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INTERNATIONAL STANDARD



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Copper and copper alloys — Determination of arsenic — Photometric method

Cuivre et alliages de cuivre — Dosage de l'arsenic — Méthode photométrique

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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.

International Standard ISO 3220 was drawn up by Technical Committee ISO/TC 26, *Copper and copper alloys*, and circulated to the Member Bodies in June 1973.

It has been approved by the Member Bodies of the following countries :

Australia	Germany	Sweden
Austria	Ireland	Switzerland
Belgium	Japan	Thailand
Bulgaria	Mexico	Turkey
Canada	New Zealand	United Kingdom
Chile	Norway	U.S.A.
Egypt, Arab Rep. of	Poland	Yugoslavia
Finland	Romania	
France	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

Copper and copper alloys — Determination of arsenic — Photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of arsenic in copper and copper alloys.

The method is applicable for the determination of arsenic as an alloying element or as an impurity in copper and all types of copper alloys listed in ISO Recommendations or International Standards.

2 PRINCIPLE

Extraction of arsenic from a hydrochloric, hydrobromic and perchloric acid-containing solution using benzene. After re-extraction with water, photometric determination as molybdenum blue.

Phosphorus and silicon in milligram amounts do not interfere.

3 REAGENTS

All the reagents shall be of analytical grade. Distilled or deionized water shall be used.

3.1 Benzene.

3.2 Hydrochloric acid ρ 1,19 g/ml.

3.3 Hydrobromic acid ρ 1,5 g/ml.

3.4 Perchloric acid ρ 1,67 g/ml.

3.5 Chloric acid.

3.6 Hydrochloric acid, 6 N.

Dilute 100 ml of hydrochloric acid (ρ 1,19 g/ml) with 100 ml of water.

3.7 Sulphuric acid solution.

Dilute 95 ml of sulphuric acid (ρ 1,84 g/ml) with water to 2 000 ml.

3.8 Ascorbic acid solution.

Dissolve 2 g of ascorbic acid in 100 ml of water; use freshly prepared solutions.

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

3.10 Potassium permanganate, 0,1 N solution.

3.11 Molybdate solution.

Dissolve 0,95 g of ammonium heptamolybdate, $[(NH_4)_6Mo_7O_{24} \cdot 4H_2O]$, in 200 ml of the sulphuric acid (3.7); use freshly prepared solutions.

3.12 Arsenic stock solution.

Dissolve 132 mg of arsenious oxide (As_2O_3) with 10 ml of sodium hydroxide, (NaOH), 1 N, in a polyethylene beaker, add 12,5 ml of hydrochloric acid (HCl), 1 N, and dilute in a 250 ml volumetric flask with water to the mark.

3.13 Arsenic standard solution.

Add to 25 ml of the arsenic stock solution (3.12) in a 500 ml volumetric flask 20 ml of the hydrochloric acid (3.6) and dilute with water to the mark. (1 ml is equivalent to 20 μ g of arsenic.)

4 APPARATUS

4.1 Normal laboratory apparatus

4.2 Photometer.

5 SAMPLING

Carry out the sampling in accordance with the requirements of ISO . . . 1)

6 PROCEDURE

Test portion mass for arsenic contents up to 0,02 % : 2,00 g.

1) In preparation.

Test portion mass for arsenic contents between 0,02 and 0,8 % : 0,500 g.

Dissolve the test portion in a 100 ml Erlenmeyer flask with 20 ml of the hydrochloric acid (3.6) by adding a total amount of 10 ml of the hydrogen peroxide solution (3.9) in several small portions. Cool the solution during this operation. Complete the dissolution by heating gently.

In the case of arsenic contents less than 0,02 % (test portion mass : 2 g) evaporate the solution to approximately 3 ml; in the case of arsenic contents between 0,02 and 0,08 % (test portion mass : 0,5 g) evaporate the solution to approximately 5 ml.

If arsenic contents between 0,08 and 0,8 % are to be expected (test portion mass : 0,5 g) transfer the solution into a 50 ml volumetric flask and dilute with water to the mark. If arsenic contents between 0,08 and 0,4 % are present take an aliquot of 10 ml; if arsenic contents between 0,4 and 0,8 % are present take an aliquot of 5 ml and transfer it into a 100 ml beaker. In these last two cases, add 1 ml of the hydrogen peroxide solution (3.9) or 1 ml of the chloric acid (3.5) and evaporate to approximately 3 ml.

After cooling to room temperature transfer the evaporated solution with 20 ± 2 ml of the hydrochloric acid (3.2) into a 100 ml separating funnel, which contains 5 ml of the hydrobromic acid (3.3). After the addition of 10 ml of the perchloric acid (3.4) extract the arsenic present with 25 ml of the benzene (3.1) by shaking vigorously for 1 min. After separation of the phases, draw off the aqueous phase and discard it.

Wash the benzene phase by shaking with 10 ml of the hydrochloric acid (3.2) for 30 s and again discard the aqueous phase obtained.

Re-extract the arsenic from the benzene phase by shaking it vigorously with 25 ml of water for 1 min. Draw off the aqueous phase into a 100 ml volumetric flask, add 1 ml of the potassium permanganate solution (3.10) and heat to approximately 60°C (oxidation of the arsenic).

After cooling add 10 ml of the molybdate solution (3.11) and 10 ml of the ascorbic acid solution (3.8), swirling after each addition. Heat to boiling and hold at this temperature for $2 \text{ min} \pm 15 \text{ s}$. After cooling to approximately 20°C dilute with water to the mark and mix well. Measure the blue-coloured solution obtained with the photometer (4.2) at a wavelength of 840 nm (maximum absorption) using 2 cm cells if up to 150 μg of arsenic are present.

If arsenic amounts between approximately 150 and 300 μg are present, use 1 cm cells and measure at the same wavelength. If the amount of arsenic exceeds 300 μg , the solution may be measured at 578 nm (mercury line) using 1 cm cells. Use water in the comparison path. The colour of the solution is stable for several hours.

Determine the blank of all the reagents used under the same condition as for the analysis itself and correct the measured result.

Prepare the calibration graph as follows :

Dissolve 5,00 g of arsenic-free copper in a 400 ml beaker with 50 ml of the hydrochloric acid (3.6) by addition of a total amount of 25 ml of the hydrogen peroxide solution (3.9) as described above. After cooling transfer the solution obtained into a 100 ml volumetric flask and dilute with water to the mark.

Pipette 5 ml of this solution into each of six 100 ml beakers and add respectively 0, 2, 4, 8, 15 and 20 ml of the arsenic standard solution (3.13) containing respectively 0, 40, 80, 160, 300 and 400 μg of arsenic. Add to each 1 ml of the hydrogen peroxide solution (3.9) or 1 ml of the chloric acid (3.5) and evaporate to approximately 3 ml.

Follow the procedure given above and measure the colour depth according to the three given conditions :

- 840 nm, 2 cm cells;
- 840 nm, 1 cm cells;
- 578 nm, 1 cm cells.

Each calibration graph obtained is a straight line.

7 EXPRESSION OF RESULTS

Read from the relevant calibration graph the arsenic value in micrograms corresponding to the measured extinction value and calculate the arsenic content, as a percentage by mass, as follows :

$$\text{for the 2 g test portion : As \% (m/m) = } \frac{m}{20\,000}$$

$$\text{for the 10 ml aliquot of the 0,5 g test portion : } \text{As \% (m/m) = } \frac{m}{1\,000}$$

$$\text{for the 5 ml aliquot of the 0,5 g test portion : } \text{As \% (m/m) = } \frac{m}{500}$$

where m is the mass, in micrograms, of arsenic.

8 TEST REPORT

The test report shall include the following particulars :

- the reference of the method used;
- the results and the method of expression used;
- any characteristics noted during the determination;
- any operation not included or regarded as optional.