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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 13748.3-2026

Replacing GB/T 13748.3-2005

**Methods for chemical analysis of magnesium and magnesium
alloys - Part 3: Determination of lithium and silver content -
Flame atomic absorption spectrometry**

镁及镁合金化学分析方法 第3部分：锂、银含量的测定 火焰原子吸收光谱法

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part

1. Structure and Drafting Rules of Standardization Documents". Drafting. This document is Part 3 of GB/T 13748, "Chemical Analysis Methods for Magnesium and Magnesium Alloys". GB/T 13748 has been published with the following... part.

- 1.Determination of Aluminum Content;
- 2.Determination of Tin, Beryllium, Copper, Nickel and Titanium Content by Spectrophotometry;
- 3.Determination of Lithium and Silver Content by Flame Atomic Absorption Spectrometry;
- 4.Determination of Manganese and Zirconium Content by Spectrophotometry;
- 8.Determination of Rare Earth Content;
- 9.Determination of Iron and Silicon Content by Spectrophotometry;
- 13.Determination of Lead, Calcium, Potassium and Sodium Content by Flame Atomic Absorption Spectrometry;
- 15.Determination of Zinc Content;
- 18.Determination of Chlorine Content using the Silver Chloride Turbidity Method;
- 20.Determination of Elemental Content by Inductively Coupled Plasma Atomic Emission Spectrometry;
- 21.Determination of Elemental Content by Direct-Reading Atomic Emission Spectrometry;
- 22.Determination of Thorium Content;

3 Determination of Lithium and Silver Content Flame atomic absorption spectrometry

1.Scope This document describes a method for determining the lithium and silver content in magnesium and magnesium alloys using flame atomic absorption spectrometry. This document applies to the determination of lithium and silver content in magnesium and magnesium alloys. Determination range (mass fraction). Lithium content 0.0020%~ 16.00%, with a silver content of 0.010%~4.00%.

3.Terms and Definitions This document does not contain any terms or definitions that need to be defined.

4.Instruments and Equipment Flame atomic absorption spectrometer, equipped with a lithium-silver hollow cathode lamp. The instrument should meet the following conditions.

---Characteristic concentration. In a solution consistent with the matrix of the measured sample solution, the characteristic concentration of lithium is not greater than 0.018 µg/mL, and the characteristic concentration of silver is... The characteristic concentration is no greater than 0.020 µg/mL.

---Precision. The standard deviation of 10 absorbance measurements with the highest concentration standard solution should not exceed 1.0% of the average absorbance; using

the most... Ten absorbance measurements of a low-concentration standard solution (not a zero-concentration solution) should have a standard deviation not exceeding that of the highest-concentration standard solution. 0.5% of the average absorbance.

---Linearity of the working curve. The working curve is divided into 5 equal segments according to concentration. The absorbance difference of the highest segment and the absorbance difference of the lowest segment are... The ratio is not less than 0.7.

The sample was processed into fragments with a thickness of no more than 1 mm.

6 Determination of Lithium Content

6.1 Method Overview The sample was dissolved in nitric acid, and the lithium absorption was measured at a wavelength of 670.8 nm using an air-acetylene lean flame and a flame atomic absorption spectrometer. The photometric value was used to calculate the mass fraction of lithium using the working curve method.

6.2 Reagents Unless otherwise specified, only reagents confirmed to be of analytical grade and grade II water as specified in GB/T 6682 shall be used in the analysis.

6.2.1 Hydrogen peroxide ($\rho=1.10\text{g/mL}$).

6.2.2 Hydrofluoric acid ($\rho=1.14\text{g/mL}$).

6.2.3 Nitric acid (1 1). Transfer 500 mL of nitric acid ($\rho=$

1.42 g/mL, analytical grade) to 500 mL of water and mix well.

6.2.4 Sulfuric acid (1 1). Transfer 500 mL of sulfuric acid ($\rho=$

1.84 g/mL, analytical grade) to 500 mL of water and mix well.

6.2.5 Lithium Standard Stock Solution (1 mg/mL). Weigh 5.3228 g of lithium carbonate ($w_{\text{Li}_2\text{CO}_3}\geq 99.99\%$) into a 500 mL beaker, and slowly... Add 120 mL of nitric acid (1.9), heat until completely dissolved, boil for 5 minutes, and cool to room temperature. Transfer the solution to a 1000 mL volumetric flask. Dilute with water to the mark and mix well. 1 mL of this solution contains 1 mg of lithium. Alternatively, prepare using a certified standard solution.

6.2.6 Lithium Standard Solution A (10 $\mu\text{g/mL}$). Transfer

10.00 mL of lithium standard stock solution (6.2.5) to a 1000 mL volumetric flask, and add water. Dilute to the mark and mix well. 1 mL of this solution contains 10 μg of lithium.

6.2.7 Lithium Standard Solution B (50 $\mu\text{g/mL}$). Transfer

5.00 mL of lithium standard stock solution (6.2.5) to a 100 mL volumetric flask and dilute

with water. Dilute to the mark and mix well. 1 mL of this solution contains 50 µg of lithium.

6.2.8 Magnesium Solution A (20 mg/mL). Weigh

20.00 g of magnesium ($w_{\text{Mg}} \geq 99.99\%$, $w_{\text{Li}} \leq 0.0005\%$) into a 2000 mL beaker. Add 600 mL of nitric acid in portions (6.2.3), cover with a watch glass, heat until completely dissolved, cool to room temperature, and transfer to a 1000 mL container. Dilute with water to the mark in a volumetric flask and mix well.

6.2.9 Magnesium Solution B (1 mg/mL). Transfer

25.0 mL of magnesium solution A (6.2.8) to a 500 mL volumetric flask and dilute to the mark with water. Mix well.

6.3 Test Procedure

6.3.1 Sample Weigh 0.50g of the sample (Chapter 5), accurate to 0.0001g, and record it as m_0 .

6.3.2 Parallel Tests Perform the experiment twice in parallel and take the average value.

6.3.3 Blank Test A blank test was performed along with the sample.

6.3.4 Measurement

6.3.4.1 Place the sample in a 250mL beaker, add 10mL of water, and slowly add 10mL of nitric acid (6.2.3). Wait until the vigorous reaction stops. Then add 10 mL of nitric acid (6.2.3), and a few drops of hydrogen peroxide (6.2.1) can be added to dissolve it. Cover with a watch glass and heat until completely dissolved. If any... Dissolve the solids, filter and wash. Place the residue and filter paper in a platinum crucible, ashed, ignite at 550°C, and cool. Add 2 mL of sulfuric acid (6.2.4). Add 5 mL of hydrofluoric acid (6.2.2) and nitric acid (6.2.3) dropwise until the solution is clear. Heat to evaporate to near dryness, ignite at 750°C for 5 minutes, and then cool. Bring to room temperature, dissolve the residue with a small amount of nitric acid (6.2.3), filter if necessary, and combine the filtrate with the original test solution.

6.3.4.2 Transfer the test solution (including the combined solution after treating insoluble matter) into the corresponding volumetric flask according to Table 1, dilute with water to the mark, and mix well.

6.3.4.3 At a wavelength of 670.8 nm using a flame atomic absorption spectrometer, an air-acetylene lean flame was used, with water used for zeroing, and compared with the corresponding series of standard... Simultaneously measure the absorbance of lithium in the standard solution, and find the corresponding lithium mass concentration (ρ_{Li}) from the working curve.