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Zinc alloys — Determination of magnesium content — Flame atomic absorption spectrometric method

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

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3750 was prepared by Technical Committee ISO/TC 18, *Zinc and zinc alloys*, Subcommittee SC 1, *Methods of sampling and analysis of zinc and zinc alloys*.

This second edition cancels and replaces the first edition (ISO 3750:1976), which has been technically revised. It is based on European standard EN 12441-2:2001, *Zinc and zinc alloys — Chemical analysis — Part 2: Determination of magnesium in zinc alloys — Flame atomic absorption spectrometric method.*

Zinc alloys — Determination of magnesium content — Flame atomic absorption spectrometric method

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of magnesium in zinc alloys. It is applicable to the products specified in ISO 301 and ISO 752.

It is suitable for the determination of magnesium contents (mass fractions) between 0,002 % and 0,08 %.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 301, *Zinc alloy ingots intended for casting*

ISO 752, *Zinc ingots*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

ISO 20081, *Zinc and zinc alloys — Method of sampling — Specifications*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20081 and the following apply.

3.1
flame atomic absorption spectrometry
measurement of the absorption of electromagnetic radiation, emitted by an element at a determined wavelength, by an absorbent medium (flame) formed of atoms of the same element that are in the ground state

NOTE Each element absorbs radiation of specific wavelengths and the intensity of the absorbed radiation is proportional to the concentration of the said element.

4 Principle

A sample of the alloy is dissolved in a mixture of hydrochloric and nitric acid and, after adequate dilution and atomisation of the solution in an air/acetylene (or nitrous oxide/acetylene) flame, the content of magnesium is determined by atomic absorption spectrometry at a wavelength of 285,21 nm.

5 Reagents

5.1 General

During the test, use only reagents of known or analytical grade and distilled or demineralised water.

5.2 Hydrochloric acid, $\rho = 1{,}19$ g/ml

5.3 Nitric acid, $\rho = 1{,}4$ g/ml

5.4 Hydrochloric acid/nitric acid mixture

Mix 180 volumes of hydrochloric acid (5.2) with 4 volumes of nitric acid (5.3). This mixture shall be freshly prepared just before use.

5.5 Lanthanum, 5 % solution

Put 29,5 g of lanthanum oxide (La_2O_3) in a 400 ml beaker. Add 5 ml of water, then carefully add 50 ml of hydrochloric acid (5.2). After dissolution, cool to room temperature. Transfer quantitatively to a 500 ml volumetric flask. Dilute to the mark with water and mix.

5.6 Zinc, 10 g/l solution

Dissolve 10 g of zinc (99,99 %), free of magnesium (see 8.3.1), with 60 ml of the acid mixture (5.4). 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 and mix.

5.7 Aluminium, 1,0 g/l solution

To 1,0 g of aluminium (99,99 %), free of magnesium (see 8.3.1), add 10 ml of water and then dissolve with a minimum of hydrochloric acid (5.2). Heat gently to aid dissolution. Cool to room temperature. Transfer quantitatively to a 100 ml volumetric flask. Dilute to the mark with water and mix. After verification of the magnesium contents (see 8.3.1), transfer exactly 50 ml to a 500 ml volumetric flask. Dilute to the mark with water and mix.

5.8 Magnesium, 0,5 g/l standard solution

Into a 250 ml beaker covered with a watch-glass, pour 20 ml of water, then 5 ml of the hydrochloric acid (5.2). Add 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 and mix.

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

5.9 Magnesium, standard solution, 0,01 g/l

Transfer exactly 20 ml of the magnesium solution (5.8) to a 1 000 ml volumetric flask. Add 5 ml of the hydrochloric acid (5.2). Dilute to the mark with water and mix.

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

5.10 Magnesium standard solution, 0,001 g/l

Transfer exactly 50 ml of the standard magnesium solution (5.9) to a 500 ml volumetric flask. Add 5 ml of the hydrochloric acid (5.2). Dilute to the mark with water and mix.

1 ml of this solution contains 0,001 mg of magnesium.

5.11 Aqua regia

Mix 3 volumes of hydrochloric acid (5.2) with 1 volume of nitric acid (5.3).

6 Apparatus

6.1 General

All glassware used for the preparation of the solutions and for the implementation of the method shall be cleaned with boiling aqua regia (5.11) prior to use.

6.2 Specific equipment

In addition to standard laboratory apparatus, an atomic absorption spectrometer, equipped with a premix burner, with facilities for using the oxidizer/fuel combinations of air/acetylene or nitrous oxide/acetylene, shall be used.

Excitation sources should be operated in accordance with the manufacturer's recommendations. The optical path length within the flame should be between 5 cm and 10 cm.

7 Sampling

The test sample shall be selected and prepared in accordance with the procedure given in ISO 20081.

8 Procedure

8.1 Test portion

Weigh 5 g of the test sample to the nearest 0,001 g.

8.2 Preparation of the test solution

8.2.1 Put the test portion (8.1) in a 250 ml beaker fitted with a watch-glass and dissolve by carefully adding 40 ml of the acid mixture (5.4). Evaporate carefully to a syrupy consistency and, after cooling to room temperature, dilute with 40 ml to 50 ml of water. Add 25 ml of the hydrochloric acid (5.2) and warm gently to dissolve any salts.

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

8.2.3 Transfer exactly 10 ml of this solution (8.2.2) to a 100 ml volumetric flask. Add 4 ml of the hydrochloric acid (5.2) and 5 ml of the lanthanum solution (5.5). Dilute to the mark with water and mix.

8.3 Preparation of the calibration solutions

8.3.1 To verify that the magnesium content of solutions (5.6) and (5.7) is low enough, proceed as follows:

— introduce, into two 100 ml volumetric flasks, 0 ml and 2 ml respectively of the standard magnesium solution (5.10) corresponding to 0 ml/l and 0,02 mg/l of magnesium;

— dilute to the mark with water and mix;

— compare solutions (5.6) and (5.7) with these calibration solutions by spectrophotometric measurement of the atomic absorption as specified in 8.4.

The spectrometric measurement response shall not exceed that of the 0,02 mg/l solution.

8.3.2 Introduce 5 ml of the hydrochloric acid (5.2) into each flask of a series of eight 100 ml volumetric flasks.

8.3.3 Add a ml of the zinc solution (5.6) and b ml of the aluminium solution (5.7) to each flask, according to Table 1.

Table 1 — Volumes a and b

Aluminium content (mass fraction) % (mass fraction)	a ml	b ml
Smaller than 0,05	20	0
Between 3,7 and 6,0	19	10
Between 8,0 and 11,0	18	20
Between 25 and 28,0	15	50

8.3.4 Then add 0,00 ml, 2,00 ml, 5,00 ml, 7,00 ml, 10,0 ml, 12,0 ml, 14,0 ml and 16,0 ml aliquots of the standard magnesium solution A (5.9). These aliquots correspond to contents (mass fractions) in the test portion of 0,00 %, 0,01 %, 0,025 %, 0,035 %, 0,05 %, 0,06 %, 0,07 % and 0,08 % of magnesium.

For the analysis of products ZL6 and ZP0610, prepare 2 additional calibration solutions corresponding to magnesium contents (mass fractions) of 0,002 % and 0,005 %, by taking respectively volumes of 4,00 ml and 10,00 ml of the standard magnesium solution (5.10).

8.3.5 Add 5 ml of the lanthanum solution (5.5) to each flask. Dilute to the mark with water and mix.

8.4 Spectrometric measurements

Measure the absorbances of the calibration solutions and the test solution(s) by taking alternate readings to ensure that the settings of the burner and of the apparatus do not change during the readings.

The wavelength of the line used shall be 285,21 nm.

To comply with the concentration ranges recommended by the manufacturer of the apparatus, the same dilutions for the calibration solutions and the test solution(s) shall be made if necessary.

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

9 Calculation and expression of results

9.1 Method of calculation

Establish a calibration graph by plotting the measured absorbances of the calibration solutions against their respective contents (mass fractions).

Determine, from the measured absorbance of the test solution, the associated amount of magnesium from the calibration graph. If a number of determinations are carried out then the mean of all results shall be calculated.

The results shall be expressed as specified in ISO 301 and ISO 752.

9.2 Precision

A planned trial of this method was carried out by 10 laboratories, using 7 samples with 4 levels of magnesium contents, each laboratory making three determinations of magnesium content in each sample (see Notes 1 and 2).

NOTE 1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725-1; i.e. one operator, same apparatus, identical operating conditions, same calibration and a minimum period of time.

NOTE 2 The third determination was carried out at a different time (on a different day), by the same operator as in Note 1, using the same apparatus and a different calibration.

The details of the samples used and the mean results obtained are given in Tables A.1 and A.2.

The results obtained were treated statistically in accordance with ISO 5725-2 and ISO 5725-3.

The data obtained showed a logarithmic relationship between the magnesium content and the repeatability limit (r) and reproducibility limits (R_w and R) of the test results (see Note 3), as summarised in Table 2. The graphical representation of the data is shown in Figure B.1.

NOTE 3 From the two values obtained in day 1, the repeatability limit (r) and the reproducibility limit (R) were calculated using the procedure specified in ISO 5725-3. From the first value obtained on day 1 and the value obtained on day 2, the within-laboratory reproducibility limit (R_w] day was calculated using the procedure specified in ISO 5725-3.

Table 2 — Repeatability limit and reproducibility limits

Magnesium content % (mass fraction)	Repeatability limit	Reproducibility limits	
	r	R_w	R
0,01	0,000 2	0,000 3	0,003 0
0,02	0,000 4	0,000 4	0,002 5
0,05	0,001 0	0,000 7	0,002 0