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Magnesium alloys — Determination of zinc — Volumetric method

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 1783 replaces ISO Recommendation R 1783-1970 drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys.*

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The Member Bodies of the following countries approved the Recommendation :

Australia	Iran	South Africa, Rep. of
Belgium	Israel	Spain
Canada	Italy	Sweden
Czechoslovakia	Korea, Rep. of	Switzerland
Egypt, Arab Rep. of	Netherlands	Thailand
Germany	New Zealand	Turkey
Greece	Norway	United Kingdom
Hungary	Peru	U.S.A.
India	Poland	

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

France

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Magnesium alloys — Determination of zinc — Volumetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of zinc in magnesium alloys not containing cadmium.

This method is applicable to the determination of zinc content between 0,10 and 8,0 %.

2 PRINCIPLE

Hydrochloric acid attack and elimination of the excess of acid by evaporation. Taking up of the residue in hydrochloric acid solution (2 N) and passage of the solution through a strongly basic anion exchange resin.

Elution of the zinc absorbed on the resin with hydrochloric acid solution (0,005 N).

Titration of the zinc by EDTA standard solution using dithizone indicator.

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3 REAGENTS

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

3.1 Strongly basic anion exchange resin, either heteroporous or isoporous, of the polystyrene type with quaternary ammonium groups (for example Dowex 1 X 2, De-Acidite FF SRA 62 or equivalent) in the chloride form, containing from 2 to 3 % cross linking (expressed as a percentage by mass of D.V.B. (divinylbenzene)), and preferably with a particle size between 150 and 295 μm (− 52 + 100 mesh).

3.2 Acetone, ρ approximately 0,79 g/ml.

3.3 Hydrochloric acid, ρ approximately 1,18 g/ml, 37 % (*m/m*) solution or approximately 12 N.

3.4 Hydrogen peroxide, ρ approximately 1,135 g/ml, approximately 36 % (*m/m*) solution.

3.5 Hydrochloric acid, ρ approximately 1,03 g/ml, approximately 2 N solution.

Dilute 170 ml of the hydrochloric acid (3.3) with water and make up the volume to 1 000 ml.

3.6 Hydrochloric acid, ρ approximately 1,01 g/ml, approximately N solution.

Dilute 85 ml of the hydrochloric acid (3.3) with water and make up the volume to 1 000 ml.

3.7 Hydrochloric acid, ρ approximately 1,0 g/ml, approximately 0,005 N solution.

Dilute 5 ml of the hydrochloric acid (3.6) with water and make up the volume to 1 000 ml.

3.8 Nitric acid, ρ approximately 1,4 g/ml, approximately 67 % (*m/m*) solution or 15 N.

3.9 Ammonia, ρ approximately 0,90 g/ml, approximately 28 % (*m/m*) solution or 14 N.

3.10 Acetic acid, ρ approximately 1,007 g/ml, approximately N solution.

Dilute 58 ml of glacial acetic acid (ρ approximately 1,05 g/ml), approximately 17,4 N solution, with water and make up the volume to 1 000 ml.

3.11 Ammonium acetate, 500 g/l solution.

Dissolve 50 g of ammonium acetate (CH_3COONH_4) in water and make up the volume to 100 ml.

3.12 Zinc, standard solution containing 2 g of zinc per litre.

Weigh, to the nearest 0,001 g, 2,00 g of extra pure zinc and dissolve in 25 ml of the hydrochloric acid solution (3.3) diluted with approximately 75 ml of water. Dilute and transfer the solution quantitatively to a 1 000 ml volumetric flask. Make up to volume and mix.

1 ml of this solution contains 2 mg of zinc.

3.13 Disodium salt of ethylenediaminetetra-acetic acid (EDTA), standard volumetric solution 0,02 M.

3.13.1 *Preparation of the solution*

Dissolve approximately 7,5 g of EDTA in water, filter if necessary, and make up the volume to 1 000 ml. Keep in a plastics bottle.

3.13.2 *Standardization of the solution*

Take 25,0 ml of the zinc standard solution (3.12), corresponding to 50,0 mg of zinc, and place in a vessel of suitable capacity (for example 400 ml). Dilute to about 100 ml, introduce a piece of the litmus paper (3.14) into the solution and, stirring constantly, add the ammonia solution (3.9) until the litmus changes colour. Remove the piece of litmus paper and wash with water.

Add 10 ml of the acetic acid solution (3.10) and 10 ml of the ammonium acetate solution (3.11). Check the pH of the solution by means of the indicator paper (3.15). This value should be between 5 and 5,5. If necessary, bring it back to the indicated value by adding the acetic acid solution (3.10) drop by drop. Then add 50 ml of the acetone (3.2), 2 ml of the dithizone solution (3.16) and titrate with the EDTA solution (3.13) until the indicator changes from red to orange-yellow. This colour should not vary after the addition of 2 drops of the EDTA solution in excess.

3.13.3 *Calculation*

The correction factor, corresponding to the fact that the solution is not exactly 0,02 M, is given by the formula

$$\frac{38,24}{V}$$

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where

38,24 is the volume, in millilitres, of EDTA solution 0,02 M (theoretical value : 1 ml ≙ 1,307 6 mg of zinc) necessary for the titration of 50,0 mg of Zn (50,0 : 1,307 6 = 38,237 9).

V is the volume, in millilitres, of the EDTA solution (3.13) used for the titration of 25,0 ml of the zinc standard solution (3.12) (2 mg × 25,0 = 50,0 mg).

3.14 Litmus paper

3.15 Indicator paper for pH within the range 5 to 6, with 0,2 unit intervals.

3.16 Dithizone, 0,25 g/l ethanolic solution.

Dissolve 0,025 g of dithizone (diphenylthiocarbazone) in ethanol 95 % (*V/V*) and make up the volume to 100 ml with the same ethanol.

It is preferable to prepare the solution just before use.

4 APPARATUS

4.1 Ordinary laboratory equipment

4.2 Glass column 20 mm diameter, approximately 400 mm tall, provided with a stopcock.

5 SAMPLING

5.1 Laboratory sample[1)]

5.2 Test sample

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

6 PROCEDURE

6.1 Preparation of the ion exchange column

First remove any fine particles present in the anion resin (3.1) by means of successive washings with dilute hydrochloric acid (3.7), decanting until a clear solution is obtained. Then allow the resin to stand for several hours (preferably overnight) in the hydrochloric acid solution (3.7). Place a little glass wool at the bottom of the column (4.2), above the stopcock, as a support for the resin.

While shaking, transfer the suspension of resin to the prepared column, taking care to avoid the formation of air bubbles or channels, and operate so as to obtain, after decantation, a column of resin approximately 150 mm high. Wash the column with approximately 100 ml of the hydrochloric acid solution (3.7) at a rate of 7 ml per minute.

Condition the exchange column by introducing, at the same rate, 200 ml of the hydrochloric acid solution (3.5), to which 0,5 ml of the nitric acid solution (3.8) has been added.

While the exchange column is being prepared and during the analysis, the resin should always be covered by the liquid. (See clause 8.)

6.2 Test portion

Weigh, to the nearest 0,001 g, approximately 4 g of the test sample (5.2).

6.3 Blank test

Carry out, parallel with the analysis, a blank test using the same procedure and the same quantities of all the reagents.

6.4 Preparation of the test solution

Introduce the test portion (6.2) into a beaker of a suitable capacity (for example 400 ml). Add approximately 50 ml of water. Cover with a watch glass, then add, in small portions and with care, 30 ml of the hydrochloric acid solution (3.3).

When the reaction appears to be complete, add 2 drops of the hydrogen peroxide (3.4) and heat gently to boiling. Continue boiling gently until all of the hydrogen peroxide has decomposed.

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

If the zinc content is greater that 1 %, transfer the solution quantitatively to a 250 ml volumetric flask. Cool, make up to volume and mix. Then take an aliquot of this solution as indicated in the following table :

Zinc content	Volume of aliquot to be taken	Corresponding mass of test portion
%	ml	g
0,1 to 1,0	Total	4
1 to 3	100	1,6
3 to 6	50	0,8
6 to 8	25	0,4

Introduce the aliquot into a beaker of suitable capacity (for example 250 ml). Place the beaker on a boiling water bath and evaporate just to crystallization. Allow to cool, take up with 100 ml of the hydrochloric acid solution (3.5) and 0,5 ml of the nitric acid solution (3.8) and heat to facilitate dissolving.

If the solution contains insoluble zirconium or if during the evaporation silica has been precipitated, filter the solution through a fine filter and wash thoroughly with small quantities of warm hydrochloric acid solution (3.5).

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6.5 Ion exchange

Cool the test solution (6.4) and pass through the exchange column (6.1) at a rate of 5 to 7 ml per minute. Wash the beaker and the exchange column with four successive 25 ml portions of the hydrochloric acid solution (3.5). Then wash the resin with 100 ml (see 8.3) of the hydrochloric acid solution (3.6), still at the rate of 5 to 7 ml per minute.

Elute the zinc retained by the resin by passing 250 ml of the hydrochloric acid solution (3.7) through the exchange column at the same rate. Collect the eluate in a vessel of suitable capacity (for example 400 ml). Concentrate the eluate to a volume of about 100 ml.

6.6 Titration

Place the litmus paper (3.14) in the eluate and add, while shaking, the ammonia solution (3.9) until the paper changes colour.

Remove the litmus paper and wash with water. Add 20 ml of the acetic acid solution (3.10) and 10 ml of the ammonium acetate solution (3.11). Check the pH value of the solution by means of indicator paper (3.15). This value should be between 5 and 5,5. If necessary, bring it back to the indicated value by adding the acetic acid solution (3.10) drop by drop. Then add 50 ml of the acetone (3.2), 2 ml of the dithizone solution (3.16) and titrate with the EDTA solution (3.13) until the indicator changes from red to orange-yellow. This colour should not vary after the addition of 2 drops of the EDTA solution in excess.

7 EXPRESSION OF RESULTS

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

$$\frac{(V - V_1) \times f \times 1{,}307\,6 \times D}{10\,m}$$

where

V is the volume, in millilitres, of the EDTA solution (3.13) used for the titration of the zinc present in the test sample solution or aliquot taken;

V_1 is the volume, in millilitres, of the EDTA solution (3.13) used for the titration of the zinc present in the same aliquot of the blank test;

f is the correction factor (3.13.3) of the EDTA solution (3.13);

D is the ratio between the volume of the test solution and the volume of the aliquot taken;

m is the mass, in grams, of the test portion;

1,307 6 is the mass, in milligrams, of zinc corresponding to 1 ml of EDTA solution exactly 0,02 M.

8 NOTES

8.1 When not in use, the resin in the exchange column shall always be covered by the hydrochloric acid (3.7).

8.2 The conditioning of the resin shall be carried out immediately before use.

8.3 In the case of alloys containing lead, the quantity of the hydrochloric acid (3.6) must be increased from 100 to 200 ml.

9 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) any unusual features noted during the determination;

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

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