
INTERNATIONAL STANDARD



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Zinc alloys – Determination of magnesium content – Atomic absorption method

Alliages de zinc – Dosage du magnésium – Méthode par absorption atomique

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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.

International Standard ISO 3750 was drawn up by Technical Committee ISO/TC 18, *Zinc and zinc alloys*, and was circulated to the Member Bodies in June 1975.

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It has been approved by the Member Bodies of the following countries:

Australia	Germany	South Africa, Rep of
Austria	India	Spain
Belgium	Ireland	Turkey
Brazil	Italy	United Kingdom
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Czechoslovakia	Mexico	Yugoslavia
Egypt, Arab Rep. of	Norway	
France	Poland	

No Member Body expressed disapproval of the document.

Zinc alloys – Determination of magnesium content – Atomic absorption method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an atomic absorption method for the determination of the magnesium content of zinc alloys.

This method is applicable to the zinc alloys defined in ISO/R 301. It is suitable for the determination of magnesium contents between 0,01 and 0,08 %.

2 REFERENCES

ISO/R 301, *Zinc alloy ingots*.

ISO 3752, *Zinc alloy ingots – Selection and preparation of samples for chemical analysis*.

3 PRINCIPLE

Dissolution of a test portion in a hydrochloric acid-nitric acid medium. Suitable dilution and addition of a lanthanum salt to attenuate the effect of aluminium.

Atomization of the solution in an air-acetylene (or nitrous oxide-acetylene) flame and determination of magnesium content by spectrophotometric measurement of the atomic absorption at the wavelength 285,21 nm emitted by a magnesium hollow-cathode lamp.

4 REAGENTS

All reagents shall be of analytical reagent grade. Distilled water or demineralized water of equivalent purity shall be used for preparing the solutions.

4.1 Hydrochloric acid, ρ 1,19 g/ml.

4.2 Nitric acid, ρ 1,4 g/ml.

4.3 Hydrochloric acid-nitric acid mixture.

Mix 180 ml of the hydrochloric acid (4.1) and 4 ml of the nitric acid (4.2).

4.4 Lanthanum, 5 % solution.

Dissolve 29,5 g of lanthanum oxide (La_2O_3) in 25 ml of the hydrochloric acid (4.1). Transfer the solution quantitatively to a 500 ml volumetric flask. Dilute to the mark with water. Mix.

4.5 Zinc, 10 g/l solution.

Attack 10 g of zinc (99,99 %), free from magnesium¹⁾, with 60 ml of the acid mixture (4.3). Evaporate to a syrupy consistency. Take up with water and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water. Mix.

4.6 Aluminium, 0,8 g/l solution.

Attack 0,8 g of aluminium (99,99 %), free from magnesium¹⁾, with the minimum of hydrochloric acid (4.1). Transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water. Mix.

4.7 Magnesium, standard solution corresponding to 0,5 g of Mg per litre.

Into a 250 ml beaker covered with a watch-glass, pour 20 ml of water, then 5 ml of the hydrochloric acid (4.1). Introduce 0,5 g of magnesium of purity at least 99,95 %, weighed to $\pm 0,001$ g. After dissolution of the metal, cool and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water. Mix.

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

1) To check that the magnesium content of solution (4.5) or (4.6) is low enough, proceed as follows :

Introduce into two 100 ml volumetric flasks 0 and 0,5 ml respectively of the standard magnesium solution (4.7) corresponding to 0 and 0,05 mg/l of magnesium.

Dilute to the mark with water. Mix.

Compare solution (4.5) or (4.6) with these calibration solutions by spectrophotometric measurement of the atomic absorption as specified in 7.4.

The response shall not exceed that of the solution 0,05 mg/l.

4.8 Magnesium, standard solution corresponding to 0,01 g of Mg per litre.

Transfer exactly 20 ml of the standard magnesium solution (4.7) to a 1 000 ml one-mark volumetric flask. Add 5 ml of the hydrochloric acid (4.1). Dilute to the mark with water. Mix.

1 ml of this standard solution contains 0,01 mg of magnesium.

5 APPARATUS

Ordinary laboratory equipment and

5.1 Atomic absorption spectrophotometer, fitted with a magnesium hollow-cathode lamp and a burner supplied with an air-acetylene mixture¹⁾. The optical path length within the flame is within the limits 5 to 10 cm.

6 SAMPLING

Sampling shall be carried out in accordance with the specifications of ISO 3752.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, a 5 g test portion.

7.2 Preparation of the calibration solutions

7.2.1 To each of a series of eight 100 ml volumetric flasks, transfer 5 ml of the hydrochloric acid (4.1).

7.2.2 Add 20 ml of the zinc solution (4.5), 10 ml of the aluminium solution (4.6) and 0 – 2 – 5 – 7 – 10 – 12 – 14 – 16 ml of the standard magnesium solution (4.8), corresponding respectively to 0 – 0,01 – 0,025 – 0,035 – 0,05 – 0,06 – 0,07 – 0,08 % of magnesium.

7.2.3 Add 5 ml of the lanthanum solution (4.4). Dilute to the mark with water and mix.

7.3 Preparation of test solution.

7.3.1 Transfer the test portion to a 250 ml beaker fitted with a watch-glass and attack with 40 ml of the acid mixture (4.3).

7.3.2 Evaporate to a syrupy consistency, then take up with 40 to 50 ml of water.

7.3.3 Add 25 ml of the hydrochloric acid (4.1) and warm gently to dissolve any salts.

7.3.4 Transfer to a 250 ml volumetric flask. Dilute to the mark with water and mix.

7.3.5 Transfer exactly 10 ml of this solution to a 100 ml volumetric flask. Add 4 ml of the hydrochloric acid (4.1) and 5 ml of the lanthanum solution (4.4). Dilute to the mark with water. Mix.

7.4 Spectrophotometric measurements

Measure the atomic absorption of the 285,21 nm line emitted by the magnesium hollow-cathode lamp, taking simultaneous readings on the test solution and on the calibration solutions in a sequence such that it is ensured that the setting conditions of the burner and the apparatus do not change during the series of readings. Between each passage of the solution, atomize a little water to clean the burner.

To obtain better reproducibility and greater sensitivity, it is recommended that a slightly reducing flame be used.

8 EXPRESSION OF RESULTS

Plot the results of the spectrophotometric measurements of the calibration solutions against the concentration. Determine the concentration of the test solution by reference to the calibration curve thus obtained. Deduce the magnesium content of the sample from this determination.

9 TEST REPORT

The test report shall include the following particulars :

- reference to the method employed;
- the results obtained and the form in which they are expressed;
- all information necessary to identify the sample completely;
- details of any operating procedure not specified in this International Standard or optional procedure, and any circumstances which may have affected the results.

1) If the appropriate apparatus is available and after preliminary agreement between the interested parties, a nitrous oxide-acetylene flame may be used instead of the air-acetylene flame.