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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13748.8-2026

Replacing GB/T 13748.8-2013

Methods for chemical analysis of magnesium and magnesium alloys - Part 8: Determination of rare earth content

镁及镁合金化学分析方法 第8部分：稀土含量的测定

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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 8 of GB/T 13748, "Chemical Analysis Methods for Magnesium and Magnesium Alloys". GB/T 13748 has published 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;

4 Instruments and Equipment

4.1 High-temperature furnace. The temperature should meet 950°C±10°C.

4.4 Spectrophotometer.

4.5 Porcelain crucible.

5 Samples

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

6 Oxalate Gravimetric Method

6.1 Method Overview The sample was dissolved in hydrochloric acid, zirconium was precipitated with ammonia, and rare earth elements were initially precipitated with sebacic acid in an ammonia medium. The two precipitates were then dissolved. The rare earth oxalate is reprecipitated, the oxides of rare earth elements are ignited and weighed, and the rare earth content is calculated.

6.2 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.

6.2.1 Ammonium chloride (NH_4Cl).

6.2.2 Hydrochloric acid (rho=1.19g/mL).

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

6.2.4 Ammonia (1 1).

6.2.5 Ammonia (1 4).

6.2.6 Ammonia washing solution (1 49).

6.2.7 Nitric acid-hydrogen peroxide solution. Add 30 mL of nitric acid (rho=

1.42 g/mL) and 30 mL of hydrogen peroxide (6.2.3) to 150 mL of water. Mix well. Prepare fresh before use.

6.2.8 Saturated oxalic acid solution. Weigh 150g of oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) into a beaker, add 1000mL of water, and heat to boiling for 1 minute. Then, cool and filter.

6.2.9 Oxalic acid washing solution. Transfer 70 mL of saturated oxalic acid solution (6.2.8) and dilute with water to 500 mL.

6.2.10 Sebacic acid solution (50 g/L). Weigh 50 g of sebacic acid ($C_{10}H_{18}O_4$) and dissolve it in 400 mL of ammonia water (rho =

0.90 g/mL), add... Filter 300 mL of water, dilute with water to 1000 mL, and mix well. Store in a polyethylene bottle.

6.2.11 Bromophenol blue solution (4 g/L). Weigh

0.4 g of bromophenol blue into a mortar, add

8.25 mL of sodium hydroxide solution (5 g/L), and grind. Continue until completely dissolved, then dilute with water to 100 mL and mix well.

6.3 Test Procedure

6.3.1 Sample Weigh the sample according to Table 1, accurate to 0.0001g, and record it as m0.

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

6.3.3 Measurement

6.3.3.1 Place the sample in a 400mL beaker, add 75mL of water, cover with a watch glass, and add hydrochloric acid in portions according to the total integral in Table 1 (6.2.2). After the reaction stops, add an appropriate amount of hydrogen peroxide (6.2.3), heat to boiling for 5 minutes. If there is still residue, filter with medium-speed filter paper and hot water. Wash the beaker and precipitate 4-5 times, discarding the precipitate. Dilute or evaporate the filtrate to approximately 100 mL and cool. If the magnesium alloy contains silver... Before filtering, a small amount of fiber pulp is first spread on the filter paper.

6.3.3.2 Add 3 drops of bromophenol blue solution (6.2.11) to the test solution, adjust the solution to just turn blue-purple with ammonia water (6.2.5), and heat on an electric furnace. Bring to a boil, remove from heat, let stand for 5 minutes, stirring occasionally, filter with high-speed filter paper, and wash the precipitate thoroughly with boiling water to

ensure the filtrate volume is no greater than [missing value]. 250 mL of this solution is filtrate A; retain this solution. Dissolve the residue on the filter paper in portions using 20 mL of boiled nitric acid-hydrogen peroxide solution (6.2.7). The precipitate was placed back into the original beaker. The filter paper was washed 5-6 times with hot water. The solution volume was evaporated by heating to approximately 25 mL. This solution is filtrate B. This filtrate should be retained. Solution.

6.3.3.3 Add 10g of ammonium chloride (6.2.1) to filtrate A, first with ammonia water (6.2.4), then with ammonia water (6.2.5), and adjust the solution on the pH meter. The pH of the solution is 8.5, then add an excess of 10 mL of ammonia water (6.2.5). Heat the solution to 90°C, remove it from the heat, and add 20 mL of decane while stirring. The diacid solution (6.2.10) was left to stand for 15 minutes, stirring occasionally, and then filtered through medium-speed quantitative filter paper. The solution was then thoroughly washed with ammonia solution (6.2.6). If zinc is present, wash the precipitate again with 20 mL of ammonia water (6.2.4).

6.3.3.4 The porcelain crucible is ignited at 940°C~960°C and brought to constant weight. The weight is recorded as m

1.Filter paper containing the precipitate is placed into the constant-weight porcelain crucible. After complete ashing at below 500°C (do not allow the filter paper to ignite), calcine in a high-temperature furnace at 750°C~800°C for 30 minutes, then remove and cool.

6.3.3.5 Wash the oxide in the porcelain crucible with water into a beaker containing filtrate B, heat the solution, and add a few drops of hydrogen peroxide (6.2.3) to dilute. Dissolve the oxalic acid oxide, remove the beaker, rinse the beaker walls with water, and dilute to approximately 125 mL. While stirring, slowly add 25 mL of saturated oxalic acid solution. (6.2.8) Place the beaker in a boiling water bath for 30 minutes, then remove it and leave it at room temperature for more than 12 hours (or let it stand overnight).

6.3.3.6 Filter the rare earth oxalate precipitate using slow-speed quantitative filter paper, wash the precipitate thoroughly with oxalic acid washing solution (6.2.9), and then place the precipitate and filter paper in a container. Place the mixture into a porcelain crucible and ashing completely below 500°C (do not allow the filter paper to ignite). Then transfer it to a high-temperature furnace and calcine at 940°C~960°C. 60 min. Remove, place in a desiccator to cool, and weigh. Repeat the ignition process until the difference between two weighings is no greater than 0.0002 g, then weigh and record the result. The value is m2.

6.5 Precision

6.5.1 Repeatability The measured values of two independent test results obtained under repeatability conditions, within the range of the average values given in Table 2, represent the two test results. The absolute difference does not exceed the repeatability limit (r), and