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Zinc alloys — Complexometric determination of magnesium

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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	Germany	Portugal
Austria	India	Romania
Belgium	Ireland	Spain
Canada	Italy	Sweden
Czechoslovakia	Japan	Thailand
Egypt, Arab Rep. of	Norway	
France	Poland	

No Member Body expressed disapproval of the document.

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Zinc alloys — Complexometric determination of magnesium

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a complexometric method for the determination of magnesium in zinc alloys.

The method is applicable to zinc alloys as defined in ISO/R 301, and to die castings made from these alloys.

It allows the determination of magnesium contents between 0,01 and 0,1 %.

2 REFERENCE

ISO/R 301, *Zinc alloy ingots.*

3 PRINCIPLE

Separation of magnesium by co-precipitation with iron(III) hydroxide and complexometric titration with C.D.T.A.

4 REAGENTS

All reagents shall be of analytical reagent grade. Distilled or demineralized water shall be used for preparing the solutions and during the actual determination.

4.1 Hydrochloric acid (ρ = 1,19 g/ml).

4.2 Hydrochloric acid (ρ = 1,19 g/ml) diluted at 1/6 (approximately 2 N).

4.3 Nitric acid (ρ = 1,3 to 1,4 g/ml).

4.4 Iron(III) chloride, solution containing 35 g of $FeCl_3.6H_2O$ and 25 ml of hydrochloric acid (4.1) per litre.

4.5 Sodium hydroxide solution, 400 g/l (10 N), freshly prepared.

4.6 Sodium hydroxide solution, 40 g/l (N).

4.7 Hydrogen peroxide, 3 % H_2O_2 (*m/m*).

4.8 Aqueous ammonia (ρ = 0,91 g/ml).

4.9 Potassium cyanide solution, 250 g/l, freshly prepared.

4.10 Tetraethylenepentamine (Tetren) solution.[1)]

Dilute 200 ml of the reagent with water to 1 l.

4.11 Methyl thymol blue (sodium salt of 3,3'bis-(*NN'*-dicarboxymethyl)-aminomethyl-thymol-sulphone-phthalein) indicator.

Mix thoroughly, by grinding 0,1 g of methyl thymol blue with 10 g of KNO_3.

4.12 Magnesium, standard solution, 0,1 g/l.

Weigh 1 g of 99,95 % pure magnesium with an accuracy of ± 0,001 g and attack with 10 ml of hydrochloric acid (4.1) and 10 ml of water. Dilute with water to 1 000 ml. Transfer 25 ml of this solution to a 250 ml one-mark volumetric flask and dilute to the mark with water. Mix.

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

4.13 C.D.T.A. (Cyclohexane 1,2-diamino tetra-acetic acid) solution 0,01 M.

Weigh 3,64 g of C.D.T.A. Transfer to a 1 000 ml one-mark volumetric flask and dissolve in 100 ml of water containing 20 ml of sodium hydroxide solution (4.6). Dilute to the mark with water. Mix.

1) After preliminary agreement between the interested parties, this solution (4.10) may be used instead of the potassium cyanide solution (4.9).

The magnesium equivalent of this solution is determined as follows :

- transfer exactly 20 ml of the magnesium standard solution (4.12) to a 600 ml beaker;
- add 10 ml of hydrochloric acid (4.1), 250 ml of water and 100 ml of aqueous ammonia (4.8);
- introduce a small knife tip of indicator (4.11) and titrate with the C.D.T.A. solution until the blue coloration disappears;
- carry out a blank test, following the same procedure, but omitting the addition of magnesium standard solution (4.12).

The magnesium equivalent T is given by

$$T = \frac{0{,}002}{V_t - V_{ot}}$$

where

V_t is the volume, in millilitres, of the C.D.T.A. solution used to titrate the magnesium standard solution;

V_{ot} is the volume, in millilitres, of the C.D.T.A. solution used in the blank test.

5 APPARATUS

Ordinary laboratory equipment.

6 SAMPLING

The requirements of ISO...[1] concerning sampling and preparation of samples for analysis shall apply.

7 PROCEDURE

7.1 Test portion

Weigh a 5 g test portion to ± 0,01 g.

7.2 Blank test

Simultaneously with the actual determination, carry out a blank test, proceeding as follows :

7.2.1 Introduce into a 400 ml beaker, equipped with a watch-glass, 20 ml of hydrochloric acid (4.1) and 5 ml of nitric acid (4.3).

7.2.2 Boil for approximately 10 min and proceed as outlined in 7.3.3 to 7.3.9.

7.3 Determination of the magnesium content

7.3.1 Introduce the test portion into a 400 ml beaker, equipped with a watch-glass, and attack with 20 ml of hydrochloric acid (4.1). Complete the dissolution by adding 5 ml of nitric acid (4.3).

7.3.2 Boil gently for 10 min.

7.3.3 Add successively :

- 2 ml of iron(III) chloride solution (4.4);
- 100 ml of sodium hydroxide solution (4.5), added slowly while swirling vigorously;
- 5 ml of potassium cyanide solution (4.9) or 20 ml of Tetren solution (4.10);
- 150 ml of water.

7.3.4 Boil. Digest near boiling point for 30 min to obtain a good flocculation of the iron(III) and magnesium hydroxides.

7.3.5 Filter through a 110 mm diameter close-texture filter paper. Wash the precipitate and the walls of the beaker with hot sodium hydroxide solution (4.6), and twice with hot water.

7.3.6 Dissolve the precipitate on the filter paper with 40 ml of hot hydrochloric acid (4.2) and wash the filter with 100 ml of hot water, receiving the solution and the washings in the original beaker.

7.3.7 Add 1 ml of hydrogen peroxide (4.7). Add aqueous ammonia (4.8), drop by drop until the precipitation of iron(III) hydroxide starts, then 4 or 5 drops in excess.

7.3.8 Boil. Heat gently for 5 min and filter into a 600 ml beaker. Wash five times with hot water.

7.3.9 Allow the solution to cool. Add :

- 100 ml of aqueous ammonia (4.8);
- 4 ml of potassium cyanide solution (4.9) or 15 ml of Tetren solution (4.10);
- a small knife tip of indicator (4.11).

Titrate with the standardized 0,01 M C.D.T.A. solution (4.13) until the blue coloration disappears.

1) In preparation.

8 EXPRESSION OF RESULTS

The magnesium content, as a percentage by mass, is obtained from the formula :

$$\text{Mg}\ \%\ (m/m) = T \times (V - V_0) \times \frac{100}{m}$$

where

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

V is the volume, in millilitres, of the C.D.T.A. solution (4.13) used;

V_0 is the volume, in millilitres, of the C.D.T.A. solution (4.13) used in the blank test.

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results obtained;

c) all details required for complete identification of the sample;

d) all operational details not provided for in this International Standard, or any optional details, as well as any circumstances which could have influenced the results.

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