
**Magnesium and its alloys —
Determination of lead and cadmium**

Magnésium et alliages de magnésium — Dosage du plomb et du cadmium



Reference number
ISO 11707:2011(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 11707 was prepared by Technical Committee ISO/TC 79, *Light metals and their alloys*, Subcommittee SC 5, *Magnesium and alloys of cast or wrought magnesium*.

Magnesium and its alloys — Determination of lead and cadmium

1 Scope

This International Standard specifies wet analytical methods for lead and cadmium. There are two methods for the simultaneous determination of lead and cadmium in magnesium and its alloys. Method A uses a matrix-matching technique for unalloyed magnesium and its alloys by inductively coupled plasma/atomic emission spectrometry (ICP/AES) or flame atomic absorption spectrometry (FAAS). Method B uses an extraction method for magnesium alloys with pretreatment procedures. Generally, Method A is recommended; also, Method B is designated where the matrix-matching method cannot be adopted due to complicated preparation of the assay standard solutions or where the combination of solvent extraction and FAAS is useful for analysts. These methods are applicable to the determination of lead and cadmium in the ranges of mass fractions of 0,000 5 % to 0,04 % and 0,000 5 % to 0,07 %, respectively.

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 648:2008, *Laboratory glassware — Single-volume pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

3 Method A — Matrix-matching method

3.1 Principle

The sample is dissolved in a mixture of nitric and hydrochloric acids and diluted to a suitable volume. Lead and cadmium are determined by ICP/AES or FAAS at the wavelengths noted in Table 1.

Table 1 — Spectral lines

Element	Wavelength nm	
	ICP/AES	FAAS
Pb	220,353	217,0
Cd	228,802	228,8

3.2 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled water or water of equivalent purity. The solution shall be freshly prepared.

3.2.1 Hydrochloric acid, ρ : 1,17 g/mL, 35 % to 37 %.

3.2.2 Nitric acid, ρ : 1,42 g/mL, 69 % to 71 %.

3.2.3 Lead standard solution (100 mg Pb/L).

Weigh 0,25 g of lead (99,99 % or higher in purity) to the nearest 0,1 mg, dissolve in 30 mL of nitric acid (1 + 1), heat to complete exhaustion of brown-coloured NO_x gas and cool it. Transfer the solution quantitatively to a calibrated 250 mL volumetric flask, then dilute to the mark with water and mix. Using a volumetric pipette, transfer 10 mL of the prepared solution to a 100 mL volumetric flask, thus allowing a ten-fold dilution. Dilute to the mark with water and mix. Keep the flask at the same temperature throughout.

3.2.4 Cadmium standard solution (100 mg Cd/L).

Weigh 0,25 g of cadmium (99,99 % or higher in purity) to a digit of 0,1 mg, dissolve in 30 mL of nitric acid (1 + 1), heat to complete exhaustion of brown-coloured NO_x gas and cool it. Transfer the solution quantitatively to a calibrated 250 mL volumetric flask, then dilute to the mark with water and mix. Using a volumetric pipette, transfer 10 mL of the prepared solution to a 100 mL volumetric flask, thus allowing a ten-fold dilution. Dilute to the mark with water and mix. Keep the flask at the same temperature throughout.

3.3 Apparatus

All volumetric glassware shall be of class A and calibrated in accordance with ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory apparatus is also acceptable.

3.3.1 Inductively coupled plasma/atomic emission spectrometry (ICP/AES).

The ICP/AES used will be satisfactory after optimizing in accordance with the manufacturer's instructions.

3.3.2 Flame atomic absorption spectrometer (FAAS).

The FAAS used will be satisfactory after optimizing in accordance with the manufacturer's instructions.

3.4 Procedure

3.4.1 Mass of sample

Weigh 1,0 g of the sample to a digit of 0,1 mg.

3.4.2 Preparation of sample solution

Weigh out the sample and transfer it into a 250 mL beaker. Add 20 mL of water. After a mixture of 15 mL of hydrochloric acid (3.2.1) and 5 mL of nitric acid (3.2.2) has been added slowly, cover the beaker with a watch-glass and heat it on a hotplate to complete dissolution.

Take up the residue in 25 mL of water, warm gently to complete the solution and cool.

Filter, if necessary, through a paper-pulp pad and wash the precipitate with a small amount of water. Add the washings to the sample solution.

Transfer the solution to a 100 mL volumetric flask and dilute to the mark with water and mix.

Spray the solution into ICP/AES plasma or an AAS flame to measure the emission intensity or the absorbance of lead and cadmium therein.

3.4.3 Reagent blank test

Add matrix elements, such as Mg, Al, Zn and other elements, so that the concentration of the major components are the same as those of the sample. Magnesium oxide (99,99 % or higher in purity), aluminum, zinc and other metals and compounds of high purities should be used. Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents as with the sample.

3.5 Calibration

Weigh 1,658 g of magnesium oxide (99.99 % or higher in purity) and transfer it to a 250 mL beaker. Add 20 mL of water. After a mixture of 15 mL of hydrochloric acid (3.2.1) and 5 mL of nitric acid (3.2.2) has been added slowly, cover the beaker with a watch-glass. Heat it on a hotplate for complete dissolution.

Add matrix elements, such as Mg, Al, Zn and other elements.

Add 0 mL, 1 mL, 3 mL and 5 mL of lead (3.2.3) and cadmium (3.2.4) standard stock solutions to the beaker using volumetric pipettes, transfer the solutions to 100 mL volumetric flasks, dilute them to the mark with water and mix.

Spray them into ICP/AES plasma or an AAS flame and plot calibration curves by measuring emission or absorption strength at the wavelengths noted in Table 1.

3.6 Calculation

Obtain the mass of lead and cadmium from the calibration curve prepared (3.5). Calculate the mass fractions of lead and cadmium according to the following equations.

$$w_{\text{Pb}} = \frac{(A_1 - A_2) \times V \times 10^{-4}}{m}$$

$$w_{\text{Cd}} = \frac{(A_1 - A_2) \times V \times 10^{-4}}{m}$$

where

w_{Pb} , w_{Cd}	are the mass fractions of Pb and Cd in the sample, in percent (%);
A_1	is the concentration of the element in the test solution, in milligrams per litre (mg/L);
A_2	is the concentration of the element in the reagent blank solution, in milligrams per litre (mg/L);
V	is the final dilution volume of the test solution, in millilitres (mL);
m	is the mass of sample weighed, in grams (g).

4 Determination Method B — Extraction method

4.1 Principle

Dissolve the sample in a mixture of nitric and hydrochloric acids, add ammonium citrate solution and adjust the pH of the solution with ammonia solution and nitric acid. Add potassium cyanide to separate lead and cadmium selectively from matrixes with diphenylthiocarbazone (dithizone) in chloroform and dilute to a suitable volume. Lead and cadmium are determined by ICP/AES or FAAS.

4.2 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity. The solutions should be freshly prepared prior to use.

4.2.1 Nitric acid (1:1).

4.2.2 Nitric acid (1:10).

4.2.3 Ammonia solution (1:10).

4.2.4 Perchloric acid, ρ : 1,54 g/mL, 60 to 62 %.

4.2.5 Ammonium citrate solution.

Dissolve 250 g of ammonium citrate in 1 000 mL of water.

4.2.6 Sodium hydroxide solution.

Dissolve 125 g of sodium hydroxide in 1 000 mL of water.

4.2.7 Potassium cyanide solution.

Dissolve 50 g of potassium cyanide in 500 mL of water.

4.2.8 Dithizone solution in chloroform (0,2 %).

Dissolve 2 g of dithizone in 1 000 mL of chloroform. Prepare freshly just prior to use, and store in a brown bottle. Filter if necessary through a paper-pulp pad (5A)¹⁾. Inhalation and ingestion of chloroform are harmful.

WARNING — Please wear gloves and safety glasses and use good ventilation.

4.2.9 Dithizone solution in chloroform (0,02 %).

Dissolve 0,2 g of dithizone in 1 000 mL of chloroform. Prepare freshly just prior to use, and store in a brown bottle. Filter if necessary through a paper-pulp pad (5A).

4.3 Apparatus

4.3.1 pH meter.

4.3.2 Flame atomic absorption spectrometer (FAAS).

The FAAS used will be satisfactory after optimizing in accordance with the manufacturer's instructions.

4.3.3 Inductively coupled plasma/atomic emission spectrometry (ICP/AES).

The ICP/AES used will be satisfactory after optimizing in accordance with the manufacturer's instructions.

4.4 Procedure

4.4.1 Mass of sample

Weigh 1,0 g of the sample to a digit of 0,1 mg.

4.4.2 Preparation of sample solution

Weigh out the sample and transfer it into a 250 mL beaker. Add 20 mL of water. After a mixture of 15 mL of hydrochloric acid (3.2.1) and 5 mL of nitric acid (3.2.2) has been added slowly, cover the beaker with a watch-glass and heat it on a hotplate until complete dissolution.

Add 30 mL of ammonium citrate solution (4.2.5) and dilute with water until the volume of the solution reaches approximately 100 mL. Adjust the pH of the solution to alkaline with ammonia solution (4.2.3).

1) Paper-pulp pad (5A) is the trade name of a product supplied by ADVANTEC Co. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Add 3 mL of potassium cyanide solution (4.2.7) and adjust the pH of the solution to $9,2 \pm 0,1$ with ammonia solution (4.2.3) and nitric acid (4.2.2).

Transfer the solution into a separating funnel. Add 20 mL of 0,2 % dithizone-chloroform (4.2.8) and shake it for 5 min.

Allow to stand until separation of the chloroform layer and water layer is completed, which takes about 5 min. Transfer the lower layer of chloroform to a new beaker.

Repeat the steps in the two previous paragraphs. Add the lower layer of chloroform to the sample solution.

Add 10 mL of dithizone-chloroform (0,02 %) to a separating funnel and shake it again for 5 min. Allow to stand until separation of the layers is completed. Add the lower layer of chloroform to the sample solution again.

Heat the solution in a warm bath at 60 °C to 80 °C for complete evaporation of chloroform to dryness.

Add 10 mL of nitric acid and 5 mL of perchloric acid to the dried beaker. Heat it to completely evaporate the liquids and dry the beaker. If the organic compounds are not fully decomposed, add 5 mL of nitric acid.

Add 20 mL of nitric acid (4.2.1) to the beaker, dissolve the deposited salts, boil it gently for 1 min to 2 min, and cool it to room temperature.

Transfer the contents quantitatively to the calibrated 100 mL volumetric flask. Dilute to the marked line with water and mix.

Spray the solution into ICP/AES plasma or an AAS flame and measure the emission intensity or the absorbance of lead and cadmium therein.

4.4.3 Reagent blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents as with the sample.

4.5 Calibration

Add 0 mL, 1 mL, 3 mL and 5 mL of lead (3.2.3) and cadmium (3.2.4) standard stock solutions to the beaker (see 3.5) using volumetric pipettes, and transfer the respective solutions to 100 mL volumetric flasks. Add 15 mL of nitric acid (3.2.2), dilute it to the marked line with water and mix.

Spray them into ICP/AES plasma or an AAS flame and plot calibration curves by measuring emission or absorption strength at the wavelengths noted in Table 1.

4.6 Calculation

Obtain the mass of lead and cadmium from the calibration curve prepared (4.5). Calculate the mass fractions of lead and cadmium according to the following equations.

$$w_{\text{Pb}} = \frac{(A_1 - A_2) \times V \times 10^{-4}}{m}$$

$$w_{\text{Cd}} = \frac{(A_1 - A_2) \times V \times 10^{-4}}{m}$$

where

- $w_{\text{Pb}}, w_{\text{Cd}}$ are the mass fractions in the sample, in percent (%);
- A_1 is the concentration of the element in the test solution, in milligrams per litre (mg/L);
- A_2 is the concentration of the element in the reagent blank solution, in milligrams per litre (mg/L);
- V is the final dilution volume of the test solution, in millilitres (mL);
- m is the mass of sample weighed, in grams (g).

5 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) all information necessary for the identification of the sample, the laboratory and the date of analysis;
- c) the method used by reference to this International Standard;
- d) the results and the form in which they are expressed;
- e) the analytical spectral line used;
- f) any unusual features noted during the determination;
- g) any operation not specified in this International Standard, or any optical operation which may have influenced the results.

Annex A (informative) Inter-laboratory test results

1) Test samples: 3 Mg-Al-Zn Certified Reference Materials (CRMs) from MBH

CRM-1: C65X MGB 10 (Mg/Al/Zn)

CRM-2: C65X MGA 10 (Mg/Al/Zn)

CRM-3: C65X MGA 50 (Mg/Al/Zn)

2) Test method: Method B

3) Participants: 5 domestic institutes (KOREA)

4) Results

Table A.1 — Inter-laboratory test results for lead

Participating laboratory	ICP			AAS		
	Pb (mg/kg)			Pb (mg/kg)		
	CRM-1 (Pb: 170 ± 10)	CRM-2 (Pb: 120 ± 10)	CRM-3 (Pb: 430 ± 20)	CRM-1 (Pb: 170 ± 10)	CRM-2 (Pb: 120 ± 10)	CRM-3 (Pb: 430 ± 20)
A	168,5	120,1	432,2	167,3	120,1	434,0
B	171,0	119,7	437,0	176,3	120,5	445,3
C	174,6	134,8	467,0	182,0	143,7	470,6
D	165,9	132,4	462,1	157,1	122,4	438,4
E	166,5	135,3	449,0	162,5	130,8	441,7
Average	169,3	128,5	449,5	169,0	127,5	446,0
SD	3,6	7,9	15,2	10,1	10,0	14,4

Table A.2 — Inter-laboratory test results for cadmium

Participating laboratory	ICP			AAS		
	Cd (mg/kg)			Cd (mg/kg)		
	CRM-1 (Cd: 700 ± 10)	CRM-2 (Cd: 154 ± 10)	CRM-3 (Cd: 35 ± 20)	CRM-1 (Cd: 700 ± 10)	CRM-2 (Cd: 154 ± 10)	CRM-3 (Cd: 35 ± 20)
A	698,0	153,0	33,7	701,2	155,2	35,2
B	667,2	153,0	33,0	692,0	149,8	33,8
C	682,3	159,3	37,8	645,8	147,0	35,0
D	676,4	160,2	38,3	675,0	155,1	36,4
E	676,7	158,8	37,0	680,0	157,8	36,6
Average	680,1	156,6	36,0	678,8	153,0	35,4
SD	11,4	3,6	2,4	21,1	4,4	1,1

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