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

GB/T 13748.4-2026

Replacing GB/T 13748.4-2013

**Methods for chemical analysis of magnesium and magnesium
alloys - Part 4: Determination of manganese and zirconium
content - Spectrophotometry**

镁及镁合金化学分析方法 第4部分：锰、锆含量的测定 分光光度法

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Foreword

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

6.1 Periodate Spectrophotometry (I)

6.1.1 Method Overview The sample was dissolved in sulfuric acid, and in the presence of fluoroboric acid and nitric acid, divalent manganese ions were oxidized to heptavalent manganese ions using potassium periodate. The oxidized manganese was then analyzed by spectrophotometry. The absorbance was measured at a wavelength of 525 nm using a thermometer. The mass of manganese was determined from the working curve, and the mass fraction of manganese was calculated.

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

6.1.2.1 Potassium periodate.

6.1.2.2 Nitric acid ($\rho=1.42\text{g/mL}$).

6.1.2.3 Sulfuric acid (1 3).

6.1.2.4 Fluoroboric acid (1 99).

6.1.2.5 Reducing agent removal water. Take 1L of water, add 10mL of sulfuric acid (6.1.2.3) and a small amount of potassium periodate (6.1.2.1), boil for about 10 minutes, and cool. Use it as a backup.

6.1.3 Test Procedure

6.1.3.1 Sample Weigh the sample according to Table 2 (Chapter 5), accurate to 0.0001g, and record it as m_0 .

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

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

6.1.3.4 Measurement
6.1.3.4.1 Place the sample in a 250mL beaker, cover with a watch glass, and slowly add sulfuric acid according to Table 1 (6.1.2.3) until the sample is completely dissolved. Then, add 20 mL of nitric acid (6.1.2.2) and 5 mL of fluoroboric acid (6.1.2.4), and dilute with water to approximately 60 mL. If the mass fraction of manganese is greater than... 0.50%, transfer the test solution to a 100 mL volumetric flask (V0), dilute to the mark with water, and mix well. Transfer

10.00 mL of the test solution (V1) to... In a 250mL beaker, add 15mL of sulfuric acid (6.1.2.3), 20mL of nitric acid (6.1.2.2), and 5mL of fluoroboric acid (6.1.2.4), and dilute with water to approximately [amount missing]. 60mL. **6.1.3.4.2** Boil the test solution for 1 to 2 minutes, let it cool slightly, add

0.5 g of potassium periodate (6.1.2.1), continue boiling for 3 minutes, and keep warm. After 10 minutes, cool to room temperature, transfer to a 100 mL volumetric flask, dilute to the mark with water (6.1.2.5) to remove the reducing agent, and mix well. 6.1.3.4.3 Transfer a portion of the test solution into a 1cm cuvette, add sodium nitrite solution (6.1.2.6) to the remaining test solution, and shake until the purple color just turns purple. After fading, transfer a portion of the solution to another 1cm cuvette as a reference, and measure it at a wavelength of 525nm using a spectrophotometer (Chapter 4). Measure its absorbance.

6.1.3.4.4 Subtract the absorbance of the blank test solution from the absorbance of the measured test solution, and find the mass of manganese (mMn) from the working curve.

6.1.3.5 Drawing the working curve 6.1.3.5.1 Transfer 0 mL,

15.00 mL of manganese standard solution respectively. (6.1.2.8) Place the mixture in a set of 250mL beakers, and add 15mL of sulfuric acid (6.1.2.3), 20mL of nitric acid (6.1.2.2), and 5mL of fluoroboric acid respectively. (6.1.2.4) Dilute with water to about 60 mL and proceed according to steps 6.1.3.4.2 to 6.1.3.4.3. 6.1.3.5.2 Using the "zero" concentration solution in the standard solution series of the working curve as a reference, with the mass of manganese as the abscissa and the corresponding absorbance as the ordinate. Use coordinates to plot the working curve. The linear correlation coefficient of the working curve should be no less than 0.999.

6.1.5 Precision

6.1.5.1 Repeatability The measured values of two independent test results obtained under repeatability conditions, within the range of the average values given in Table 3, represent the values of the two test results. The absolute difference does not exceed the repeatability limit (r), and the number of cases exceeding the repeatability limit (r) does not exceed 5%. The repeatability limit (r) is determined by linearity based on the data in Table 3. Obtained by interpolation or extrapolation.

6.1.5.2 Reproducibility The measured values of two independent test results obtained under reproducibility conditions, within the range of average values given in Table 4, represent the values of the two test results. The absolute difference does not exceed the reproducibility limit (R), and the number of cases exceeding the reproducibility limit (R) does not exceed 5%. The reproducibility limit (R) is calculated using linearity based on the data in Table 4. Obtained by interpolation or extrapolation.

6.2 Periodate spectrophotometry (II)

6.2.1 Method Overview The sample was dissolved in sulfuric acid, oxidized with nitric acid, and then, in the presence of phosphoric acid, dimethylmanganese ions were oxidized to heptavalent manganese ions with potassium periodate. The results were then analyzed by spectrophotometry. The absorbance was measured at a wavelength of 525 nm using a thermometer. The mass of manganese was determined from the working curve, and the

mass fraction of manganese was calculated.

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

6.2.2.1 Potassium periodate.

6.2.2.2 Nitric acid ($\rho=1.42\text{g/mL}$).

6.2.2.3 Phosphoric acid ($\rho=1.71\text{g/mL}$).

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

6.2.2.5 Sulfuric acid (1 3).

6.2.2.6 Sodium nitrite solution (20g/L), prepared fresh before use.

6.2.2.7 Reducing agent removal water. Take 1L of water, add 10mL of sulfuric acid (6.2.2.5) and a small amount of potassium periodate (6.2.2.1), boil for about 10 minutes, and cool. Use it as a backup.

6.2.2.8 Manganese standard stock solution (1 mg/mL). Prepared in any of the following ways, or using a certified standard solution.

---Weigh 1.0000g of electrolytic manganese [$w_{\text{Mn}}\geq 99.9\%$], pre-wash with sulfuric acid (6.2.2.5) to remove surface oxides, then wash with water and... [Wash with ethanol and dry, then cool] Place in a 400mL beaker, add 40mL of sulfuric acid (6.2.2.5), heat to dissolve, and add approximately... Boil 80 mL of water for 5 minutes, cool to room temperature, transfer to a 1000 mL volumetric flask, dilute to the mark with water, and mix well. This solution... 1 mL contains 1 mg of manganese. Weigh 2.8770 g of potassium permanganate (primary standard) into a 400 mL beaker and dissolve it in approximately 200 mL of water. Add 40 mL of... Sulfuric acid (6.2.2.5), appropriate amount of sodium sulfite or hydrogen peroxide ($\rho=1.10\text{g/mL}$), boil, cool, and transfer to a 1000mL volumetric flask. Dilute with water to the mark and mix well. 1 mL of this solution contains 1 mg of manganese.

6.2.2.9 Manganese Standard Solution (100 $\mu\text{g/mL}$). Transfer

10.00 mL of manganese standard stock solution (6.2.2.8) to a 100 mL volumetric flask, and add water. Dilute to the mark and mix well. 1 mL of this solution contains 100 μg of manganese.

6.2.3 Test Procedure

6.2.3.1 Sample Weigh the sample according to Table 5 (Chapter 5), accurate to 0.0001g, and record it as m_1 .

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

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