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# NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA



# GB/T 13748.15-2026

Replacing GB/T 13748.15-2013

## Methods for chemical analysis of magnesium and magnesium alloys - Part 15: Determination of zinc content

镁及镁合金化学分析方法 第15部分：锌含量的测定

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## Foreword

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

## 3 Transfer

15.00 mL. The test solution was placed in a 125 mL separatory funnel.

6.4.4.3 Add 20 mL of tri-n-octylamine xylene solution (6.3.7), shake for 1 min, discard the aqueous phase after separation. Add 10 mL of [amount missing] to the organic phase. Hydrochloric acid (6.3.4), shake for 10s, discard the aqueous phase after separation.

6.4.4.4 Add 20 mL of potassium nitrate-potassium iodide mixed solution (6.3.8) to the organic phase and shake for 1 min. After separation, transfer the aqueous phase to another... Liquid funnel. Add 5 mL of potassium nitrate-potassium iodide mixed solution (6.3.8) to the organic phase, shake for 10 s, and after separation, combine the aqueous phase with the previous solution. In the aqueous phase, the organic phase is discarded.

6.4.4.5 Add 5 mL of sulfosalicylic acid solution (6.3.9) to the aqueous phase, mix well, add 2 mL of ammonia solution (6.3.5), mix well, and add 1 mL of BCO. Add

1.00 mL of PAN ethanol solution (6.3.10), mix well, and let stand for 3-5 minutes. Add

1.00 mL of PAN ethanol solution (6.3.6), mix well, and let stand for 30 seconds. Add

25.00 mL of chloroform (6.3.1), shake for 2 min, allow to stand and separate into layers, and then filter the organic phase into a 1 cm absorption cell.

6.4.4.6 Using the blank test solution accompanying the sample as a reference, measure the absorbance at a wavelength of 550 nm using a spectrophotometer. From the working curve... The corresponding zinc mass can be found online.



## 5 Flame Atomic Absorption Spectrometry

5.1 Method Overview The sample was decomposed with hydrochloric acid, hydrogen peroxide, and/or hydrofluoric acid using an air-acetylene lean flame, and the analysis was performed at a flame atomic absorption spectrometer wavelength. The absorbance of zinc was measured at 213.9 nm. The mass concentration of zinc was found from the working curve, and the mass fraction of zinc was calculated.

5.2 Instruments and Equipment A flame atomic absorption spectrometer equipped with a zinc 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 zinc is not greater than 0.025 μg/mL.

---Precision. The standard deviation of 10 absorbance measurements using the highest concentration standard solution should not exceed 1.0% of the average absorbance. The standard deviation of 10 absorbance measurements using the lowest concentration standard solution (not the "zero" concentration standard solution) should not exceed that of the highest concentration. 0.5% of the average absorbance.

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

5.3 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.

5.3.1 Hydrofluoric acid (rho=1.14g/mL).

5.3.2 Hydrogen peroxide (rho=1.10g/mL).

5.3.3 Hydrochloric acid (1 1).

5.3.4 Zinc standard stock solution (1 mg/mL). Use a certified standard solution, or prepare it as follows: Weigh 1.0000g of zinc (wZn>=99.99%) into a 400mL beaker, cover with a watch glass, and add 20mL of water and 50mL of salt. Dissolve the acid (5.3.3) completely by heating at low temperature. Transfer to a 1000 mL volumetric flask, dilute to the mark with water, and mix well. One mL of the solution contains 1 mg of zinc.

---Weigh 1.2600g of zinc oxide (pre-calcined at 1000°C for 1h and cooled to room temperature in a desiccator) and place it in a container. In a 400mL beaker, cover with a watch glass, add 50mL of hydrochloric acid (5.3.3), transfer to a 1000mL volumetric flask, and dilute with water to the specified concentration. Graduate the scale and mix well. 1 mL of this solution contains 1 mg of zinc.



### 5.3.5 Zinc Standard Solution (50 μg/mL). Transfer

25.00 mL of zinc standard stock solution (5.3.4) to a 500 mL volumetric flask and dilute with water. Fill to the mark and mix well. 1 mL of this solution contains 50 μg of zinc.

## 5.4 Test Procedure

5.4.1 Sample Weigh 0.25g of sample (Chapter 4), accurate to 0.0001g, and record it as m0.

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

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

### 5.4.4 Measurement

5.4.4.1 Place the sample in a 250mL beaker, cover with a watch glass, and add 20mL of hydrochloric acid (5.3.3) and 5 drops of peroxide in portions. Hydrogen (5.3.2), heat at low temperature, add 2 drops of hydrofluoric acid (5.3.1) if necessary, continue heating until completely dissolved, boil for 5 minutes, and cool.

5.4.4.2 Based on the mass fraction of zinc in the sample, proceed as follows:

---When the zinc mass fraction is 0.0050%~0.100%, transfer the test solution to a 100mL volumetric flask, filter if necessary, and dilute with water to the specified concentration. Scale the mixture, mix it thoroughly, and record the volume as V0.

---When the zinc mass fraction is greater than 0.100%~10.00%, transfer the test solution into the corresponding volumetric flask according to Table 1, filter if necessary, and use... Dilute with water to the mark, mix well, and record the volume as V0; then transfer a certain volume (V1) of the test solution into the corresponding volumetric flask according to Table

1, and dilute with water. Dilute to the mark, mix well, and record the volume as V2.

5.4.4.3 Operate simultaneously with the corresponding standard curve using an air-acetylene lean flame at a flame atomic absorption spectrometer wavelength of 213.9 nm. Zero the instrument with water, measure the absorbance of zinc, subtract the absorbance of the blank test solution that accompanied the sample, and then find the corresponding zinc absorbance from the working curve. Mass concentration (rho).

### 5.4.5 Plotting the Working Curve

5.4.5.1 Based on the mass fraction of zinc in the sample, the standard solutions for the working curve series are prepared as follows:

---Zinc mass fraction is 0.0050%~0.010%. Transfer 0 mL,

0.25 mL,

0.50 mL,

2.00 mL of zinc standard. The solution (5.3.5) was placed in a set of 100mL volumetric flasks, diluted with water to the mark, and mixed well.

---For zinc concentrations greater than 0.010%~10.00% by mass. transfer 0 mL,

0.50 mL,

5.00 mL of zinc standard solution (5.3.5) was placed in a set of 250 mL volumetric flasks, diluted with water to the mark, and mixed well.

5.4.5.2 The series of standard solutions were analyzed on a flame atomic absorption spectrometer using an air-acetylene lean flame at a wavelength of 213.9 nm. Zero the instrument with water and measure the absorbance of a series of standard solutions. Plot the zinc mass concentration on the x-axis and subtract the absorbance of the "zero concentration" solution from the corresponding absorbance. Using luminance as the ordinate, plot the working curve.

### 5.6 Precision

5.6.1 Repeatability The measured values of two independent test results obtained under repeatability conditions, within the range of the average values given in Table 2, represent 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 calculated using linear data from Table 2. It can be obtained by interpolation or extension.

5.6.2 Reproducibility The measured values of two independent test results obtained under reproducibility conditions, within the range of the average values given in Table 3, represent the two test results. The absolute difference should not exceed the reproducibility limit (R),