International Standard 4742

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Copper alloys — Determination of nickel content — Gravimetric method

Alliages de cuivre — Dosage du nickel — Méthode gravimétrique

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Foreword

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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.

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It has been approved by the member bodies of the following countries :

Australia	Germany, F.R.	Poland
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Bulgaria	Italy	Spain
Canada	Japan	Sweden
China	Korea, Dem. P. Rep. of	Switzerland
Czechoslovakia	Korea, Rep. of	Turkey
Egypt, Arab Rep. of	Netherlands	USA
France	Norway	USSR

No member body expressed disapproval of the document.

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Copper alloys — Determination of nickel content — Gravimetric method

WARNING : Throughout this International Standard, normal precautions regarding the use of perchloric acid in laboratory work should be observed.

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of the nickel content in all types of copper alloys listed in International Standards.

The method is applicable to the determination of nickel contents between 2 and 50 % (m/m).

2 Reference

ISO 1554, *Wrought and cast copper alloys — Determination of copper content — Electrolytic method.*

3 Principle

Dissolution of a test portion in nitric acid and removal of tin and silicon, if present. Separation of copper by electrolysis and precipitation of nickel from the copper-free electrolysate by the sodium salt of dimethylglyoxime in the presence of citric acid. Separation of the precipitate by filtration.

4 Reagents

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

4.1 Nitric acid, ϱ 1,40 g/ml.

4.2 Perchloric acid, ϱ 1,67 g/ml.

4.3 Hydrobromic acid, ϱ 1,38 g/ml.

4.4 Ammonia solution, ϱ 0,925 g/ml.

4.5 Nitric acid solution, 1 + 1.

Dilute 100 ml of the nitric acid (4.1) with 100 ml of water.

4.6 Sulfamic acid, 100 g/l solution.

4.7 Citric acid, 250 g/l solution.

4.8 Sodium dimethylglyoximate, 25,9 g/l solution.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Beakers, electrolytic, capacity 300 to 400 ml.

5.2 Electrolysis equipment, including current source and platinum electrodes, as specified in ISO 1554.

5.3 Filter crucible, of fritted glass, pore size 16 to 40 µm.

6 Procedure

6.1 Test portion

6.1.1 Nickel content between 2 and 4,25 % (m/m)

Weigh, to the nearest 0,000 1 g, about 2 g of the test sample. The nickel content of the test portion will be 40 to 85 mg.

6.1.2 Nickel content between 4 and 8,5 % (m/m)

Weigh, to the nearest 0,000 1 g, about 1 g of the test sample. The nickel content of the test portion will be 40 to 85 mg.

6.1.3 Nickel content between 8 and 50 % (m/m)

Weigh, to the nearest 0,000 1 g, between 0,25 and 1 g of the test sample so that the nickel content of the test portion will be 80 to 125 mg.

6.2 Dissolution of test portion

6.2.1 Test portions free of tin and silicon

Transfer the test portion (6.1) to a 250 ml beaker. Add 25 ml of the nitric acid solution (4.5). Dissolve first at ambient temperature, then heat gently until dissolution is complete.

When the test portion has completely dissolved, increase the temperature and allow the solution to boil for several minutes to expel the oxides of nitrogen. Remove from heat and add 50 ml of water. If the test solution is clear, transfer to an electrolytic beaker (5.1) and proceed as specified in 6.3.

6.2.2 Test portions containing tin and silicon

If the tin content is sufficiently high, the test solution (6.2.1) will be cloudy. In this case, let the test solution stand for 1 h at 80 °C to flocculate tin oxide. Remove from heat, add filter pulp, and filter through a close-textured filter paper. Collect the filtrate in an electrolytic beaker (5.1). Wash the precipitate several times with warm nitric acid solution (diluted 1 + 99), adding the washings to the filtrate.

Transfer the filter paper and precipitate to the original beaker. Add 15 to 20 ml of the nitric acid (4.1) and 10 to 15 ml of the perchloric acid (4.2). Cover and heat to the evolution of copious white fumes. Continue heating until all organic matter is destroyed. Cool, rinse the cover and sides of the beaker, and add 15 ml of hydrobromic acid (4.3). Heat to the evolution of copious white fumes to volatilize tin. Repeat the hydrobromic acid addition and heating until the solution is clear, then evaporate the solution to near dryness. Cool, dissolve the residue in a small volume of water, and add the solution to the original filtrate in the electrolytic beaker.

6.3 Electrolysis

Add 5 ml of the sulfamic acid solution (4.6) to the test solution (6.2) and dilute to about 200 ml with water. Connect the electrolysis apparatus (5.2), introduce the platinum electrodes to the test solution, and electrolyse as specified in ISO 1554. The current density may be increased by stirring the electrolyte vigorously. When deposition of copper is complete, remove the electrodes and reserve the electrolysate.

6.4 Precipitation

6.4.1 Add 5 ml of the nitric acid (4.1) and 10 ml of the perchloric acid (4.2) to the electrolysate and evaporate the solution to the evolution of copious white fumes. Allow to cool and add 100 ml of water. Transfer to an 800 ml beaker, filtering if necessary. Add 10 ml of the citric acid solution (4.7), then add the ammonia solution (4.4) until the test solution turns blue. Add 1 ml in excess. Dilute to 400 ml and heat to 60 to 70 °C.

6.4.2 In the case of nickel contents of 40 to 85 mg (6.1.1, 6.1.2), add 44 ml of the sodium dimethylglyoximate solution (4.8) to the test solution (6.4), stirring vigorously. Allow the test solution to cool to ambient temperature, with occasional stirring.

6.4.3 In the case of nickel contents of 80 to 125 mg (6.1.3), add 60 ml of the sodium dimethylglyoximate solution (4.8) to the test solution (6.4), stirring vigorously. Allow the test solution to cool to ambient temperature, with occasional stirring.

6.5 Filtration

Filter the precipitate on a tared, medium-porosity fritted glass filter crucible (5.3), previously dried for 1 h at 150 °C and allowed to cool in a desiccator. Wash the precipitate 10 to 12 times with small amounts of water, allowing the filter to drain completely between washings. Dry the precipitate for 1 h at 150 °C. Allow to cool in a desiccator and weigh.

7 Expression of results

The nickel content, expressed as a percentage by mass, in given by the formula

$$\frac{m_1}{m_0} \times 20{,}32$$

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in grams, of the precipitate (6.5).

8 Test report

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

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