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GB/T 13748.22-2026

Replacing GB/T 13748.22-2013

Methods for chemical analysis of magnesium and magnesium alloys - Part 22: Determination of thorium content

镁及镁合金化学分析方法 第22部分：钍含量的测定

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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 22 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;

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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;

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4 Instruments and Equipment

4.1 High-temperature furnace. controllable temperature 950°C±20°C.

4.2 Desiccant. Color-changing silica gel is used as the desiccant.

4.3 pH meter. with glass electrode.

5 Samples

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

6 Gravimetric method

6.1 Method Overview The sample was dissolved in hydrochloric acid, and thorium was initially separated in the form of benzoate. The precipitate was dissolved, and thorium was redeprecipitated in the form of oxalate. The mixture was then ignited and weighed. The mass of thorium oxide is used to calculate the mass fraction of thorium in the sample.

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 Hydroxylamine hydrochloride (NH2OH·HCl).

6.2.2 Ammonium chloride (NH4Cl).

6.2.3 Hydrochloric acid (1 1). Transfer 500 mL of hydrochloric acid (rho=

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

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6.2.4 Hydrochloric acid (1 3). Transfer 500 mL of hydrochloric acid (rho=

1.19 g/mL, analytical grade) and dissolve it in 1500 mL of water, then mix well.

6.2.5 Ammonia (1 3).

6.2.6 Benzoic acid solution (20 g/L). Weigh 10 g of benzoic acid (C6H5COOH) into a 500 mL beaker, add water and heat gently until completely dissolved. (Filter while hot if necessary), dilute to 500 mL, and mix well. Dissolve gently by heating before use.

6.2.7 Benzoic acid cleaning solution (

1.25 g of benzoic acid (C6H5COOH) into a 500 mL beaker, add water and heat gently until dissolved. Cool thoroughly, dilute to 500 mL, and mix well. Filter if necessary.

6.2.8 Saturated oxalic acid solution.

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

6.2.10 Bromophenol Blue Solution (4 g/L). Weigh

0.4 g of bromophenol blue (C19H10Br4O5S) into a mortar, add

8.25 mL of sodium hydroxide solution. Grind the liquid (5g/L) until completely dissolved, dilute with water to 100mL, 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 Blank Test A blank test was performed along with the sample.

6.3.4 Measurement

6.3.4.1 Place the sample in a 400mL beaker, cover with a watch glass, add 50mL of water, and add hydrochloric acid in portions according to Table 1 (6.2.3). React vigorously. After stopping, boil for 5 minutes. If there are insoluble substances, filter with medium-speed filter paper (when analyzing magnesium alloys containing silver, place a small amount of paper on the filter paper before filtering). Wash the beaker and insoluble matter with hot water, add the washing liquid to the test solution, dilute or evaporate to make the solution volume about 100 mL, and cool.

6.3.4.2 Add 3 drops of bromophenol blue solution (6.2.10) to the test solution [if rare earth elements are present, first add 1g of hydroxylamine hydrochloride (6.2.1)], and then use

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ammonia water. Adjust the solution to a purple color using (6.2.5) or hydrochloric acid (6.2.4). Add 10g of ammonium chloride (6.2.2), heat to boiling, and while stirring, add 100mL of [amount missing] mL... A benzoic acid solution at a temperature not lower than 80°C (6.2.6) is heated for another 10 minutes. After standing for several minutes, it is filtered while still hot using medium-speed filter paper, and then washed with benzoic acid. Wash the precipitate and filter paper three times each with liquid (6.2.7), and discard the filtrate.

6.3.4.3 Transfer the precipitate back to the original beaker in portions of 50 mL boiling water, and wash it in portions of 10 mL hydrochloric acid (6.2.4) and 50 mL boiling water. Filter paper, combine the filtrates, and boil. Remove and cool, wash the beaker walls, dilute the solution to 125 mL, and slowly add 25 mL while stirring and boil. The saturated oxalic acid solution (6.2.8) was kept at around 80°C for 2 hours and then allowed to stand at room temperature for 12 hours (or overnight).

6.3.4.4 Filter with slow filter paper and wash the precipitate 10 times with oxalic acid washing solution (6.2.9).

6.3.4.5 Place the filter paper and precipitate in a platinum crucible (m2) that has been preheated to constant weight at 950°C, and ashing the filter paper completely in a high-temperature furnace at 500°C. The sample was heated at 950°C for 2 hours, then removed and cooled in a desiccator for 30 minutes before weighing. The heating process was repeated until the difference between two weighings was no greater than [value missing]. 0.0002g, denoted as m1.

6.4 Experimental Data Processing Thorium content is expressed as thorium mass fraction (wTh) and calculated according to formula (1).

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 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 2. Obtained by interpolation or extrapolation.

6.5.2 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), and the number of cases exceeding the reproducibility limit (R) should not exceed 5%. The reproducibility limit (R) is calculated using linear regression based on the data in Table 3. It can be obtained by interpolation or extension.

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