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Magnesium and magnesium alloys — Determination of aluminium — Chromazurol S photometric method

Magnésium et alliages de magnésium — Dosage de l'aluminium — Méthode photométrique au chromazurol S

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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	France	Romania
Belgium	Germany	South Africa, Rep. of
Bulgaria	Hungary	Spain
Canada	Ireland	Sweden
Chile	Italy	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
Finland	Poland	U.S.A.

No Member Body expressed disapproval of the document.

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Magnesium and magnesium alloys — Determination of aluminium — Chromazurol S photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method using chromazurol S for the determination of aluminium in magnesium and certain magnesium alloys.

The method is applicable to the determination of aluminium content between 0,01 and 0,20 %.

This method does not apply to alloys containing zirconium and/or rare earths and/or thorium.

2 PRINCIPLE

Dissolution with sulphuric acid.

Complexing of the iron and the copper by the addition of ascorbic acid and sodium thiosulphate. Formation of the aluminium-chromazurol S complex in a buffered medium (pH 5,3 to 5,5).

Photometric measurement of the coloured complex at a wavelength of about 545 nm.

3 REAGENTS

During the analysis use only distilled water or water of equivalent purity.

3.1 Sulphuric acid, ρ 1,26 g/ml, approximately 9 N solution.

Carefully add 250 ml of sulphuric acid (ρ 1,84 g/ml), 35,6 N approximately, to about 500 ml of water. After cooling, make up the volume to 1 000 ml and mix.

3.2 Ascorbic acid, 10 g/l solution.

Dissolve 1 g of ascorbic acid ($C_6H_8O_6$) in water. Make up the volume to 100 ml and mix.

Use a freshly prepared solution.

3.3 Buffer solution.

Dissolve 150 g of sodium acetate ($CH_3.COONa.3H_2O$) and 5 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in water, filter if necessary, make up the volume to 1 000 ml and mix.

Use a freshly prepared solution.

3.4 Chromazurol S, 0,1 g/l methanolic solution.

Dissolve 0,1 g of chromazurol S in a 50 % (*V*/*V*) solution of water and methanol. Make up the volume to 1 000 ml with the same methanol solution and mix.

3.5 Acetone.

3.6 Aluminium, 0,05 g/l standard solution.

Dissolve 0,879 2 g of aluminium potassium sulphate $[Al_2(SO_4)_3.K_2SO_4.24H_2O]$ in water. Transfer the solution quantitatively to a 1 000 ml volumetric flask, make up to volume and mix.

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

3.7 Aluminium, 0,012 5 g/l standard solution.

Transfer 50,0 ml of standard aluminium solution (3.6) to a 200 ml volumetric flask, make up to volume and mix.

1 ml of this standard solution contains 0,012 5 mg of Al.

Prepare this solution at the moment of use.

4 APPARATUS

Ordinary laboratory apparatus, and

4.1 Spectrophotometer, or

4.2 Photoelectric absorptiometer, fitted with filters guaranteeing maximum absorption between 535 and 555 nm.

5 SAMPLING

5.1 Laboratory sample[1])

5.2 Test sample

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

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

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,001 g, 0,5 g of the test sample (5.2).

6.2 Blank test

Carry out, in parallel with the analysis, a blank test, using the same procedure and the same quantities of all reagents as for the determination, except that the quantity of sulphuric acid (3.1) shall be reduced to 5 ml.

6.3 Establishment of the calibration curves

6.3.1 *Preparation of the standard matching solutions* (related to photometric measurements carried out with an optical path length of 2 cm)

6.3.1.1 ALUMINIUM CONTENTS OF BETWEEN 0,01 AND 0,05 %

Into a series of six 250 ml volumetric flasks, transfer 5 ml of sulphuric acid (3.1) and then respectively the volumes of the standard aluminium solution (3.7) indicated in table 1.

TABLE 1

Volume of standard aluminium solution (3.7)	Corresponding mass of aluminium	Aluminium in the sample
ml	mg	%
0*	0	–
2,0	0,025 0	0,01
5,0	0,062 5	0,02
10,0	0,125 0	0,03
15,0	0,187 5	0,04
20,0	0,250 0	0,05

* Compensation solution

Make up to volume and mix.

Transfer 20,0 ml of each standard matching solution respectively to 100 ml volumetric flasks. Add 5 ml of ascorbic acid solution (3.2), 20 ml of buffer solution (3.3), 20 ml of chromazurol S solution (3.4) and 2 ml of acetone (3.5). Make up to volume and mix. The pH of the solutions shall be between 5,3 and 5,5.

The mass of the Al in these standard matching solutions is respectively :

0 – 0,002 – 0,005 – 0,010 – 0,015 – 0,020 mg.

6.3.1.2 ALUMINIUM CONTENTS OF BETWEEN 0,05 AND 0,20 %

To a series of six 250 ml volumetric flasks, transfer 5 ml of sulphuric acid (3.1) and then respectively the quantities of the standard aluminium solution (3.6) indicated in table 2. 2.

TABLE 2

Volume of standard aluminium solution (3.6)	Corresponding mass of aluminium	Aluminium in the sample
ml	mg	%
0*	0	–
2,0	0,100	0,02
5,0	0,250	0,05
10,0	0,500	0,10
15,0	0,750	0,15
20,0	1,000	0,20

* Compensation solution

Make up to volume and mix.

Transfer 5,0 ml of each standard matching solution respectively to 100 ml volumetric flasks. Add 5 ml of ascorbic acid solution (3.2), 5 ml of buffer solution (3.3), 20 ml of chromazurol S solution (3.4) and 2 ml of acetone (3.5). Make up to volume and mix. The pH of the solutions shall be between 5,3 and 5,5.

The mass of the Al in these standard matching solutions is respectively :

0 – 0,002 – 0,005 – 0,010 – 0,015 – 0,020 mg.

6.3.2 *Photometric measurements*

After 25 min, but before 30 min, carry out the photometric measurements by means of the spectophotometer (4.1), at the maximum of the absorption curve (wavelength about 545 nm) or the photoelectric absorptiometer (4.2) fitted with suitable filters, after having adjusted the instruments to zero absorbance in relation to the compensation solution.

6.3.3 *Plotting of the calibration curves*

Plot two graphs for aluminium contents between 0,01 and 0,05 % (6.3.3.1) and for aluminium contents between 0,05 and 0,20 % (6.3.3.2) respectively – having for example as abscissae the values, expressed in milligrams, of the quantities of aluminium (Al) contained in 100 ml of the standard matching solution and as ordinates the corresponding values of absorbance.

6.4 Determination

6.4.1 *Preparation of the test solution*

Transfer the test portion (6.1) to a beaker of suitable size (250 ml, for example), cover with a watch glass and add 20 ml of water. Then add, in small portions, 10 ml of

sulphuric acid (3.1). Once the reaction is completed, heat to boiling and boil for 1 to 2 min. Cool, filter if necessary and transfer the solution quantitatively to a 250 ml volumetric flask. Make up to volume and mix.

6.4.2 *Development of colour*

The aliquot portion to be taken according to the aluminium content to be determined, as well as the quantity of buffer solution to be used, is indicated in table 3.

TABLE 3

Presumed aluminium content	Aliquot portion to be taken for the colour reaction		Volume of buffer solution (3.3)
	volume	corresponding mass of aluminium	
%	ml	mg	ml
0,01 to 0,05	20	0,004 to 0,020	20
0,05 to 0,20	5	0,005 to 0,020	5

To the corresponding aliquot portion, placed in a 100 ml volumetric flask, add 5 ml of ascorbic acid solution (3.2), the quantity of buffer solution (3.3) indicated in table 3, 20 ml of chromazurol S solution (3.4) and 2 ml of acetone (3.5). Make up to volume and mix. The solution shall have a pH of between 5,3 and 5,5.

6.4.3 *Photometric measurements*

After 25 min, but before 30 min, carry out the photometric measurement according to the procedure described in 6.3.2, after having adjusted the instrument to zero absorbance against the blank test solution.

7 EXPRESSION OF RESULTS

By means of the calibration curve 6.3.3.1 or 6.3.3.2, determine the quantity of aluminium corresponding to the value of the photometric measurement of the aliquot portion of the test solution.

The aluminium (Al) content is given, as a percentage by mass, by the formula

$$\frac{m_1 \times R \times 100}{m_0}$$

where

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

m_1 is the mass, in milligrams, of aluminium found in the aliquot portion of the test solution;

R is the ratio between the volume of the test solution and the volume of the aliquot portion taken for the colour reaction.

8 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) account of any particular details noted during the test;

d) account of any operations not specified in this International Standard.

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