International Standard 4194

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Magnesium alloys — Determination of zinc content — Flame atomic absorption spectrometric method

Alliages de magnésium — Dosage du zinc — Méthode par spectrométrie d'absorption atomique dans la flamme

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Foreword

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

Australia	Germany, F. R.	South Africa, Rep. of
Austria	Hungary	Spain
Brazil	India	Sweden
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The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

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Magnesium 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 magnesium alloys.

The method is applicable to products having zinc (Zn) contents between 0,1 and 6 % (*m/m*).

2 Principle

Dissolution of a test portion in hydrochloric acid solution in the presence of hydrogen peroxide and hydrofluoric acid. Aspiration of the solution into an air-acetylene flame and comparison of the absorbance of resonance energy of zinc by the test solution (wavelength of 213,9 nm normally) with that of standard solutions.

3 Reagents

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

3.1 Magnesium, extra pure (purity 99,99 %), free from zinc.

3.2 Hydrochloric acid, ϱ approximately 1,19 g/ml, about 38 % (*m/m*) or approximately 12 mol/l solution.

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

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

3.5 Magnesium, 1 g/l solution.

Weigh, to the nearest 0,001 g, 1 g of the extra pure magnesium (3.1), transfer it to a 250 ml beaker and cover with a watch glass. Add 50 ml of water and, in small portions, 20 ml of the hydrochloric acid solution (3.2), warming, if necessary, in order to complete the dissolution. Add 5 drops of the hydrogen peroxide solution (3.3) and boil for 5 min. After cooling, quantitatively transfer the solution so obtained to a 1 000 ml volumetric flask, dilute to the mark and mix.

3.6 Zinc, standard solution corresponding to 1 g of Zn per litre.

Prepare this standard solution by one of the following methods :

3.6.1 Weigh, to the nearest 0,1 mg, 1 g of extra pure zinc (purity ⩾99,99 %), transfer it to a 400 ml beaker and cover with a watch glass. Add, in small portions, 25 ml of the hydrochloric acid solution (3.2) and heat gently, if necessary, to complete the dissolution. After cooling, quantitatively transfer it to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 1 mg of zinc.

3.6.2 Weigh, to the nearest 0,1 mg, 1,26 g of zinc oxide (ZnO), previously calcinated at 1 000 °C for 1 h and cooled in a desiccator. Transfer it to a 400 ml beaker, and dissolve it in 25 ml of the hydrochloric acid solution (3.2). Dilute the solution, quantitatively transfer it to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 1 mg of zinc.

3.7 Zinc, standard solution corresponding to 0,050 g of Zn per litre.

Transfer 50,0 ml of the standard zinc solution (3.6) to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0,05 mg of zinc.

3.8 Zinc, standard solution corresponding to 0,020 g of Zn per litre.

Transfer 20,0 ml of the standard zinc solution (3.6) to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0,020 mg of zinc.

4 Apparatus

Normal laboratory apparatus and

4.1 Burette, graduated in 0,05 ml.

4.2 Atomic absorption spectrometer, fitted with an air-acetylene burner.

4.3 Compressed air (laboratory installation or gas cylinders).

4.4 Acetylene, in gas cylinders.

4.5 Zinc hollow-cathode lamp.

5 Sampling

5.1 Laboratory sample[1)]

5.2 Test sample

Use chips, not more than 1 mm thick, obtained by milling or drilling.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the test sample (5.2).

6.2 Preparation of the calibration curve

6.2.1 Preparation of the standard solutions

6.2.1.1 Zinc contents between 0,1 and 1,0 % (m/m)

Into a series of seven 100 ml volumetric flasks, introduce the volumes of the standard zinc solution (3.8) shown in table 1, using the burette (4.1). Add to each flask, 20 ml of the magnesium solution (3.5), dilute to the mark and mix.

Table 1

Standard zinc solution (3.8)	Corresponding mass of zinc	Zinc content
ml	mg	% (m/m)
0*	0	0
1,0	0,02	0,1
3,0	0,06	0,3
5,0	0,10	0,5
7,0	0,14	0,7
9,0	0,18	0,9
10,0	0,20	1,0

* Blank test of calibration curve reagents.

6.2.1.2 Zinc contents between 1,0 and 6,0 % (m/m)

Into a series of seven 100 ml volumetric flasks, introduce the volumes of the standard zinc solution (3.7) shown in table 2, using the burette (4.1). Add to each flask, 5 ml of the magnesium solution (3.5), dilute to the mark and mix.

Table 2

Standard zinc solution (3.7)	Corresponding mass of zinc	Zinc content
ml	mg	% (m/m)
0*	0	0
1,0	0,05	1,0
2,0	0,10	2,0
3,0	0,15	3,0
4,0	0,20	4,0
5,0	0,25	5,0
6,0	0,30	6,0

* Blank test of calibration curve reagents.

6.2.2 Spectrometric measurements

Switch on the spectrometer (4.2), fitted with the zinc hollow-cathode lamp (4.5), sufficiently in advance to allow it to stabilize. Adjust the wavelength to about 213,9 nm, and the sensitivity and the slot according to the characteristics of the apparatus. Adjust the pressure of the air and acetylene according to the characteristics of the aspirator-burner, so as to obtain a clear, non-luminous, oxidizing flame.

Aspirate the standard solutions (6.2.1.1 or 6.2.1.2) into the flame and measure their absorbances. Take care to ensure that the volume of standard solutions aspirated per unit time into the flame is kept constant throughout the procedure for preparation of the calibration curve.

NOTE — Aspirate water into the burner after each measurement.

6.2.3 Plotting the graph

Plot a graph, having, for example, the masses, in milligrams, of zinc contained in 100 ml of the standard solutions as abscissae, and the corresponding values of absorbance, corrected for the blank test of the calibration curve reagents (zero term), as ordinates.

6.3 Determination

6.3.1 Preparation of the test solution

Place the test portion (6.1) in a 250 ml beaker and cover with a watch glass. Add about 50 ml of water, and, in small portions, 20 ml of the hydrochloric acid solution (3.2), warming gently, if necessary, to complete the dissolution. Add 5 drops of the hydrogen peroxide solution (3.3). When the reaction is complete, add 2 drops of the hydrofluoric acid solution (3.4) and boil for 5 min. Filter, if necessary, quantitatively transfer the solution to a 100 ml volumetric flask, diluted to the mark with water and mix.

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

6.3.1.1 Zinc contents between 0,1 and 1,0 % (m/m)

Transfer a 10,0 ml aliquot portion of the solution (6.3.1) to a 500 ml volumetric flask, dilute to the mark and mix.

Use calibration curve 6.2.1.1 for this solution.

6.3.1.2 Zinc contents between 1,0 and 6,0 % (m/m)

Transfer a 5,0 ml aliquot portion of the solution (6.3.1) to a 1 000 ml volumetric flask, dilute to the mark and mix.

Use calibration curve 6.2.1.2 for this solution.

6.3.2 Blank test

Carry out a blank test, in parallel with the analysis, using the same procedure and the same quantities of all reagents used in the determination, but replacing the test portion by 0,5 g, weighed to the nearest 0,001 g, of the extra pure magnesium (3.1).

6.3.3 Spectrometric measurements

Measure the absorbances of the test solution (6.3.1.1 or 6.3.1.2), the blank test solution (6.3.2) and the appropriate standard solutions (6.2.1.1 or 6.2.1.2), proceeding as specified in 6.2.2, and taking care to bracket the measurement of absorbance of the test solution and of the blank test solution between two standard solutions having zinc contents as close as possible, respectively, to that to be determined.

7 Expression of results

By means of the calibration curve, determine the quantity of zinc corresponding to the spectrometric measurements of the test solution and of the blank test solution.

The zinc, Zn, content, expressed as a percentage by mass, is given by the formula

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

where

m_0 is the mass, in grams, of test portion in the test solution;

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;

R is the ratio between the dilution-volume of the test solution and the volume of the standard solutions for calibration ($R = 50$ for test solutions prepared in accordance with 6.3.1.1 and $R = 200$ for test solutions prepared in accordance with 6.3.1.2).

8 Test report

The test report shall include the following particulars :

a) identification of the test sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

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

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