ICS 77.120.01
CCS H 30

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 6609.13-2026

Replacing GB/T 6609.13-2004

Chemical analysis and physical property determination methods for alumina - Part 13: Determination of calcium oxide content - Flame atomic absorption spectrometry

氧化铝化学分析方法和物理性能测定方法 第13部分：氧化钙含量的测定 火焰原子吸收光谱法

Issued on: May 25, 2026 **Implemented on: December 1, 2026**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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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 13 of GB/T 6609, "Chemical Analysis Methods and Physical Property Determination Methods for Alumina". GB/T 6609 has been... The following sections were published.

1.Determination of Trace Element Content by Inductively Coupled Plasma Atomic Emission Spectrometry;

2.Determination of mass loss at 300°C and 1000°C;

---Determination of silica content by molybdenum blue spectrophotometry;

---Determination of ferric oxide content by o-phenanthroline spectrophotometry;

5.Determination of sodium oxide and potassium oxide content;

7.Determination of the content of titanium dioxide, chromium trioxide, copper oxide, fluorine, chlorine, boron trioxide, phosphorus pentoxide, and sulfate. Fixed-point spectrophotometry;

---Determination of vanadium pentoxide content by benzoylphenylhydroxylamine extraction spectrophotometry;

11.Determination of Manganese Monoxide and Magnesium Oxide Content by Flame

Atomic Absorption Spectrometry;

12.Determination of Zinc Oxide Content by Flame Atomic Absorption Spectrometry;

13.Determination of Calcium Oxide Content by Flame Atomic Absorption Spectrometry;

19.Determination of Lithium Oxide Content by Flame Atomic Absorption Spectrometry;

---Determination of gallium trioxide content by butylrhodamine B spectrophotometry;

5.4 Lanthanum chloride solution (50 g/L). Weigh

29.32 g of lanthanum oxide (wLa2O3>=99.999%) into a 250 mL beaker, and add hydrochloric acid in portions. (5.1) Dissolve lanthanum oxide, transfer it to a 500 mL volumetric flask, dilute with water to the mark, and mix well.

5.5 Aluminum matrix solution.

a) Aluminum matrix solution I. Weigh 5.295g of aluminum shavings [wAl>=99.99%], soak them in a small amount of dilute nitric acid (1.3) beforehand, and wash them with water to remove the dilute nitric acid. Nitric acid (1 3), rinsed twice with anhydrous ethanol and dried, was placed in a 500 mL beaker and 300 mL of hydrochloric acid (6.1) was added. The mixture was allowed to settle. After the vigorous reaction has stopped, place the beaker on a hot plate and slowly heat until completely dissolved, then allow it to cool naturally to room temperature. Transfer the solution to... Dilute to the mark with water in a 500mL volumetric flask and mix well. 1mL of this solution contains 20mg of aluminum oxide.

b) Aluminum matrix solution II. Weigh 5.295g of aluminum shavings (wAl>=99.99%), soak them in a small amount of dilute nitric acid (1.3) beforehand, and wash them with water to remove the nitric acid. The acid (washed twice with anhydrous ethanol and dried) was placed in a 500 mL beaker, and 330 mL of sulfuric acid (5.2) was added. The mixture was allowed to react vigorously until the reaction stopped. After the solution has dissolved completely, place the beaker on a hot plate and heat slowly until completely dissolved, then allow it to cool naturally to room temperature. Transfer the solution to a 500mL volumetric immersion tank. Dilute with water to the mark in the bottle and mix well. 1 mL of this solution contains 20 mg of aluminum oxide.

5.6 Calcium oxide standard stock solution. Weigh 1.7847 g of reference calcium carbonate [pre-dried in an oven (6.5) at 110°C±5°C for 2 h, and...] [Cooled to room temperature in a desiccator (6.4)] Placed in a 500mL beaker, 10mL of water and 20mL of hydrochloric acid (5.1) were added. After complete dissolution... Transfer to a 1000mL volumetric flask, dilute to the mark with water, and mix well. Transfer to a polyethylene bottle for storage. 1mL of this solution contains 1mg of oxygen. Calcium carbonate.

5.7 Calcium oxide standard solution. Transfer

25.00 mL of calcium oxide standard stock solution (5.6) to a 500 mL volumetric flask and

dilute with water to the specified concentration. Graduated the solution and mixed well. Transfer to a polyethylene bottle for storage. 1 mL of this solution contains 50 μg of calcium oxide.

6 Instruments and Equipment

6.1 Flame atomic absorption spectrometer, equipped with a calcium hollow cathode lamp. Under optimal instrument operating conditions, flame atomic absorption spectrometers that can achieve the following specifications... Absorption spectrometers can all be used.

---Characteristic concentration. In a solution consistent with the matrix of the analytical solution, the characteristic concentration of calcium oxide is no greater than 0.5 μg/mL;

---Precision. The standard deviation of 10 absorbance measurements using the highest concentration standard solution should not exceed 1.5% of the average absorbance. The absorbance of the lowest concentration standard solution (not the "zero" concentration standard solution) measured 10 times should have a standard deviation not exceeding that of the highest concentration standard solution. 0.5% of the average absorbance of the standard solution;

---Linearity of the working curve. The working curve is divided into five equal segments according to concentration. The absorbance difference of the highest segment and the absorbance difference of the lowest segment are linear. The ratio is not less than 0.7.

6.2 Microwave digestion apparatus.

6.3 PTFE sealed sample container, schematic diagram shown in Figure 1.

6.4 The dryer uses activated alumina as a desiccant and should be activated at 300°C before each use.

6.5 Oven, temperature can be controlled at 300°C±10°C.

7 Samples

Sampling shall be carried out in accordance with the provisions of Chapter 6 of GB/T 6609.22-2026, and sample preparation shall be carried out in accordance with Section

7.4 of GB/T 6609.22-2026. The regulations shall be followed.

8 Test Procedure

8.1 Samples Weigh 0.25g of the sample (Chapter 7), accurate to 0.0001g.

8.2 Parallel Tests Perform two parallel experiments and take the average value.

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

8.4.1 Sample Dissolution Method I

8.4.1.1 Place the sample in the reaction vessel of the polytetrafluoroethylene sample dissolving apparatus (6.1), add

8.0 mL of hydrochloric acid (5.1), cover the reaction vessel, and return it to its original position. In the polytetrafluoroethylene sealed sample dissolver (6.3), close the sample dissolver lid.

8.4.1.2 Place the sample dissolving apparatus into the steel sleeve, tighten the sleeve cap, and place it in an oven (6.5). Heat to 240°C±3°C and maintain the temperature for 5 hours. Remove and dissolve from the oven. Then cool to room temperature.

8.4.1.3 Remove the reaction vessel, transfer the solution to a 50 mL volumetric flask, rinse the reaction vessel with water, add the washings to the volumetric flask, and add

5.0 mL of [unspecified solution]. Strontium chloride solution (5.3), diluted with water to the mark, and mixed well.

8.4.2.1 Place the sample in the digestion vessel of the microwave digester (6.2), add

10.0 mL of sulfuric acid (5.2), close the lid of the digestion vessel, and return it to the microwave. Digester (6.2). Dissolve the food according to the pre-set digestion program.

8.4.2.2 After the procedure is complete, remove the digestion vessel, transfer the solution to a 50mL volumetric flask, rinse the reaction vessel with water, and add the washings to the volumetric flask. Add

5.0 mL of lanthanum chloride solution (5.4), dilute with water to the mark, and mix well.

8.5 Measurement According to the instrument's operating conditions, on a flame atomic absorption spectrometer (6.1), using an air-acetylene flame, at a wavelength of 422.7 nm, water was used for adjustment. Zero, determine the absorbance of the analytical solution (8.4) and the blank solution (8.3) prepared along with the sample, and find the corresponding oxidation from the working curve. Calcium mass concentration.

8.6 Drawing the Working Curve

8.6.1 Depending on the sample dissolution method, the following different series of standard solution preparation methods shall be selected.

---When using dissolution method I, transfer 0 mL,

0.25 mL,

0.50 mL,