INTERNATIONAL STANDARD ISO 3256

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

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

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FOREWORD

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International Standard ISO 3256 was developed by Technical Committee ISO/TC 79, *Light metals and their alloys,* and was circulated to the member bodies in September 1976.

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

Australia	Italy	South Africa, Rep. of
Belgium	Japan	Spain
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United Kingdom

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Aluminium and aluminium alloys – Determination of magnesium – Atomic absorption spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an atomic absorption spectrophotometric method for the determination of magnesium in aluminium and its alloys. The method is applicable to products having magnesium contents between 0,01 and 5 %.

2 REFERENCE

ISO . . . , *General conditions of application of atomic absorption spectrophotometry.*[1]

3 PRINCIPLE

Dissolution of the test portion with hydrochloric acid and hydrogen peroxide. Atomization of this solution in an acetylene/air flame, in the presence of strontium chloride, or in a dinitrogen monoxide/acetylene flame of the atomic absorption apparatus.

Comparison of the absorption of resonance energy of magnesium (wavelength 285,2 nm, normally, or 279,6 nm : less sensitive) with that of standard solutions.

4 REAGENTS

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

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

Dilute 500 ml of hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution, with 500 ml of water.

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

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

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

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

4.5 Nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution.

4.6 Aluminium, base solution : 20 g/l.

Weigh, to the nearest 0,01 g, 20 g of previously pickled aluminium free from magnesium (purity ⩾ 99,99 %); place in a beaker of suitable size (1 000 ml, for instance), and cover. Add, in small portions, 800 ml of the hydrochloric acid (4.1) and, if necessary, a drop of metallic mercury to assist the attack. If necessary, warm gently to help the dissolution; then add a few drops of the hydrogen peroxide (4.2), and boil for a few minutes to remove the excess of hydrogen peroxide. After cooling, quantitatively transfer the solution so obtained to a 1 000 ml volumetric flask; dilute to volume and mix.

25 ml of this solution contain 0,5 g of aluminium.

4.7 Aluminium, base solution : 1 g/l.

Transfer 50,0 ml of the base aluminium solution (4.6) to a 1 000 ml volumetric flask; dilute to volume and mix.

100 ml of this solution contain 0,1 g of aluminium.

25 ml of this solution contain 0,025 g of aluminium.

5 ml of this solution contain 0,005 g of aluminium.

4.8 Strontium chloride ($SrCl_2$), 50 g/l solution.

Weigh, to the nearest 0,01 g, 76 g of $SrCl_2.6H_2O$; transfer it to beaker of suitable size (400 ml for instance) and dissolve with about 400 ml of water. Transfer quantitatively to a 500 ml volumetric flask; dilute to volume and mix.

Keep this solution in a bottle made of plastic material.

4.9 Magnesium, standard solution, 1 g/l.

Weigh, to the nearest 0,000 2 g, 1 g of extra-pure magnesium (purity ⩾ 99,95 %) and transfer it to a conical flask of suitable size (1 000 ml, for instance). Add about 200 ml of water, then 30 ml of the hydrochloric acid (4.1) and

1) In preparation.

cover. When dissolution is complete, quantitatively transfer the solution to a 1 000 ml volumetric flask; dilute to volume and mix.

1 ml of this solution contains 1 mg of magnesium.

4.10 Magnesium, standard solution, 0,05 g/l.

Transfer 50,0 ml of the standard magnesium solution (4.9) to a 1 000 ml volumetric flask; dilute to volume and mix.

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

5 APPARATUS

Ordinary laboratory apparatus and :

5.1 Burette, graduated in 0,05 ml.

5.2 Atomic absorption spectrophotometer fitted with a burner supplied by cylinders of compressed air, acetylene and/or dinitrogen monoxide.

5.3 Magnesium hollow-cathode lamp.

6 SAMPLING

6.1 Laboratory sample[1)]

6.2 Test sample

Chips not more than 1 mm thick shall be obtained by milling or drilling.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,000 1 g, 0,5 g of the test sample (6.2).

7.2 Preparation of the calibration curves

7.2.1 *Preparation of the standard solutions*

7.2.1.1 MAGNESIUM CONTENTS BETWEEN 0,01 AND 0,05 %

Into a series of six 250 ml volumetric flasks, introduce the volumes of the standard magnesium solution (4.10) shown in table 1, using the burette (5.1). Add to each flask 25 ml of the aluminium solution (4.6) and, only when the acetylene/air flame is used, 20 ml of the strontium chloride solution (4.8). Dilute to volume and mix.

TABLE 1

Standard magnesium solution (4.10)	Corresponding mass of magnesium	Mass of aluminium present	Magnesium in the sample
ml	mg	g	%
0*	0	0,5	0
1	0,05	0,5	0,01
2	0,10	0,5	0,02
3	0,15	0,5	0,03
4	0,20	0,5	0,04
5	0,25	0,5	0,05

* Blank test of the calibration curve reagents.

7.2.1.2 MAGNESIUM CONTENTS BETWEEN 0,05 AND 0,25 %

Into a series of six 250 ml volumetric flasks, introduce the volumes of the standard magnesium solution (4.10) shown in table 2, using the burette (5.1). Add to each flask 100 ml of the aluminium solution (4.7) and, only when the acetylene/air flame is used, 5 ml of the strontium chloride solution (4.8). Dilute to volume and mix.

TABLE 2

Standard magnesium solution (4.10)	Corresponding mass of magnesium	Mass of aluminium present	Magnesium in the sample
ml	mg	g	%
0*	0	0,1	0
1	0,05	0,1	0,05
2	0,10	0,1	0,10
3	0,15	0,1	0,15
4	0,20	0,1	0,20
5	0,25	0,1	0,25

* Blank test of the calibration curve reagents.

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

7.2.1.3 MAGNESIUM CONTENTS BETWEEN 0,25 AND 1 %

Into a series of six 250 ml volumetric flasks, introduce the volumes of the standard magnesium solution (4.10) shown in table 3, using the burette (5.1). Add to each flask 25 ml of the aluminium solution (4.7) and, only when the acetylene/air flame is used, 5 ml of the strontium chloride solution (4.8). Dilute to volume and mix.

TABLE 3

Standard magnesium solution (4.10)	Corresponding mass of magnesium	Mass of aluminium present	Magnesium in the sample
ml	mg	g	%
0*	0	0,025	0
1	0,05	0,025	0,2
2	0,10	0,025	0,4
3	0,15	0,025	0,6
4	0,20	0,025	0,8
5	0,25	0,025	1,0

* Blank test of the calibration curve reagents.

7.2.1.4 MAGNESIUM CONTENTS BETWEEN 1 AND 5 %

Into a series of six 250 ml volumetric flasks, introduce the volumes of the standard magnesium solution (4.10) shown in table 4, using the burette (5.1). Add to each flask 5 ml of the aluminium solution (4.7) and, only when the acetylene/air flame is used, 5 ml of the strontium chloride solution (4.8). Dilute to volume and mix.

TABLE 4

Standard magnesium solution (4.10)	Corresponding mass of magnesium	Mass of aluminium present	Magnesium in the sample
ml	mg	g	%
0*	0	0,005	0
1	0,05	0,005	1
2	0,10	0,005	2
3	0,15	0,005	3
4	0,20	0,005	4
5	0,25	0,005	5

* Blank test of the calibration curve reagents.

7.2.2 *Spectrophotometric measurements and plotting of the calibration curves*

Atomize the standard solutions into the flame and measure the intensity of the unabsorbed radiations at a wavelength of, for example, 285,2 nm. Then plot the calibration curves.

7.3 Determination

7.3.1 *Preparation of the test solution*

Transfer the test portion (7.1) to a beaker of suitable size (250 ml for instance), and cover. Add about 30 to 40 ml of water, and then, in small portions, 20 ml of the hydrochloric acid (4.1), warming gently, if necessary, in order to complete the dissolution. Add a few drops of hydrogen peroxide (4.2) and boil for about 10 min to remove excess hydrogen peroxide. Filter if necessary.

NOTE — For silicon contents higher than 1 %, proceed as follows :

Place the filter containing the silicon into a platinum crucible and char it, taking care that it does not burn; then calcine it at about 550 °C. After cooling, add 2 ml of the sulphuric acid (4.3), 5 ml of the hydrofluoric acid (4.4) and, drop by drop, some nitric acid (4.5) in such a manner as to obtain a clear solution (about 1 ml). Evaporate to dryness and calcine again, at approximately 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 hydrochloric acid (4.1); filter if necessary and add this filtrate quantitatively to the foregoing filtrate.

7.3.1.1 MAGNESIUM CONTENTS BETWEEN 0,01 AND 0,05 %

Transfer the solution (7.3.1) quantitatively to a 250 ml volumetric flask; add (only when the acetylene/air flame is used) 20 ml of the strontium chloride solution (4.8); dilute to volume and mix. Use the calibration curve 7.2.1.1.

7.3.1.2 MAGNESIUM CONTENTS BETWEEN 0,05 AND 0,25 %

Transfer the solution (7.3.1) quantitatively to a 500 ml volumetric flask; dilute to the mark and mix. Then pipette 100,0 ml of the solution obtained into a 250 ml volumetric flask; add (only when the acetylene/air flame is used) 5 ml of the strontium chloride solution (4.8); dilute to volume and mix. Use the calibration curve 7.2.1.2.

7.3.1.3 MAGNESIUM CONTENTS BETWEEN 0,25 AND 1 %

Transfer the solution (7.3.1) quantitatively to a 500 ml volumetric flask; dilute to the mark and mix. Then pipette 25,0 ml of the solution obtained into a 250 ml volumetric flask; add (only when the acetylene/air flame is used) 5 ml of the strontium chloride solution (4.8); dilute to volume and mix. Use the calibration curve 7.2.1.3.

7.3.1.4 MAGNESIUM CONTENTS BETWEEN 1 AND 5 %

Transfer the solution (7.3.1) quantitatively to a 500 ml volumetric flask; dilute to the mark and mix. Then pipette 5,0 ml of the solution obtained into a 250 ml volumetric flask; add (only when the acetylene/air flame is used) 5 ml of the strontium chloride solution (4.8); dilute to volume and mix. Use the calibration curve 7.2.1.4.

8 EXPRESSION OF RESULTS

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

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

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

where

m_0 is the mass, in grams, of the test portion (0,5 g);

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

m_2 is the mass, in milligrams, of magnesium found in the test solution (totality or aliquot part) submitted to the spectrophotometric reading;

r is the ratio between the dilution volume of the whole test portion (250 or 1 250 or 5 000 or 25 000 ml) and the volume to which the standard solutions were brought (250 ml).

9 CONFIDENCE INTERVAL OF RESULTS

[Under study]

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

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

c) any unusual features noted during the test;

d) any operation not included in this International Standard or regarded as optional.

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