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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 16477.2-2026

Replacing GB/T 16477.2-2010

Chemical analysis methods for rare earth ferrosilicon and rare earth magnesium ferrosilicon alloys - Part 2: Determination of calcium, magnesium, manganese, aluminium, barium, antimony, bismuth, strontium, phosphorus and titanium contents

稀土硅铁合金及镁硅铁合金化学分析方法 第2部分：钙、镁、锰、铝、钡、锑、铋、锶、磷和钛含量的测定

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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 2 of GB/T 16477 "Chemical Analysis Methods for Rare Earth Ferrosilicon Alloys and Magnesium Ferrosilicon Alloys". GB/T 16477 has been... The following sections were published.

1.Determination of total rare earth content and content of fifteen rare earth elements;

2.Determination of the content of calcium, magnesium, manganese, aluminum, barium, antimony, bismuth, strontium, phosphorus and titanium;

3.Determination of Magnesium Oxide Content by Inductively Coupled Plasma Atomic

Emission Spectrometry;

4.Determination of Silicon Content;

5.Determination of Titanium Content by Inductively Coupled Plasma Emission Spectrometry This document replaces GB/T 16477.2-2010 "Chemical Analysis Methods for Rare Earth Ferrosilicon Alloys and Magnesium Ferrosilicon Alloys - Part

2.Calcium, Magnesium, "Determination of Manganese Content by Inductively Coupled Plasma Atomic Emission Spectrometry" has been revised compared to GB/T 16477.2-2010, except for structural adjustments and editorial changes. Aside from the changes, the main technical changes are as follows:

a) The scope of application and measurement range of the method have been changed (see Chapter 1, Chapter 1 of the.2010 edition);

b) Regulations regarding reagents and water have been added (see 4.2, 5.2);

c) Added preparation guidelines for standard stock solutions and mixed standard solutions of aluminum, barium, antimony, bismuth, strontium, phosphorus, and titanium (see 4.2);

d) The instrumentation requirements for Method 1 have been changed (see 4.3, Chapter 4 of the.2010 edition);

e) The recommended analytical spectral wavelength for Method 1 has been changed (see 4.3,

6.6.1 in the.2010 edition);

4 Inductively Coupled Plasma Emission Spectrometry (Method 1)

4.1 Principle The sample was decomposed by nitric acid and hydrofluoric acid, and the fluorine was removed by the fuming effect of perchloric acid. The fluoride was then analyzed by inductively coupled plasma atomic emission spectrometry in a hydrochloric acid medium. The contents of calcium, magnesium, manganese, aluminum, barium, antimony, bismuth, strontium, phosphorus and titanium were determined at a fixed wavelength.

4.2 Reagents or Materials Unless otherwise specified, only reagents confirmed to be of analytical grade or higher and Class II water conforming to GB/T 6682 shall be used in the analysis. Liquids All reagents are stored in polyethylene bottles, and certified standard solutions are preferred.

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

4.2.2 Nitric acid (rho=1.42g/mL).

4.2.3 Hydrofluoric acid (rho=1.15g/mL).

4.2.4 Perchloric acid (rho=1.66g/mL).

4.2.5 Hydrochloric acid (1 1).

4.2.6 Nitric acid (1 1).

4.2.7 Sulfuric acid (1 1).

4.2.8 Sulfuric acid (1 9).

4.2.9 Calcium Standard Stock Solution. Weigh 0.1399 g of calcium oxide that has been ignited at 850°C for

0.5 h and cooled to room temperature in a desiccator. (w>=99.99%), placed in a 150mL beaker, moistened with a small amount of water, dissolved in 10mL hydrochloric acid (4.2.5), cooled to room temperature, and transferred to... Dilute to the mark with water in a 100mL volumetric flask and mix well. 1mL of this solution contains 1mg of calcium.

4.2.10 Magnesium Standard Stock Solution. Weigh 0.1658 g of magnesium oxide that has been ignited at 850°C for

0.5 h and cooled to room temperature in a desiccator. (w>=99.99%), placed in a 150mL beaker, moistened with a small amount of water, dissolved in 10mL hydrochloric acid (4.2.5), cooled to room temperature, and transferred to... Dilute to the mark with water in a 100mL volumetric flask and mix well. 1mL of this solution contains 1mg of magnesium.

4.2.19 Mixed Standard Solution. Transfer

10.00 mL of calcium standard stock solution (4.2.9) and

10.00 mL of manganese standard stock solution (4.2.11).

10.00 mL aluminum standard stock solution (4.2.12),

10.00 mL barium standard stock solution (4.2.13),

10.00 mL antimony standard stock solution (4.2.14),

10.00 mL of bismuth standard stock solution (4.2.15), and

10.00 mL of strontium standard stock solution (4.2.16) are placed in a 100 mL volumetric flask. Add 5 mL of hydrochloric acid (4.2.1) and dilute to the mark with water. 1 mL of this solution contains 100 μg calcium, 100 μg manganese, 100 μg aluminum, and 100 μg... Barium, 100 μg antimony, 100 μg bismuth, 100 μg strontium.

4.2.20 Magnesium Standard Solution. Transfer

10.00 mL of magnesium standard stock solution (4.2.10) to a 100 mL volumetric flask, add 5 mL of hydrochloric acid. (4.2.1) Dilute with water to the mark. 1 mL of this solution contains 100 μg of magnesium.

4.2.21 Phosphorus Standard Solution. Transfer

10.00 mL of the phosphorus standard stock solution (4.2.17) to a 100 mL volumetric flask and dilute to the mark with water. One mL of the solution contains 100 μg of phosphorus.

4.2.22 Titanium Standard Solution. Transfer

10.00 mL of titanium standard stock solution (4.2.18) to a 100 mL volumetric flask, add 2 mL of sulfuric acid. (4.2.8) Dilute with water to the mark. 1 mL of this solution contains 100 μg of titanium.

4.3 Instruments and Equipment The inductively coupled plasma atomic emission spectrometer achieves the following specifications under optimal instrument operating conditions.

---Complies with JJG768;

---The recommended analytical spectral wavelengths for element determination are shown in Table 2.

4.4 Samples The alloy sample was made into a powder and passed through a 0.125mm sieve.

4.5 Test Procedure

4.5.1 Sample Weigh 0.10g of sample (4.4), accurate to 0.0001g.

4.5.2 Parallel Tests Perform two parallel experiments.

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

4.5.4 Preparation of analytical solutions

4.5.4.1 Place the sample (4.5.1) in a.200 mL dry polytetrafluoroethylene beaker, add 5 mL of nitric acid (4.2.2), and slowly add 2 mL of nitric acid dropwise. After the hydrofluoric acid (4.2.3) reaction stops violently, heat at a low temperature until the sample is completely decomposed. Add 3 mL of perchloric acid (4.2.4) and heat at a low temperature until fumes are produced. Continue heating until the test solution is clear (approximately 3-4 mL remaining), remove from heat, allow to cool slightly, add 5 mL of hydrochloric acid (4.2.1), heat to dissolve the salts, and cool to room temperature. Transfer the solution to a 100mL volumetric flask, dilute with water to the mark, and mix well.

4.5.4.2 According to the content range of the element to be tested, accurately aliquot the test solution (4.5.4.1) into a 50mL volumetric flask according to Table 3, and add 10mL of hydrochloric acid. (4.2.5) Dilute with water to the mark and mix well.