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Magnesium and magnesium alloys — Determination of mercury

Magnésium et alliages de magnésium — Dosage du mercure

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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 is the lightest of all the common metals and has been prepared for industry use as metal ingots and alloys since these have the best strength-to-weight ratio of any of the commonly used structural alloys. Chemical compositions of magnesium and its alloys are widely standardized from major to trace quantities. Mercury has generally been a non-analysing element to monitor because it seems to be volatilized on heating due to its low boiling point. Thus, mercury has not been prescribed solely in standards, but has been included as impurities in standards. ISO 8287 for unalloyed magnesium specifies that the sum of hazardous elements, including mercury, in all materials should be less than 0,01 % mass fraction. ISO 16220 for magnesium alloy ingots and casting denotes the impure elements should not be less than 0,01 % separately.

However, there exists no standardized analytical methods for determination of mercury in magnesium and magnesium alloys. Moreover, a new global mercury treaty called the Minamata Convention that came into effect in 2020, which regulates and controls mercury globally, has encouraged the development of the analysis of mercury on some trading materials and products.

This document specifies the methods for determination of trace levels of mercury in magnesium and magnesium alloys.

Magnesium and magnesium alloys — Determination of mercury

1 Scope

This document specifies the methods for the determination of mercury in magnesium and magnesium alloys by inductively coupled plasma (ICP) atomic mass spectrometric analysis and by atomic absorption spectrometric analysis.

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 384, *Laboratory glass and plastics ware — Principles of design and construction of volumetric instruments*

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*

ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological 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 Classification of methods for determination

The method for the determination of tin shall be in accordance with any one of the following methods.

a) ICP mass spectrometric analysis (Method A).

This method is applicable to magnesium and magnesium alloy of 0,000 01 % (mass fraction) or over up to and including 0,01 % (mass fraction) in mercury content.

b) Atomic absorption spectrometric analysis (Method B).

This method is applicable to magnesium and magnesium alloy of 0,000 02 % (mass fraction) or over up to and including 0,001 % (mass fraction) in mercury content.

5 Sampling, storing and weighing of analytical samples

5.1 Sampling

Sampling shall be carried out as follows.

a) When the chips are sampled from a casting sample (A) or a product sample (B), select the sampling position so as to represent the quality of the sample, and penetrate the sample by boring at right angles to its surface. In the case of a sample with a thickness not penetrable from one direction, another suitable method (e.g. boring from two directions) shall be used.

b) Prior to boring for chipping, clean the drill (which is not more than 10 mm in diameter) using ethanol. Remove the adhered matters on the surface of the sampling position, and then carry out the boring, without using any sort of oils or lubricant and with just enough force to drill without oxidizing the sample chips. At this time, adjust the pressure exerted or the revolution frequency of the drill so that no excessive heat is generated. Do not attempt to cool off or stop the temperature from rising by pouring water or another liquid over the sample.

   The use of cutting tools other than a drill, such as a lathe, is permissible.

c) Collect all of the sample chips (which shall be no larger than 10 mm), and remove iron powder, etc., using a strong magnet. Then, mix the chips together thoroughly to create the analytical sample and leave them in a desiccator to cool to room temperature.

d) If the sampling from specimens such as thin sheets, pipes, etc., cannot be in accordance with the specifications given in a) to c), the sampling method shall be as agreed by the purchaser and the supplier.

5.2 Storing of analytical sample

The analytical sample shall be stored as follows.

a) Store the sample in a glass container with a lid sealed hermetically to prevent contamination.

b) If there is the possibility that substances (e.g. oil) have adhered on the surface, clean the analytical sample using a product (e.g. ethanol, acetone) and dry it before using.

5.3 Weighing of the analytical sample

The analytical sample shall be weighed as follows.

a) Mix the sample thoroughly so that any portion weighed out represents the average composition.

b) Weigh out 0,1 g (method A) or 0,5 g (method B) of the sample to a digit of 1 mg by using an analytical balance described in 6.3.

6 Apparatus

Use normal laboratory apparatus and, in particular, the following.

6.1 Volumetric glassware, of Class A in accordance with ISO 384, ISO 648 and ISO 1042, and used in accordance with ISO 4787.

6.2 Analytical balance, sensitive to 0,1 mg.

6.3 ICP mass spectrometer, to measure the ion intensities of mercury with separate masses from the inductively coupled plasma.

6.4 Atomic absorption spectrometer, to measure the light absorption of specific wavelength arising from mercury in the quartz cell.

7 ICP mass spectrometric analysis (Method A)

7.1 Summary

The aqueous solution including the chipped magnesium sample is added with solution of permanganate ion prior to dissolution. The solution is then decomposed with nitric acid, thus supressing volatilization of mercury in the sample. Precipitates of manganese(IV) oxide generated during decomposition are reduced with hydroxyl ammonium chloride. The prepared solution is sprayed into the argon plasma of the ICP mass spectrometer and the ion intensities of mercury are measured.

7.2 Reagents

During the analysis, use only reagents of recognized analytical grade and water that conforms to grade 1 or 2 of ISO 3696.

7.2.1 **Hydrochloric acid,** 1+1.

Dilute hydrochloric acid [35 % to 37 % (mass fraction)] of analytical grade twice with water.

7.2.2 **Nitric acid**, 1+1.

Dilute nitric acid [60 % to 61 % (mass fraction)] of analytical grade twice with water.

7.2.3 **Potassium permanganate solution**, Mn (VII): 50 g/l.

Weigh out 14,5 g of potassium permanganate [not less than 99,3 % (mass fraction)] and transfer it into a beaker (100 ml). Add 50 ml of water to dissolve, then transfer it into a 100 ml brown coloured volumetric flask by using water, and dilute it up to the mark with water.

7.2.4 **Hydroxyl ammonium chloride solution**, 200 g/l.

Weigh out 20 g of hydroxyl ammonium chloride [not less than 98,0 % (mass fraction)] and transfer it into a beaker (100 ml). Add 50 ml of water to dissolve, then prepare it as a 100 ml solution in a volumetric flask with water.

7.2.5 **L-Cysteine solution**, 1 g/l.

Weigh out 0,20 g of L-cysteine [not less than 98,0 % (mass fraction)] and transfer it into a beaker (100 ml). Add 50 ml of water to dissolve, then prepare it as a 200 ml solution in a volumetric flask with water.

7.2.6 **Magnesium solution**, Mg(II): 40 mg/ml.

Weigh out 33,2 g of magnesium oxide [not less than 99,99 % (mass fraction)] and transfer it into a beaker (500 ml). Add 50 ml of water, cover it with a watch glass and add 280 ml of nitric acid (1+1). After the reaction has stopped, decompose it completely by heating. After cooling it down to a normal temperature, prepare it as a 500 ml solution in a volumetric flask with water.

7.2.7 **Aluminium solution**, Al(III): 1,0 mg/ml.

Weigh out 0,20 g of aluminium [not less than 99,99 % (mass fraction)], transfer it into a beaker (300 ml) and cover it with a watch glass. Add 10 ml of water and then decompose it by adding the premixed acid solution from 10 ml of hydrochloric acid (1+1) and 30 ml of nitric acid (1+1). When the reaction has stopped, decompose it completely by heating. After cooling it down to a normal temperature, wash the