INTERNATIONAL STANDARD

ISO 11707

First edition
2011-08-01

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)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.