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Aluminium and aluminium alloys — Determination of copper — Atomic absorption spectrophotometric method

Aluminium et alliages d'aluminium — Dosage du cuivre — Méthode par spectrophotométrie d'absorption atomique

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FOREWORD

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International Standard ISO 3980 was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys,* and was circulated to the member bodies in November 1975.

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

Austria	Japan	Spain
Belgium	Korea, Rep. of	Switzerland
Czechoslovakia	Mexico	Turkey
France	Norway	United Kingdom
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The member body of the following country expressed disapproval of the document on technical grounds :

Australia

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Aluminium and aluminium alloys — Determination of copper — Atomic absorption spectrophotometric method

1 SCOPE

This International Standard specifies a method for the determination of copper in aluminium and its alloys by atomic absorption spectrophotometry.

2 FIELD OF APPLICATION

This method is applicable to copper (Cu) contents between 0,005 and 5 % (*m/m*).

3 PRINCIPLE

Dissolution of a test portion with hydrochloric acid and hydrogen peroxide. Atomization of this solution into an air-acetylene (or nitrous oxide-acetylene) flame of the atomic absorption apparatus. Comparison of the absorption of the resonance energy of copper (wavelength normally 324,7 nm) with that of calibration solutions.

4 REAGENTS

During the analysis use only reagents of analytical grade and only distilled or de-ionized water.

4.1 Hydrochloric acid, ρ about 1,1 g/ml, solution about 20 % (*m/m*).

Dilute 500 ml of hydrochloric acid, ρ about 1,19 g/ml, solution about 33 % (*m/m*), with 500 ml of water.

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

4.3 Sulphuric acid, ρ about 1,48 g/ml, solution about 58 % (*m/m*).

While stirring and cooling, add 50 ml of sulphuric acid, ρ about 1,84 g/ml, solution about 96 % (*m/m*), to 40 ml of water. Cool again; then make up to volume in a 100 ml volumetric flask and mix.

4.4 Hydrofluoric acid, ρ about 1,13 g/ml, solution about 40 % (*m/m*).

4.5 Nitric acid, ρ about 1,4 g/ml, solution about 68 % (*m/m*).

4.6 Aluminium, 20 g/l base solution.

Weigh, to the nearest 0,01 g, 20 g of aluminium free from copper (purity $\geqslant$ 99,99 %) previously pickled; transfer it to a 1 000 ml beaker (for example) and cover. Add, in small portions, 600 ml of the hydrochloric acid solution (4.1) and if necessary a drop of metallic mercury to facilitate the attack. If necessary, warm gently to assist the dissolution; then add some drops of the hydrogen peroxide solution (4.2), and boil some minutes to remove the excess of hydrogen peroxide.

After cooling, transfer the solution thus obtained quantitatively to a 1 000 ml volumetric flask; make up to volume and mix.

50 ml of this solution contains 1 g of aluminium and 30 ml of hydrochloric acid solution (4.1).

5 ml of this solution contains 0,1 g of aluminium and 3 ml of hydrochloric acid solution (4.1).

2 ml of this solution contains 0,04 g of aluminium and 1,2 ml of hydrochloric acid solution (4.1).

4.7 Copper, standard solution corresponding to 0,1 g of Cu per litre.

Transfer 1 ± 0,001 g of electrolytic copper (purity $\geqslant$ 99,95 %) to a 250 ml beaker (for example) and cover. Add 5 ml of water and 5 ml of the nitric acid solution (4.5) and warm gently until the dissolution is complete and crystallization begins. Take up with water, and heat, if necessary, to complete the dissolution of the salts. Cool, transfer quantitatively to a 1 000 ml volumetric flask; make up to volume and mix.

Transfer 100,0 ml of this solution to a 1 000 ml volumetric flask; make up to volume with water and mix.

1 ml of this standard solution contains 0,1 mg of Cu.

4.8 Copper, standard solution corresponding to 0,01 g of Cu per litre.

Transfer 10,0 ml of the standard copper solution (4.7) to a 100 ml volumetric flask; make up to volume and mix.

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

5 APPARATUS

Usual laboratory equipment and :

5.1 Burette graduated in 0,05 ml.

5.2 Atomic absorption spectrophotometer fitted with a burner supplied with compressed air or nitrous oxide, and acetylene.

5.3 Copper hollow-cathode lamp.

6 SAMPLING

6.1 Laboratory sample[1)]

6.2 Test sample

Chips 1 mm thick or less shall be obtained by milling or drilling.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the test sample (6.2).

7.2 Establishment of the calibration curves

7.2.1 *Preparation of the reference solutions*

7.2.1.1 COPPER CONTENTS BETWEEN 0,005 AND 0,05 %

To a series of seven 100 ml volumetric flasks transfer the volumes of the standard copper solutions (4.7 and 4.8) shown in table 1, using the burette (5.1). Add to each flask 50 ml of the aluminium solution (4.6); make up to volume and mix.

TABLE 1

Standard copper solution (4.8)	Corresponding mass of copper	Corresponding mass of aluminium	Copper in sample
ml	mg	g	% (*m/m*)
0*	0	1	0
5	0,05	1	0,005
10	0,10	1	0,01
20	0,20	1	0,02
Standard copper solution (4.7)			
ml			
3	0,30	1	0,03
4	0,40	1	0,04
5	0,50	1	0,05

* Blank test of the reagents used for the calibration curve.

NOTE — If necessary, appropriately amplify the signal of the spectrophotometer.

7.2.1.2 COPPER CONTENTS BETWEEN 0,05 AND 0,5 %

To a series of seven 100 ml volumetric flasks transfer the volumes of the standard copper solutions (4.7 and 4.8), shown in table 2, using the burette (5.1). Add to each flask 5 ml of the aluminium solution (4.6), make up to volume and mix.

TABLE 2

Standard copper solution (4.8)	Corresponding mass of copper	Corresponding mass of aluminium	Copper in sample
ml	mg	g	% (*m/m*)
0*	0	0,1	0
5	0,05	0,1	0,05
10	0,10	0,1	0,10
20	0,20	0,1	0,20
Standard copper solution (4.7)			
ml			
3	0,30	0,1	0,30
4	0,40	0,1	0,40
5	0,50	0,1	0,50

* Blank test of the reagents used for the calibration curve.

NOTE — If necessary, appropriately amplify the signal of the spectrophotometer.

7.2.1.3 COPPER CONTENTS BETWEEN 0,5 AND 5 %

To a series of seven 100 ml volumetric flasks transfer the volumes of the standard copper solution (4.7) shown in table 3, using the burette (5.1). Add to each flask 2 ml of the aluminium solution (4.6), make up to volume and mix.

TABLE 3

Standard copper solution (4.7)	Corresponding mass of copper	Corresponding mass of aluminium	Copper in sample
ml	mg	g	% (*m/m*)
0*	0	0,04	0
2	0,2	0,04	0,5
4	0,4	0,04	1,0
7	0,7	0,04	1,75
10	1,0	0,04	2,5
15	1,5	0,04	3,75
20	2,0	0,04	5,0

* Blank test of the reagents used for the calibration curve.

NOTE — If necessary, appropriately amplify the signal of the spectrophotometer.

1) The sampling of aluminium and aluminium alloys will form the subject of a future International Standard.

7.2.2 *Spectrophotometric measurements and plotting of the calibration curves*

Atomize the reference solutions into the flame and measure the intensity of the non-absorbed radiations at a wavelength of 324,7 nm, for example. Then plot the calibration curves.

7.3 Determination

7.3.1 *Preparation of the test solution*

Transfer the test portion (7.1) to a 250 ml beaker (for example) and cover. Add about 30 to 40 ml of water, then, in small portions, 30 ml of the hydrochloric acid solution (4.1), heating gently, if necessary, to complete the dissolution. Add some drops of the hydrogen peroxide solution (4.2) and heat for about 10 min to remove the excess of hydrogen peroxide. Filter if necessary.

NOTE – For silicon contents greater than 1 % proceed as follows :

Transfer the filter containing the silicon to a platinum crucible and ignite it taking care that it does not inflame, then heat at about 550 °C. After cooling add 2 ml of the sulphuric acid solution (4.3), 5 ml of the hydrofluoric acid solution (4.4) and, drop by drop, the nitric acid solution (4.5) until a clear solution is obtained (about 1 ml). Evaporate to dryness and heat again, at about 700 °C for some minutes, to volatilize the silicon completely. After cooling, bring the non-volatile matter into solution with the least possible quantity of the hydrochloric acid solution (4.1), filter if necessary and add this filtrate quantitatively to the previous filtrate.

7.3.1.1 COPPER CONTENTS BETWEEN 0,005 AND 0,05 %

Transfer the solution (7.3.1) quantitatively to a 100 ml volumetric flask, make up to volume and mix.

Use calibration curve 7.2.1.1.

7.3.1.2 COPPER CONTENTS BETWEEN 0,05 AND 0,5 %

Transfer the solution (7.3.1) quantitatively to a 1 000 ml volumetric flask, make up to volume and mix.

Use calibration curve 7.2.1.2.

7.3.1.3 COPPER CONTENTS BETWEEN 0,5 AND 5 %

Transfer the solution (7.3.1) quantitatively to a 500 ml volumetric flask, make up to volume and mix. Then transfer 100,0 ml of the solution obtained to another 500 ml volumetric flask, make up to volume and mix.

Use calibration curve 7.2.1.3.

8 EXPRESSION OF RESULTS

By means of the calibration curves, determine the quantity of copper corresponding to the spectrophotometric measurements of the test solution and of the blank test solution.

The copper (Cu) content is given, as a percentage by mass, by the formula

$$\frac{(m_1 - m_2) \times R}{m_0 \times 10}$$

where

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

m_1 is the mass, in milligrams, of copper found in the test solution (total or aliquot) submitted to the spectrophotometric reading;

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

R is the ratio between the volume of the dilution of the whole test portion (100 or 1 000 or 2 500 ml) and the volume of standard solutions taken (100 ml).

9 CONFIDENCE INTERVAL OF RESULTS

[Under study.]

10 TEST REPORT

The test report shall include the following information :

a) the reference of the method used;

b) the results and the form in which they are expressed;

c) any particular details noted during the test;

d) any operations not included in this International Standard or any optional operations.

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