BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ZINC AND ZINC BASE ALLOYS FOR DIE CASTINGS

PART 6 DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROMETRIC METHOD

[Second Revision of IS 2600 (Part 6)]

ICS 77.120.60

| Methods of Chemical Analysis of Metals | Last date of comments |
|--|-----------------------|
| Sectional Committee, MTD 34 | XX September 2023 |

NATIONAL FOREWORD

This draft standard which is identical to ISO 3750:2006 'Zinc alloys — Determination of magnesium content — Flame atomic absorption spectrometric method' issued by the International Organization for Standardization (ISO), and subject to its finalization, is to be adopted by the Bureau of Indian Standards on the recommendation of the Methods of Chemical Analysis of Metals Sectional Committee and approval of the Metallurgical Engineering Division Council.

This standard was first published in 1964and subsequently revised in 2022. The Second Revision of this standard has been undertaken to align with the latest version ISO 3750:2006to harmonize it with the latest developments that have taken place at international level.

Other parts in this IS series are:

| Part 1 | Determination of copper, | iron, nickel, tir | n and thallium b | y spectrophotometric, |
|--------|--------------------------|-------------------|------------------|-----------------------|
|--------|--------------------------|-------------------|------------------|-----------------------|

- Part 2 Determination of copper, iron, lead and cadmium by atomic absorption spectrometric method,
- Part 3 Determination of indium by atomic absorption spectrometric method,
- Part 4 Determination of chromium by spectrometric method,
- Part 5 Analysis by inductively coupled plasma optical emission spectrometry, and
- Part 7 Determination of aluminium by titrimetric method

The text of ISO standard has been approved as suitable for publication as in Indian Standard without deviations. Certain terminologies and conventions are, however, not identical with those used in Indian Standard. Attention is especially drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, it should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker while in Indian Standards the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their respective places are listed below along with their degree of equivalence for the editions indicated:

| International Standard | Corresponding Indian Standard | Degree of Equivalence |
|---|---|-----------------------|
| ISO 301 : 2006 Zinc alloy ingots intended for casting | IS 713 : 1981 Specification for zinc base alloy ingots for die casting (<i>second revision</i>) | Not Equivalent |
| ISO 752 : 2004 Zinc ingots | IS 209 : 1992 Zinc ingot — Specification (<i>fourth revision</i>) | Not Equivalent |
| ISO 5725-1 : 2023 Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions | IS 15393 (Part 1) : 2003 Accuracy (trueness and precision) of measurement methods and results: Part 1 General principles and definitions | Identical |
| ISO 5725-3 : 2023 Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method | Accuracy (trueness and precision) of measurement methods and results: Part 3 Intermediate measures of the precision of a standard measurement method | Identical |

The Technical Committee responsible for the preparation of this standard has reviewed the provisions of following International Standards referred in these adopted standards and decided their acceptability for use in conjunction with this standard.

| International Standard | Title |
|------------------------|--|
| ISO 5725-2 : 2019 | 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 20081 : 2005 | Zinc and zinc alloys — Method of sampling — Specifications |

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

The Scope of the standard is as follows:

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 %.

The complete document/text of ISO 3750 : 2006 'Zinc alloys — Determination of magnesium content — Flame atomic absorption spectrometric method'may be made available, on request to:

/Sanjiv Maini
/Senior Director,
Scientist 'F' & Head
/Metallurgical Engg. Department
/Bureau of Indian Standards,
/Manak Bhavan, 9, B.S.Z. Marg,
New Delhi-110002
E-mail: mtd@bis.gov.in, mtd34@bis.gov.in
Tel: + 91 11 23231085

INTERNATIONAL STANDARD

Second edition 2006-06-01

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 ISO 3750:2006(E)

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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

3.1

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

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.

| 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 |

Table 1 — Volumes *a* and *b*

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.

| Magnesium content | Repeatability limit | Reproduci | bility limits |
|-------------------|---------------------|----------------|---------------|
| % (mass fraction) | 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 |

Table 2 — Repeatability limit and reproducibility limits

10 Test report

The test report shall include the following information:

- a) identification of the sample;
- b) test method used, (i.e. a reference to this International Standard);
- c) magnesium content, expressed as percentage by mass, giving, where possible, the results for the individual and mean values;
- d) any unusual occurrence during the determination;
- e) any steps in the procedure beyond those specified in this International Standard, and any circumstances that may have affected the results;
- f) date of test report;
- g) name of laboratory or testing organisation;
- h) signature of the laboratory manager or other responsible person.

Annex A (informative)

Additional information on international cooperative tests

Table A.1 was derived from the results of International analytical trials carried out in 1999 on 7 test samples in 6 countries involving 10 laboratories.

The results of the trials are shown in Table A.1.

The composition of the test samples is shown in Table A.2.

| | Magnesium content % (mass fraction) | | Precision data | | | |
|--------|--|-------------|-----------------|-----------------|---------|--|
| Sample | | | Repeatability | Reproducibility | | |
| | Reference value | Found value | r | R _w | R | |
| ZL 2 | 0,037 | 0,039 3 | 0,000 7 | 0,000 6 | 0,002 7 | |
| ZL 3 | 0,035 | 0,037 8 | 0,000 7 | 0,000 4 | 0,001 6 | |
| ZL 5 | 0,036 | 0,038 8 | 0,000 8 | 0,000 8 | 0,002 8 | |
| ZL 6 | < 0,000 5 | 0,000 2 | | | | |
| ZL 8 | 0,021 | 0,022 0 | 0,000 6 | 0,000 4 | 0,002 1 | |
| ZL 12 | 0,023 | 0,024 7 | 0,000 7 | 0,000 8 | 0,002 1 | |
| ZL 27 | 0,012 | 0,013 1 | 0,000 2 0,000 3 | | 0,003 3 | |

Table A.1 — Detailed results obtained in the interlaboratory test

| Table A.2 | — Test sample | s used in th | e interlabora | tory test |
|-----------|---------------|--------------|---------------|-----------|

Contents in % (mass fraction)

| Sample | Mg | AI | Cu | Fe | Pb | Sn | Cd | Ni | Si |
|--------|-----------|-------|--------|-------|-------|---------|---------|---------|--------|
| ZL 2 | 0,037 | 4,00 | 2,9 | 0,006 | 0,002 | < 0,001 | < 0,001 | < 0,001 | < 0,02 |
| ZL 3 | 0,035 | 4,00 | < 0,01 | 0,002 | 0,002 | < 0,001 | < 0,001 | < 0,001 | < 0,02 |
| ZL 5 | 0,036 | 3,97 | 0,8 | 0,003 | 0,002 | < 0,001 | < 0,001 | < 0,001 | < 0,02 |
| ZL 6 | < 0,000 5 | 5,75 | 1,3 | 0,006 | 0,002 | < 0,001 | < 0,001 | — | < 0,02 |
| ZL 8 | 0,021 | 8,37 | 1,1 | 0,003 | 0,003 | < 0,001 | < 0,001 | < 0,001 | < 0,02 |
| ZL 12 | 0,023 | 11,00 | 0,8 | 0,01 | 0,002 | < 0,001 | < 0,001 | — | < 0,02 |
| ZL 27 | 0,012 | 26,83 | 2,3 | 0,04 | 0,002 | < 0,001 | < 0,001 | — | < 0,02 |

Annex B

(informative)

Graphical representation of precision data

Figure B.1 indicates the logarithmic relationships between the magnesium content and the repeatability (r) and reproducibility limits (R_w and R).



where w_{Mg} is the average magnesium content, expressed as a percentage by mass, obtained from the three determinations in each laboratory.

Key

- X Magnesium content in percent
- Y Precision in percent

Figure B.1 — Logarithmic relationships between magnesium content and the repeatability (r) and reproducibility limits (R_w and R)

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