International Standard 4740

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Copper and copper alloys — Determination of zinc content — Flame atomic absorption spectrometric method

Cuivre et alliages de cuivre — Dosage du zinc — Méthode par spectrométrie d'absorption atomique dans la flamme

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Foreword

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INTERNATIONAL STANDARD ISO 4740-1985 (E)

Copper and copper alloys — Determination of zinc content — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the zinc content of all types of copper and copper alloys excluding copper alloys containing more than 10 % (*m/m*) of lead.

The method is applicable to the determination of zinc contents between 0,001 and 6 % (*m/m*).

2 Reference

ISO/R 1811, *Chemical analysis of copper and copper alloys — Sampling of copper refinery shapes.* [1)]

3 Principle

Dissolution of a test portion in fluoroboric-nitric acid, followed by aspiration into the air/acetylene flame of a flame atomic absorption spectrometer. Comparison of the absorption of the resonance energy of zinc at 213,8 nm with those of standard matching solutions of zinc.

4 Reagents

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

4.1 Fluoroboric-nitric acid, attack solution.

Mix together 300 ml of boric acid solution (40 g/l), 30 ml of hydrofluoric acid [40 % (*V/V*)], 500 ml of nitric acid (ϱ 1,40 g/ml) and 150 ml of water.

4.2 Copper, base solution

Weigh 10,0 g of copper containing not more than 0,000 2 % (*m/m*) zinc into a 1 000 ml PTFE beaker. Add 400 ml of the attack solution (3.1) and warm until the copper is dissolved. Boil the solution until all brown fumes have been expelled. Cool and transfer the solution to a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

50 ml of this solution contains 1 g of copper and 40 ml of attack solution (4.1).

4.3 Zinc, standard stock solution, corresponding to 5 g of Zn per litre.

Transfer 2,5 ± 0,000 1 g of zinc metal (99,99 % purity) to a 250 ml tall-form beaker. Add 50 ml of nitric acid solution (ϱ 1,40 g/ml, diluted 1 + 1), cover and heat gently until the metal is dissolved. Boil the solution for several minutes to expel nitrous fumes, then cool. Transfer the solution to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 5 mg of Zn.

4.4 Zinc, standard solution, corresponding to 0,5 g of Zn per litre.

Transfer 100,0 ml of the zinc standard solution (3.3) to a 1 000 ml one-mark volumetric flask.

Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,5 mg of Zn.

4.5 Zinc, standard solution, corresponding to 0,05 g of Zn per litre.

Transfer 10,0 ml of the zinc standard stock solution (3.3) to a 1 000 ml one-mark volumetric flask.

Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,05 mg of Zn.

1) Under revision

4.6 Zinc, standard solution, corresponding to 0,01 g of Zn per litre.

Transfer 20,0 ml of the zinc standard solution (3.4) to a 1 000 ml one-mark volumetric flask.

Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,01 mg of Zn.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 PTFE beakers, of capacity 250 ml.

5.2 Burette, graduated in 0,05 ml.

5.3 Flame atomic absorption spectrometer, including a light source emitting characteristic zinc spectral lines, for example a hollow-cathode or electrodeless discharge lamp.

5.4 Compressed air supply.

5.5 Cylinder of acetylene.

6 Sampling

Sampling shall be carried out in accordance with ISO/R 1811. The sample should preferably be in the form of drillings with a maximum thickness of 0,3 mm.

7 Procedure

7.1 Preparation of standard matching solutions

7.1.1 Zinc contents between 0,001 and 0,01 % (m/m)

Into a series of four 100 ml one-mark volumetric flasks, introduce the volumes of the zinc standard solution (4.6) and base solution (4.2) shown in table 1. Dilute to the mark with water and mix.

Table 1

Volume of zinc standard solution (4.6)	Volume of copper base solution (4.2)	Mass of zinc per 100 ml after dilution
ml	ml	mg
0*	50	0
1	50	0,01
5	50	0,05
10	50	0,10

* Blank test on reagents for calibration.

7.1.2 Zinc contents between 0,005 and 0,06 % (m/m)

Into a series of six 200 ml one-mark volumetric flasks, introduce the volumes of the zinc standard solution (4.5) and base solution (4.2) shown in table 2. Dilute to the mark with water and mix.

Table 2

Volume of zinc standard solution (4.5)	Volume of copper base solution (4.2)	Mass of zinc per 100 ml after dilution
ml	ml	mg
0*	50	0
1	50	0,025
2	50	0,050
4	50	0,10
8	50	0,20
12	50	0,30

* Blank test on reagents for calibration.

7.1.3 Zinc contents between 0,05 and 0,60 % (m/m)

Into a series of six 200 ml one-mark volumetric flasks, introduce the volumes of the zinc standard solution (4.4) and base solution (4.2) shown in table 3. Dilute to the mark with water and mix. Transfer 100,0 ml of these solutions to each of six 1 000 ml one-mark volumetric flasks, dilute to the mark and mix.

Table 3

Volume of zinc standard solution (3.4)	Volume of copper base solution (3.2)	Mass of zinc per 100 ml after final dilution
ml	ml	mg
0*	50	0
1	50	0,025
2	50	0,05
4	50	0,10
8	50	0,20
12	50	0,30

* Blank test on reagents for calibration.

7.1.4 Zinc contents between 0,5 and 6 % (m/m)

Into a series of six 200 ml one-mark volumetric flasks, introduce the volumes of the zinc standard stock solution (4.3) and base solution (4.2) shown in table 4. Dilute to the mark with water and mix. Transfer 10,0 ml of these solutions to each of six 1 000 ml one-mark volumetric flasks, dilute to the mark and mix.

Table 4

Volume of zinc standard stock solution (4.3)	Volume of copper base solution (4.2)	Mass of zinc per 100 ml after final dilution
ml	ml	mg
0*	50	0
1	50	0,025
2	50	0,05
4	50	0,10
8	50	0,20
12	50	0,30

* Blank test on reagents for calibration.

7.2 Preparation of test solution

7.2.1 Transfer a test portion of 1 ± 0,000 2 g to a PTFE beaker (5.1). If heating is carried out in a water bath, polypropylene or low-density polyethylene beakers may be used.

7.2.2 Add 40 ml of the attack solution (4.1), cover, and warm gently until the test portion is dissolved, then heat at about 90 °C until nitrous fumes have been expelled. Wash down the cover and sides of the beaker and cool.

7.2.3 For zinc contents between 0,001 and 0,01 % (m/m), transfer the solution (7.2.2) quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

7.2.4 For zinc contents between 0,005 and 0,06 % (m/m), transfer the solution (7.2.2) quantitatively to a 200 ml one-mark volumetric flask, dilute to the mark and mix.

7.2.5 For zinc contents between 0,05 and 0,60 % (m/m), transfer the solution (7.2.2) quantitatively to a 200 ml one-mark volumetric flask, dilute to the mark and mix. Transfer 100 ml of this solution to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

7.2.6 For zinc contents between 0,50 and 6 % (m/m), transfer the solution (7.2.2) quantitatively to a 200 ml one-mark volumetric flask, dilute to the mark and mix. Transfer 10,0 ml of this solution to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

7.3 Spectrometric measurements

7.3.1 Preparation of apparatus [1)]

Turn on the electric system and allow it to warm up. Adjust the zinc hollow-cathode lamp and set the wavelength drum to 213,8 nm. Regulate the air/acetylene flame.

7.3.2 Measurement of standard matching solutions

Transfer portions of the standard matching solutions to small beakers and atomize into the flame. Take care to maintain a constant rate of aspiration over the full range of standard matching solutions. Record the absorbance of each standard matching solution. After each aspiration of a standard matching solution, atomize a small amount of water to clean the burner.

Signal amplification may be necessary for the series of standard matching solutions having the lowest concentration of zinc (7.1.1).

7.3.3 Plotting of calibration curves

Using the measurements from 7.3.2, plot calibration curves with the zinc concentrations (mg/100 ml) on the abscissa and corresponding absorbance values on the ordinate axis, subtracting the blank value from each standard value.

NOTE — Recent work has demonstrated that calibration graphs may suffer from excessive curvature. A non-acceptable curvature would result when the mid-point calibration absorbance exceeds 0,55 times the absorbance of the maximum calibration solution. If this situation is encountered, the calibration solutions (7.1) should be diluted to the minimum standard volume needed to attain the curvature criterion stated. The test solutions (7.2) should also be diluted in the same proportion.

7.3.4 Measurement of test solution

Measure the absorbances of the test solution and blank in the same manner as the standard matching solutions (7.3.2). Bracket the test solution by two appropriate standard matching solutions. Carry out all measurements consecutively and without interruption in order to minimize instrumental fluctuations.

7.4 Blank test

Carry out a blank test at the same time as the determination and following the same procedure, using the same quantities of reagents and of pure copper as for the determination but omitting the test portion.

7.5 Check test

Make a preliminary check of the apparatus by preparing a solution of standard material or a synthetic sample containing a known amount of zinc and of composition similar to the material to be analysed, and carrying out the procedure as specified in 7.2 and 7.3.

8 Expression of results

Using the appropriate calibration curve (7.3.3), determine the amount of zinc in the test solution corresponding to the measured absorbance. Calculate the zinc content, as a percentage by mass, according to the formula

$$\frac{(m_2 - m_1) \times f \times V}{1\,000 \times m_0}$$

where

m_0 is the mass, in grams, of the test portion (1 g);

1) Refer to the manufacturer's manual for specific details of optimum instrument settings.

m_1 is the mass, in milligrams, of zinc found in the blank test solution;

m_2 is the mass, in milligrams, of zinc found in the test solution;

f is the ratio between the volume of the first flask, in millilitres, and the volume, in millilitres, transfered to the second flask; $f = 1$ when the first flask contains the final test solution, i.e., if no successive dilution is made.

V is the volume, in millilitres, of the flask containing the final test solution.

9 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 characteristics noted during the determination;

e) any operations not included in this International Standard, or regarded as optional.