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Magnesium and magnesium alloys~~--~~ — Determination of sodium ~~--~~ — Inductively coupled plasma optical emission spectrometric method

~~Magnésiumetalliages de magnésium - Détermination du sodium - Méthode par spectrométried'émissionatomique avec source à plasma induit~~

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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~~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. Sodium is, as one of the hazardous impurities leading to, creates hot brittleness of magnesium and magnesium alloys, thereby producing crackcracks during forging and rolling.. Sodium content should be controlled and monitored in order to check if its content remains at trace level. In ISO 8287:2021, sodium contents are specified to be less than 0,001- % or less than 0,01 %.

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

1 Scope

This document specifies an inductively coupled plasma atomic emission spectrometric method (ICP-OES) for the determination of sodium contents between 0,00 2002 % (mass fraction) and 0,050 % (mass fraction) in magnesium and magnesium alloys.

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 ~~terminological~~terminology databases for use in standardization at the following addresses:

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

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

4 Principle

After dissolution of a test portion with nitric acid and hydrochloric acid, the solution is nebulized into an inductively coupled plasma optical emission ~~spectrometry (ICP-OES)~~spectrometer and the intensity of the ~~sodium~~ emitted light from sodium is measured. The concentrations of sodium in the test solutions are derived from magnesium-based calibration curves.

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 sodium.

5.2 Sodium chloride, purity ≥99,99 % (mass fraction).

5.3 Hydrochloric acid, ρ about 1,~~19g~~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)~~(5.3) to 500 ml of water and mix.

5.6 Nitric acid solution 1 + 1.

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

5.7 Sodium standard solution (1 g~~/l~~).

Weigh, to the nearest 1 mg, 2,543 0 g of sodium chloride ~~(5.2)~~(5.2) previously calcined at a temperature between 500 °C and 600 °C to constant mass, and transfer into a 500 ml glass beaker.

Add 60 ml of water and, if necessary, heat gently to complete the dissolution.

Allow to cool and transfer the solution quantitatively into a ~~1000~~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 sodium.

Store the solution in a polyethylene bottle.

5.8 Sodium standard solution (0,1 g~~/l~~).

Transfer 10,00 ml of sodium standard solution ~~(5.7)~~(5.7) into a 100 ml volumetric flask, add 10 ml nitric acid solution~~(5.6),~~ (5.6), dilute to the mark with water, and mix well.

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

5.9 Sodium standard solution (0,01 g~~/l~~).

Transfer 10,00 ml of sodium standard solution ~~(5.8)~~(5.8) into a 100 ml volumetric flask, add 10 ml nitric acid solution ~~(5.6),~~(5.6), dilute to the mark with water, and mix well.

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

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 spectrometry. (ICP-OES)

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.~~6.1.3 to 6.1.4.

6.1.2 Wavelengths

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

~~In Table1, however, several~~Several suggestions are given in Table 1 together with possible interferences. These wavelengths have been carefully investigated. It is recommended to use Na 588,995 nm or Na 589,592 nm because of their high sensitivity.

Table 1 — Examples of wavelengths for sodium determination

Element	Wavelength nm	Possible interferences
Na	588,995	Sc

