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GB/T 13748.9-2026

Replacing GB/T 13748.9-2013

Methods for chemical analysis of magnesium and magnesium alloys - Part 9: Determination of iron and silicon content - Spectrophotometry

镁及镁合金化学分析方法 第9部分：铁、硅含量的测定 分光光度法

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Table of Contents

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 9 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;

6 Determination of Iron Content - o-Phenanthroline Spectrophotometric Method

6.1 Method Overview The sample was dissolved in hydrochloric acid, and ferric iron was reduced with hydroxylamine hydrochloride. In an acetate buffer medium with a pH of 3.5-4.5, ferrous ions reacted with ortho-ferric ions. The diazoxide-phenanthroline complex formed an orange-red complex, and its absorbance was measured at a wavelength of 510 nm using a spectrophotometer. The iron content was calculated based on the working curve. quality.

6.2 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.

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

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

6.2.3 Hydrochloric acid (1 1).

6.2.4 Hydroxylamine hydrochloride solution (10 g/L).

6.2.5 Acetic acid-sodium acetate buffer solution. Weigh 272g of sodium acetate ($CH_3COONa \cdot 3H_2O$) and dissolve it in 500mL of water, add... Dilute 240 mL of acetic acid (rho =

1.05 g/mL) with water to 1000 mL and mix well. 6.2.6 1,10-Phenanthroline solution (10 g/L). Weigh

5.0 g of 1,10-1,10-Phenanthroline and dissolve it in 100 mL of anhydrous ethanol, then dilute with water to a final concentration of 10 g/L. Mix 500mL thoroughly.

6.2.7 Iron Standard Stock Solution (250 μg/mL). Weigh 0.2500 g of iron (wFe >= 99.99%)

into a 100 mL beaker, add 30 mL of... Hydrochloric acid (6.2.3), cover with a watch glass, heat slowly until completely dissolved, cool, transfer to a 1000mL volumetric flask, dilute with water to the mark, mix. Uniform. This solution contains 250 μg of iron per mL. Alternatively, a certified standard solution may be used.

6.2.8 Iron Standard Solution A (25 μg/mL). Transfer

50.00 mL of the iron standard stock solution (6.2.7) to a 500 mL volumetric flask and dilute with water. Dilute to the mark and mix well. 1 mL of this solution contains 25 μg of iron.

6.2.9 Iron Standard Solution B (5 μg/mL). Transfer

50.00 mL of iron standard solution A (6.2.8) to a 250 mL volumetric flask and dilute with water. Fill to the mark and mix well. This solution contains 5 μg of iron per mL. Prepare fresh before use.

6.3 Test Procedure

6.3.1 Sample Weigh the sample according to Table 1 (Chapter 5), 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 250mL beaker, add 5mL of water, and then add 20mL of hydrochloric acid in portions (6.2.3). Cover the beaker with the top layer of water. After a vigorous reaction, add 2 drops of hydrogen peroxide (6.2.1), heat until completely dissolved, and boil to decompose excess hydrogen peroxide (6.2.1). Continue heating and evaporating until a paste is formed (the volume of the blank test solution accompanying the sample is approximately

0.5 mL). Remove from heat and cool to room temperature. Transfer the test solution to... In a 100mL volumetric flask (filter if turbid). For samples containing zirconium or silicon, if there is insoluble residue, add 1 drop of hydrogen. Fluoroic acid (6.2.2) is used as a solubilizer.

6.3.4.2 Dilute with water to approximately 50 mL and mix well. Add 4 mL of hydroxylamine hydrochloride solution (6.2.4) and 15 mL of acetate-sodium acetate. The buffer solution (6.2.5) and 10 mL of o-phenanthroline solution (6.2.6) were diluted with water to the mark, mixed well, and left to stand for

0.5 h.

6.3.4.3 Transfer a portion of the test solution into the cuvette recommended in Table 1,

using the blank test solution accompanying the sample as a reference, and measure the solution at the wavelength of the spectrophotometer. The absorbance was measured at 510 nm, and the corresponding mass of iron (mFe) was obtained from the working curve.

6.4 Drawing the working curve

6.4.1 Based on the mass fraction of iron in the sample, a series of standard solutions were prepared in the following manner.

---When the mass fraction of iron is 0.0010%~0.0050%. transfer 0 mL,

0.50 mL,

2.00 mL, respectively.

6.00 mL of iron standard solution B (6.2.9) were placed in a set of 100 mL volumetric flasks and proceeded according to 6.3.4.2.

---When the iron mass fraction is greater than 0.0050%~0.010%. transfer 0 mL,

0.50 mL,

2.00 mL, respectively.

6.00 mL of iron standard solution A (6.2.8) were placed in a set of 100 mL volumetric flasks and proceeded according to 6.3.4.2.

---When the iron mass fraction is greater than 0.010%~0.100%. transfer 0 mL,

0.50 mL,

3.00 mL, respectively.

9.00 mL of iron standard solution A (6.2.8) were placed in a set of 100 mL volumetric flasks and proceeded according to 6.3.4.2.

6.4.2 Transfer a portion of the test solution into the cuvette recommended in Table 1, using the "zero" concentration solution in the working curve series of standard solutions as a reference, and dilute it at a

6.6 Precision

6.6.1 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.

6.6.2 Reproducibility The measured values of two independent test results obtained under