G-24-01

INTERNATIONAL STANDARD 1810

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION •МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Copper alloys — Determination of nickel (low contents) — Dimethylglyoxime spectrophotometric method

Alliages de cuivre — Dosage du nickel (faibles teneurs) — Méthode spectrophotométrique à la diméthylglyoxime

First edition — 1976-11-15

UDC 669.3 : 546.74 : 543.42

Ref. No. ISO 1810-1976 (E)

Descriptors : copper alloys, chemical analysis, determination of content, nickel, spectrophotometric analysis.

Price based on 2 pages

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 1810-1971 and found it technically suitable for transformation. International Standard ISO 1810 therefore replaces ISO Recommendation R 1810-1971, to which it is technically identical.

ISO Recommendation R 1810 had been approved by the member bodies of the following countries :

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

Printed in Switzerland

Copper alloys — Determination of nickel (low contents) — Dimethylglyoxime spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a dimethylglyoxime spectrophotometric method for the determination of the nickel content of copper alloys.

The method is applicable to the determination of nickel contents up to 2,5 % (*m/m*) in any of the copper alloys listed in ISO publications.

2 REFERENCE

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

3 PRINCIPLE

Extraction of the nickel in a test portion as a yellow-coloured dimethylglyoxime complex with chloroform, and spectrophotometric measurement of the complex at a wavelength of approximately 405 nm.

4 REAGENTS

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

4.1 Chloroform.

4.2 Sodium sulphate, anhydrous.

4.3 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted 1 + 1 (*V* + *V*).

4.4 Hydrogen peroxide, 30 % (*m/m*) solution.

4.5 Hydroxylammonium chloride ($HONH_3CL$), 10 g/l solution, adjusted to pH 7 with ammonia solution.

4.6 Complexing solution.

Mix 240 ml of sodium tartrate solution (100 g/l), 90 ml of sodium hydroxide solution (40 g/l), 480 ml of sodium acetate trihydrate ($CH_3{\cdot}COONa{\cdot}3H_2O$) solution (200 g/l) and 200 ml of sodium thiosulphate pentahydrate ($Na_2S_2O_3{\cdot}5H_2O$) solution (200 g/l).

4.7 Dimethylglyoxime, 10 g/l solution in methanol.

Dissolve 10 g of dimethylglyoxime [$(CH_3CNOH)_2$] in methanol and dilute to 1 l with methanol.

4.8 Nickel, 1 g/l standard solution.

Dissolve 1,000 g of nickel (nickel content at least 99,8 %) in 10 ml of nitric acid, ρ 1,38 g/ml. Remove the oxides of nitrogen by boiling and evaporate the solution to syrupy consistency. Cool and dilute to 1 l.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Spectrophotometer, fitted with cells of optical path length 2 and 4 cm.

6 SAMPLING

Sampling shall be carried out in accordance with the procedure given in ISO/R 1811.

7 PROCEDURE

7.1 Test portion

7.1.1 For expected nickel contents up to 0,5 % (*m/m*), weigh, to the nearest 0,001 g, 1 g of the test sample.

7.1.2 For expected nickel contents between 0,5 and 2,5 % (*m/m*), weigh, to the nearest 0,001 g, 0,4 g of the test sample, and add 0,6 ± 0,001 g of nickel-free pure copper.

7.2 Blank test

In parallel with the determination, carry out a blank test following the same procedure and using the same quantities of all the reagents as used in the determination but omitting the test portion.

7.3 Determination

7.3.1 Dissolve the test portion (7.1) in 20 ml of the hydrochloric acid (4.3) and 10 ml of the hydrogen peroxide solution (4.4) added in small portions. Cool until the violent reaction has ceased. When the test portion is completely dissolved, heat the solution to boiling and continue boiling for approximately 1 min to remove the excess hydrogen peroxide, then cool to room temperature. Transfer the solution to a 500 ml volumetric flask, dilute to the mark and mix.

7.3.2 By means of a pipette, transfer 25 ml of the solution, or 10 ml if the expected nickel content is between 1,5 and 2,5 % (*m/m*), into a 250 ml separating funnel. Add 5 ml of the hydroxylammonium chloride solution (4.5) and 50 ml of the complexing solution (4.6), shaking after each addition. Check with indicator paper that the pH of the solution is between 6,5 and 7,2 and adjust, if necessary, with 1 N hydrochloric acid solution or 1 N sodium hydroxide solution. Add 3 ml of the dimethylglyoxime solution (4.7) and shake for 60 s. Then transfer, by means of a pipette, 20 ml of a the chloroform (4.1) to this solution and shake again for 40 s. Allow the phases to separate.

7.3.3 Transfer the yellow-coloured chloroform solution to a 25 ml conical flask fitted with a ground glass stopper and containing 1 g of the anhydrous sodium sulphate (4.2). Shake the stoppered flask to remove the water from the chloroform, transfer the chloroform solution to a cell of optical path length 2 cm and cover immediately.

7.3.4 Carry out the spectrophotometric measurements of the test and the blank test solutions at a wavelength of approximately 405 nm after having adjusted the spectrophotometer (5.1) to zero absorbance against the chloroform (4.1).

7.3.5 If the expected nickel content is too low for accurate measurement, use a cell of optical path length 4 cm or measure at a wavelength of approximately 365 nm.

7.4 Preparation of calibration curve

By means of a pipette, transfer 100 ml of the standard nickel solution (4.8) to a 500 ml one-mark volumetric flask. Dilute to the mark and mix. Transfer to a series of six 500 ml one-mark volumetric flasks 0, 5, 10, 20, 30 and 50 ml of this solution, diluted with liquors containing 1 g of nickel-free copper, dissolved under the conditions indicated in 7.3.1. Dilute to the mark and mix.

By means of a pipette, transfer 25 ml of the mixtures containing 0 to 30 ml of the standard nickel solution (nickel content less than 1,5 % (*m/m*)), 10 ml of the mixtures containing 30 or 50 ml of the standard nickel solution (nickel content between 1,5 and 2,5 % (*m/m*)) to a 250 ml separating funnel. Add 5 ml of the hydroxylammonium chloride solution (4.5) and 50 ml of the complexing solution (4.6), shaking after each addition, and then proceed from the appropriate point of 7.3.2. Carry out the spectrophotometric measurements of the standard colorimetric solutions at the two wavelengths given in 7.3 after having adjusted the spectrophotometer (5.1) to zero absorbance against the chloroform (4.1), and prepare the calibration curve.

8 EXPRESSION OF RESULTS

Convert the spectrophotometric readings of the test and the blank test solutions to milligrams of nickel by means of the appropriate calibration curve.

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

$$\frac{m_1}{10\, m_0}$$

where

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

m_1 is the mass, in milligrams, of nickel found in the aliquot portion of the test solution corrected for blank.

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