ALLOYS

ELECTROLYTIC DETERMINATION OF COPPER IN UNALLOYED COPPER CONTAINING NOT LESS THAN 99.90 % OF COPPER

1. SCOPE

This ISO Recommendation describes an electrolytic method for the determination of copper in unalloyed copper containing not less than 99.90 % of copper. Silver, if present, is deposited with the copper and is reported as copper.

The copper should not contain elements which leave insoluble residues in the solution for electrolysis, and should be low in content of impurities which can be deposited electrolytically together with the copper.

2. PRINCIPLE

Electrolytic determination of copper from sulphuric/nitric acid solution.

3. REAGENTS

All the reagents should be of the analytical grade. Use distilled or de-ionized water.

3.1 *Sulphuric/nitric acid* solution, prepared as follows :

Add slowly, while stirring, 300 ml of sulphuric acid (H_2SO_4), d = 1.83 to 750 ml of water. Cool, and add 210 ml of nitric acid (HNO_3), d = 1.38.

4. APPARATUS

Ordinary laboratory and electrolysis equipment.

NOTE. – Preference should be given to a 6 V accumulator as current source. If a rectifier is to be used, an additional buffer battery is recommended.

4.1 *Cathode.* It is recommended that a platinum cathode (Winkler electrode)* be used. It should be made preferably from gauze containing approximately 400 meshes per square centimetre (50 meshes per linear inch). Gauze for cathodes should be woven from wire of approximately 0.20 mm diameter. The cathode should 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 cylinder should be approximately 30 to 50 mm in diameter and 40 to 60 mm in height. The stem should be made from a platinum alloy wire, such as platinum-iridium, platinum-rhodium, or platinum-ruthenium, having a diameter of approximately 1.30 mm. It should be flattened and welded the entire length of the gauze. The overall height of the cathode should be approximately 130 mm. The cathode should be sandblasted.

4.2 *Anode.* It is recommended that a spiral anode be used. It should be made from 1 mm diameter or larger platinum alloy wire, 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 should be sandblasted.

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* Platinum cathodes formed from plain or perforated sheets may also be used.

5. SAMPLING

Follow the procedure given in ISO Recommendation R 1811, *Chemical analysis of copper and copper alloys – Sampling of copper refinery shapes.*

6. PROCEDURE

6.1 Test portion

$m_0 = 5 \pm 0.001$ g of the test sample (see Annex A).

6.2 Determination

6.2.1 Transfer the test portion to a 400 ml beaker provided with a close-fitting cover glass. Add 45 ml of the sulphuric/nitric acid mixture (3.1) and allow to stand for a few minutes until the reaction has nearly ceased. Heat at a temperature of 80 to 90 °C to complete dissolution and maintain the solution at this temperature for 2 to 3 hours to expel the nitrous oxides quantitatively. Wash the cover and sides of the beaker, and dilute the solution to 300 ml.

6.2.2 Insert the electrodes 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. Electrolyze, without stirring, at a current density of about 0.6 A/dm^2 of cathode surface. When the solution becomes colourless, reduce the current density to about 0.3 A/dm^2 and wash the cover glasses, electrode stems, and sides of the beaker. Continue the electrolysis until the deposition of the copper is complete, as indicated by failure to plate on a new surface of the electrode stem when the level of the solution is raised.

6.2.3 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 minutes. Remove the cathode and dip it in ethanol or methanol. Dry in an oven at 110 °C for 3 to 5 minutes and cool. Weigh the deposit of metallic copper as indicated in Annex A.

6.2.4 The absence of copper in the electrolyte can be proved by a colorimetric test; if the determined content of copper is less than or equal to the prescribed figure, the residual copper in the electrolyte should be determined in accordance with the method given in Annex B.

7. EXPRESSION OF RESULTS

7.1 Method of calculation and formula

Calculate the percentage of copper as follows :

$$\text{Copper, \%} = \frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grammes, of the test portion;

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

7.2 Repeatability

The determination should be carried out twice, and the results obtained should agree within 0.015 % of copper.

8. TEST REPORT

The test report should 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 ISO Recommendation or regarded as optional.

ANNEX A

WEIGHING OF TEST PORTION

A.1 When a two-pan balance is used, weigh in the following manner :

Using a marked 5 g weight, weigh 5.0050 to 5.0070 g of the test sample. Weigh the cathode, placing the marked 5 g weight used in weighing the test sample on the balance-pan with the cathode, and noting the other weights used. Deduct 5 g from the observed mass to obtain the mass of the cathode.

Weigh the cathode with the copper deposit, using the same weights as were used in weighing the cathode before the electrolysis, except that the marked 5 g weight used on the pan with the cathode shall be omitted.

A.2 When a single-pan balance is used, weigh in the following manner :

First weigh the cathode alone and then, without removing the cathode, weigh accurately about 5 g of the test sample. In subsequent weighing of the cathode with the copper deposit the same weights should be used.

The mass of the test portion is the difference between the masses of the cathode plus the sample and the cathode alone.

The mass of the copper deposit is the difference between the masses of the cathode with the deposit and the cathode alone.

ANNEX B

SPECTROPHOTOMETRIC DETERMINATION OF RESIDUAL COPPER IN THE ELECTROLYTE

B.1 SCOPE

This Annex describes a method for the spectrophotometric determination of copper.

The method is applicable to the determination of residual copper in the electrolyte after the deposition of copper on the cathode.

B.2 PRINCIPLE

Formation of the copper oxalyldihydrazide complex at pH 8.8 in the presence of acetaldehyde.

Spectrophotometry.

B.3 REAGENTS

B.3.1 *Ammonia* solution (d = 0.91).

B.3.2 *Sulphuric/nitric acid* solution, prepared as follows :

Slowly pour 300 ml of sulphuric acid (d = 1.83), while stirring, into 750 ml of water. Cool and add 210 ml of nitric acid (d = 1.38).

B.3.3 *Citric acid,* 200 g/l solution.

B.3.4 *Acetaldehyde,* 40 % solution in water or methanol.

B.3.5 *Oxalyldihydrazide,* 2.50 g/l solution.

Dissolve the oxalyldihydrazide in distilled water, heating gently.

B.3.6 *Copper* standard solution, 0.01 g/l.

In a 250 ml beaker dissolve 1 g of electrolytic copper in 10 ml of nitric acid (d = 1.38) diluted with an equal volume of water. Evaporate almost to dryness to remove the excess acid. Add 50 ml of water to dissolve the residue. Transfer to a 1000 ml one-mark volumetric flask, rinse the beaker and make up to the mark with water. Transfer 10 ml of this solution to a 1000 ml one-mark volumetric flask and dilute to the mark.

1 ml of this solution contains 0.010 mg of copper.

B.4 APPARATUS

Ordinary laboratory equipment and

B.4.1 *Spectrophotometer* (wavelength approximately 540 nm).