

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1783

CHEMICAL ANALYSIS OF MAGNESIUM AND ITS ALLOYS

DETERMINATION OF ZINC

VOLUMETRIC METHOD

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1783, *Chemical analysis of magnesium and its alloys – Determination of zinc – Volumetric method*, was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question led to the adoption of Draft ISO Recommendation No. 1783 which was circulated to all the ISO Member Bodies for enquiry in January 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

The following Member Body opposed the approval of the Draft :

France

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided to accept it as an ISO RECOMMENDATION.

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CHEMICAL ANALYSIS OF MAGNESIUM AND ITS ALLOYS
DETERMINATION OF ZINC
VOLUMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a volumetric method for the determination of zinc in magnesium alloys not containing cadmium.

This method is applicable to the determination of zinc contents between 0.10 and 8.0 %.

2. PRINCIPLE

- 2.1 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.
- 2.2 Elution of the zinc absorbed on the resin with hydrochloric acid solution (0.005 N).
- 2.3 Titration of the zinc by EDTA standard solution using dithizone indicator.

3. REAGENTS

- 3.1 *Strongly basic anion exchange resin*, either heteroporous or isoporous, of the polystyrene type with quaternary ammonium groups (for example Dowex 1 × 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* ($d =$ approximately 0.79).
- 3.3 *Hydrochloric acid* ($d =$ approximately 1.18), solution 37 % (m/m) or approximately 12 N.
- 3.4 *Hydrogen peroxide* ($d =$ approximately 1.135), solution approximately 36 % (m/m).
- 3.5 *Hydrochloric acid* ($d =$ approximately 1.03), solution approximately 2 N.
Dilute 170 ml of hydrochloric acid (3.3) with water and make up the volume to 1000 ml.
- 3.6 *Hydrochloric acid* ($d =$ approximately 1.01), solution approximately N.
Dilute 85 ml of hydrochloric acid (3.3) with water and make up the volume to 1000 ml.
- 3.7 *Hydrochloric acid* ($d =$ approximately 1.0), solution approximately 0.005 N.
Dilute 5 ml of hydrochloric acid (3.6) with water and make up the volume to 1000 ml.
- 3.8 *Nitric acid* ($d =$ approximately 1.4), solution approximately 67 % (m/m) or 15 N.
- 3.9 *Ammonia* ($d =$ approximately 0.90), solution approximately 28 % (m/m) or 14 N.

- 3.10 *Acetic acid* ($d =$ approximately 1.007), solution approximately N.
Dilute 58 ml of glacial acetic acid ($d =$ approximately 1.05), solution approximately 17.4 N, with water and make up the volume to 1000 ml.
- 3.11 *Ammonium acetate solution*, 500 g/l.
Dissolve 50 g of ammonium acetate ($\text{CH}_3\text{COONH}_4$) in water and make up the volume to 100 ml.
- 3.12 *Standard zinc 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 hydrochloric acid solution (3.3) diluted with approximately 75 ml of water. Dilute and transfer the solution quantitatively to a 1000 ml volumetric flask. Make up to volume and mix.
1 ml of this solution contains 2 mg of zinc.
- 3.13 *Disodium ethylenediaminetetracetate* (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 1000 ml. Keep in a plastics bottle.
- 3.13.2 STANDARDIZATION OF THE SOLUTION. Take 25.0 ml of standard zinc 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 litmus paper (3.14) into the solution and, stirring constantly, add ammonia solution (3.9) until the litmus changes colour. Remove the piece of litmus paper and wash with water.
Add 10 ml of acetic acid solution (3.10) and 10 ml of ammonium acetate solution (3.11). Check the pH 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 acetic acid solution (3.10) drop by drop. Then add 50 ml of acetone (3.2), 2 ml of 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 two drops of 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 following formula :
- $$\frac{38.24}{V}$$
- where
- 38.24 is the volume, in millilitres, of EDTA solution 0.02 M (theoretical value : 1 ml $\hat{=}$ 1.3076 mg of zinc) necessary for the titration of 50.0 mg of Zn ($50.0 : 1.3076 = 38.2379$).
- V is the volume, in millilitres, of EDTA solution (3.13) used for the titration of 25.0 ml of standard zinc solution (3.12) ($2 \text{ mg} \times 25.0 = 50.0 \text{ mg}$).
- 3.14 *Litmus paper*.
- 3.15 *Indicator paper* for pH within the range 5 to 6, with 0.2 unit intervals.
- 3.16 *Dithizone*, ethanolic solution, 0.25 g/l.
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.