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Magnesium and magnesium alloys — Determination of chromium — 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 the light metallic materials and show several advantageous properties, such as such as low density, high specific stiffness and strength, good damping capacity, castability, weldability and machinability, etc. Chromium, as one of the hazardous elements, has negative effects on the environment and health, so its content needs to be strictly controlled within a certain low content. 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 be inspected if used in food and medicine fields.

Magnesium and magnesium alloys — Determination of chromium — 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 chromium 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 1,0 % (mass fraction) of cerium, 3,2 % (mass fraction) of gadolinium, 0,2 % (mass fraction) of neodymium and 1,6 % (mass fraction) of manganese.

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 https://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 chromium emitted light is measured. The concentrations of chromium 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 chromium.

5.2 Potassium dichromate, reference reagent.

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

5.4 Nitric acid, ρ about 1,42 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 Chromium standard solution, 1 g/l.

Dry several grams of potassium dichromate (5.2) in an oven at 110 °C ± 5 °C for at least 2 h and cool to room temperature in a desiccator. Weigh, to the nearest 0,1 mg, 2,828 0 g of the dried potassium dichromate, transfer into a 500 ml glass beaker, add 20 ml water and 10 ml of hydrochloric acid solution (5.5), cover with a watch-glass and, if necessary, heat gently to complete the dissolution. Cool and transfer quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

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

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

Transfer 10,00 ml of the chromium standard solution (5.7) to a one-mark 100 ml volumetric flask. Add 10 ml of hydrochloric acid solution (5.5). Dilute to the mark with water and mix.

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

5.9 Chromium standard solution, 0,005 g/l.

Transfer 5,00 ml of the chromium standard solution (5.8) to a one-mark 100 ml volumetric flask. Add 10 ml of hydrochloric acid solution (5.5). Dilute to the mark with water and mix.

1 ml of this solution contains 0,005 mg of chromium.

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 the most suitable one regarding the sensitivity and 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 Cr 267,716 nm because of its high sensitivity and relatively low interference.

Table 1 — Examples of a wavelength for chromium determination

Element	Wavelength nm	Possible interferences
Cr	267,716	Mn, Ce, Gd

6.1.3 Limit of detection

Calculate the limit of detection of chromium in accordance with Annex A for the wavelength used. The limit of detection should be equal to or less than 0,005 µg/ml.

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, 0,5 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, 10 ml of nitric acid solution (5.6) in small portions 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 the solution to room temperature. Transfer quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

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. Cool the solution to room temperature. Transfer quantitatively into a series of 100 ml volumetric flasks.

Add the volumes of chromium standard solutions (5.8) and (5.9) shown in Table 2, into each 100 ml volumetric flask. Dilute to the mark with water and mix.

Table 2 — Calibration solutions for chromium in magnesium

Calibration solution label	Volume of chromium standard solution (5.9) ml	Volume of chromium standard solution (5.8) ml	Concentration of chromium in the calibration solution µg/ml	Corresponding chromium mass fraction in the test sample %
S_0	0,00	—	0,00	0
S_1	1,00	—	0,05	0,001 0
S_2	3,00	—	0,25	0,005 0
S_3	10,00	—	0,50	0,010
S_4	—	1,00	1,00	0,020
S_5	—	2,00	2,00	0,040
S_6	—	3,00	3,00	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 (I_{c0}) of the zero member from the mean absolute intensity (I_{ci}) 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 chromium on the Y-axis and the corresponding concentrations of chromium 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 Measurements 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 chromium in the test solution, expressed in micrograms per millilitre.