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Magnesium and magnesium alloys — Determination of arsenic — Inductively coupled plasma optical emission spectrometric method

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Foreword

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This document was prepared by Technical Committee ISO/TC 79, *Light metals and their alloys*, Subcommittee SC 5, *Magnesium and alloys of cast or wrought magnesium.*

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Magnesium and magnesium alloys are one kind of light metallic materials and show several advantageous properties, such as low density, high specific stiffness and strength, good damping capacity, castability, weldability and machinability, etc. Arsenic as one of the hazardous impurities has negative effects on the environment and health, and needs to be strictly controlled. The sum of the mass contents of cadmium (Cd), mercury (Hg), arsenic (As) and chromium (Cr) given in ISO 8287 is defined to be less than 0,01 % in magnesium and its alloys, which needs to be inspected if used in food and medicine fields.

Magnesium and magnesium alloys — Determination of arsenic — Inductively coupled plasma optical emission spectrometric method

1 Scope

This document specifies an inductively coupled plasma optical emission spectrometric method (ICP-OES) for the determination of arsenic contents between 0,001 0 % (mass fraction) and 0,050 % (mass fraction) in magnesium and magnesium alloys.

The method is limited to magnesium alloys containing less than 0,6 % (mass fraction) of cerium, 0,1 % (mass fraction) of gadolinium, 0,2 % (mass fraction) of neodymium and 0,8 % (mass fraction) of zirconium.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Laboratory glassware — Single-volume pipettes*

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

After dissolution of a test sample with nitric acid and hydrochloric acid, the solution is nebulized into an inductively coupled plasma optical emission spectrometer and the intensity of the arsenic emitted light is measured. The concentrations of arsenic in the test solutions are derived from a magnesium-based calibration curve.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696, or water of equivalent purity.

5.1 Pure magnesium, purity > 99,99 % (mass fraction), free from arsenic.

5.2 Pure arsenic, purity > 99,99 % (mass fraction).

5.3 Hydrochloric acid, ρ about 1,19 g/ml.

5.4 Nitric acid, ρ about 1,14 g/ml.

5.5 Hydrochloric acid solution 1 + 1

Add 500 ml of hydrochloric acid (5.3) to 500 ml of water and mix.

5.6 Nitric acid solution 1 + 1

Add 500 ml of nitric acid (5.4) to 500 ml of water and mix.

5.7 Arsenic standard solution, 1 g/l.

Weigh to the nearest 0,001 g, 0,5 g of pure arsenic (5.2) and transfer into a 250 ml beaker. Add 20 ml of nitric acid solution (5.6) and cover with a watch glass. Heat gently until the metal is dissolved. After cooling to room temperature, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1,0 mg of arsenic.

5.8 Arsenic standard solution, 0,1 g/l.

Transfer 10,00 ml of the arsenic standard solution (5.7) to a one-mark 100 ml volumetric flask. Add 10 ml of nitric acid solution (5.6). Dilute to the mark with water and mix well.

1 ml of this solution contains 0,1 mg of arsenic.

5.9 Arsenic standard solution, 0,01 g/l.

Transfer 10,00 ml of the arsenic standard solution (5.8) to a one-mark 100 ml volumetric flask. Add 10 ml of nitric acid solution (5.6). Dilute to the mark with water and mix well.

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

6 Apparatus

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

6.1 Inductively coupled plasma optical emission spectrometer

6.1.1 General

The instrument used first shall be optimized in accordance with the manufacturer's instructions and then shall meet the performance criteria given in 6.1.3 to 6.1.4.

6.1.2 Wavelength

This method does not specify any particular wavelength. Each laboratory shall carefully investigate the wavelengths available on its own equipment to find out the most suitable ones regarding the sensitivity and the absence of interferences.

A suggestion is given in Table 1 together with possible interferences. This wavelength has been carefully investigated. It is recommended to use As 188,980 nm because of its high sensitivity and relatively low interference.

Table 1 — Example of a wavelength for arsenic determination

Element	Wavelength nm	Possible interferences
As	188,980	Cr

6.1.3 Limit of detection

Calculate the limit of detection of arsenic in accordance with Annex A. The limit of detection should be equal to or less than 0,01 mg/l.

6.1.4 Linearity of the calibration curve

The linearity of the calibration curve is checked by calculating the corresponding correlation coefficient. This coefficient shall be higher than 0,999.

7 Sampling and sample preparation

Sampling shall be carried out in accordance with an appropriate national standard for magnesium. If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned with ethanol or acetone and then dried in air. The sample shall be in the form of fine drillings, chips or millings with a maximum thickness of 1 mm. In order to avoid oxidation of the surface, the chips shall be taken from an inner part of a bulk sample just before starting the analytical procedure.

8 Procedure

8.1 General

The same reagents should be used for the preparation of calibration solutions and sample solutions.

8.2 Test sample

Mix the sample well so that any portion weighed represents the average composition. Weigh, to the nearest 0,1 mg, 1,0 g of the test sample.

8.3 Preparation of the test solution

Place the test sample (see 8.2) into a 300 ml glass beaker.

Add about 50 ml of water, 15 ml of nitric acid solution (5.6) and 3 ml of hydrochloric acid solution (5.5). Cover with a watch glass and heat gently until the sample is completely dissolved. Continue heating until the volume of the remaining solution is about 50 ml.

Cool to room temperature and transfer into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

8.4 Preparation of the calibration solutions

Transfer into a series of 300 ml beakers the amounts of pure magnesium (5.1) necessary to match the matrix composition, weighed to the nearest 0,001 g. Dissolve it as described in 8.3. Transfer quantitatively into a series of 100 ml volumetric flasks.

Add the volumes of arsenic standard solutions (5.8) and (5.9) shown in Table 2, and 15 ml nitric acid solution (5.6) into each series of 100 ml volumetric flasks. Dilute to the mark with water and mix.

Table 2 — Calibration solutions for arsenic contents between 0,001 0 % and 0,050 %

Calibration solution label	Volume of arsenic standard solution (5.9) ml	Volume of arsenic standard solution (5.8) ml	Concentration of arsenic in the calibration solution (µg/ml)	Corresponding arsenic mass fraction in the test sample %
$^{S}0$	0	0	0	0
$^{S}1$	1,00	—	0,10	0,001 0
$^{S}2$	5,00	—	0,50	0,005 0
$^{S}3$	—	1,00	1,00	0,010
$^{S}4$	—	3,00	3,00	0,030
$^{S}5$	—	6,00	6,00	0,060

8.5 Adjustment of the apparatus

Start the inductively coupled plasma optimal emission spectrometer and let it stabilize in accordance with the manufacturer's instructions before any measurement. Optimize the instrument according to the manufacturer's instructions. Prepare the software to measure the intensity, and for the calculation of the mean value and coefficient of variation corresponding to the appropriate wavelength.

8.6 Measurement of the calibration solutions

Measure the absolute intensities at the appropriate wavelength beginning with the lowest calibration solution and ending up with the highest calibration solution.

Measure each of the calibration solutions three times and calculate the mean intensities.

Subtract the mean absolute intensity (Ic_0) of the zero member from the mean absolute intensity (Ic_i) of each calibration solution, in order to obtain the net absolute intensity (I_{cN}).

8.7 Calibration curve

Establish the calibration curve using the net intensities of arsenic on the Y-axis and the corresponding concentrations of arsenic on the X-axis, expressed in micrograms per millilitre.

Calculate the correlation coefficient of the calibration curve. This shall meet the specification given in 6.1.4.

8.8 Measurement of the test solution

Measure the absolute intensity of the test solution three times and calculate the mean intensity.

9 Expression of results

9.1 Method of calculation

Using the calibration curve (see 8.7) and the net absolute intensity of the test solution obtained in 8.8, calculate the concentration of arsenic in the test solution, expressed in micrograms per millilitre.

The mass fraction of arsenic, expressed as a percentage, W_i, is given by Formula (1):