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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 6609.11-2026

Replacing GB/T 6609.11-2004

Chemical analysis and physical property determination methods for alumina - Part 11: Determination of manganese oxide and magnesium oxide - Flame atomic absorption spectrometry

氧化铝化学分析方法和物理性能测定方法 第11部分：一氧化锰和氧化镁含量的测定 火焰原子吸收光谱法

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5 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 GB/T 6609.22-2026. The provisions of

7.4 shall be followed.

6 Determination of manganese monoxide content

6.1 Method Overview After the sample was dissolved in hydrochloric acid in a PTFE-sealed dissolving container at a constant temperature, the pH of the solution was adjusted to

[value missing] under the masking of potassium sodium tartrate. 11.8, the 2-methyl-8-hydroxyquinoline manganese complex was extracted with chloroform to separate manganese from interfering elements, and the organic phase was evaporated under low-temperature heating. After drying, the residue was dissolved in dilute hydrochloric acid, and its absorbance was measured at 279.5 nm using an air-acetylene flame atomic absorption spectrometer. The temperature was measured to determine the content of manganese monoxide.

6.2 Reagents Warning

---The chloroform used in this document can cause harm to the human body if inhaled or absorbed through the skin. It will react with oxygen in the air when exposed to light. The process involves the release of toxic gases, so protective equipment must be worn during the experiment, and the experiment must be conducted under well-ventilated conditions. Unless otherwise specified, only reagents confirmed to be of superior purity and Class II water conforming to GB/T 6682 shall be used in the analysis.

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

6.2.2 Chloroform, store protected from light.

6.2.3 Hydrochloric acid (1 120).

6.2.4 Sodium hydroxide solution (500g/L). Weigh 500g of sodium hydroxide and place it in a 1000mL plastic beaker. Add 400mL of water and dissolve. Completely transfer the solution to a 1L plastic volumetric flask, dilute with water to the mark, and mix well.

6.2.5 Potassium sodium tartrate solution (200 g/L). Weigh 100 g of potassium sodium tartrate into a 500 mL beaker, add 300 mL of water, and dissolve. Completely transfer the solution to a 500 mL volumetric flask, dilute with water to the mark, and mix well. 6.2.6 2-Methyl-8-hydroxyquinoline solution (20 g/L). Weigh

2.0 g of 2-methyl-8-hydroxyquinoline and dissolve it in 5 mL of glacial acetic acid in a high-boron solution. Dilute with water in a silica glass beaker, transfer to a 100mL volumetric flask, dilute to the mark with water, and mix well.

6.2.7 Manganese monoxide standard stock solution. Weigh 0.6196 g of metallic manganese (wMn>=99.9%) into a 150 mL beaker, add 12 mL of... Hydrochloric acid (1.1), cover with a watch glass, place on a hot plate and heat at low temperature until all the metallic manganese is dissolved, cool to room temperature, and transfer the solution to 1000 mL Dilute to the mark with water in a volumetric flask and mix well. 1 mL of this solution contains

0.8 mg of manganese monoxide.

6.2.8 Manganese monoxide standard solution. Transfer

10.00 mL of manganese monoxide standard stock solution (6.2.7) to a 1000 mL volumetric flask, and use... Dilute with water to the mark and mix well. 1 mL of this solution contains

0.008 mg of manganese monoxide.

6.3 Analysis Steps

6.3.1 Sample Weigh 1.50g of the sample (Chapter 5), accurate to 0.0001g.

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

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

6.3.4 Preparation of analytical solutions

6.3.4.1 Place the sample (6.3.1) into the reaction vessel of the polytetrafluoroethylene sealed sample dissolving apparatus (4.2), add 20 mL of hydrochloric acid (6.2.1), and seal the apparatus. Place the cup lid into the PTFE sealed sample dissolving container (4.2), close the sample dissolving container lid, place it in the steel sleeve, tighten the steel sleeve lid, and then place it in the oven (4.5). In the middle, the temperature is raised to 240°C±3°C, kept at this temperature for 6 hours, and then naturally cooled to room temperature.

6.3.4.2 Remove the polytetrafluoroethylene reaction vessel, transfer the solution to a 250mL beaker, wash the reaction vessel with water, and add the washing water to the beaker. Cover the beaker. Place a good watch glass on a hot plate and evaporate at a low temperature until the salts just begin to precipitate.

6.3.4.3 Remove the beaker, add 50 mL of water to dissolve all the salts, then add 25 mL of potassium sodium tartrate solution (6.2.5), stirring constantly. Slowly add 10 mL of sodium hydroxide solution (6.2.4) until the precipitated aluminum hydroxide is completely dissolved. Avoid using excessive sodium hydroxide. Add 5 mL of 2-methyl-8-hydroxyquinoline solution (6.2.6), stir until completely dissolved, allow to cool naturally to room temperature, and then dissolve using sodium hydroxide solution. (6.2.4) and hydrochloric acid (6.2.1), and the pH of the solution was adjusted to

11.8 by pH meter (4.4).

6.3.4.4 Transfer the solution to a 250 mL separatory funnel, extract with 20 mL of chloroform (6.2.2), shake for 1 min, and separate the organic phase. Continue extraction with 10 mL of chloroform (6.2.2) until the organic phase no longer turns green (usually 4 to 5 extractions are required). Collect the organic phase. Collect the contents in a beaker containing 1 mL of hydrochloric acid (6.2.1), cover with a watch glass, and evaporate to dryness on a hot plate at low temperature.

6.3.4.5 Remove the beaker, dissolve the residue with a small amount of hydrochloric acid (6.2.3), and rinse with hydrochloric acid (6.2.3) into a 25 mL volumetric flask. Allow to cool naturally until... At room temperature, dilute to the mark with hydrochloric acid (6.2.3) and mix well.

6.3.5 Measurement Under optimal operating conditions, using an air-acetylene flame on a flame atomic absorption spectrometer (4.1), at a wavelength of 279.5 nm, Zero the instrument with water, and measure the absorbance of a series of standard solutions and test solutions (6.3.4.5) and accompanying blank samples (6.3.3). Find the corresponding values from the working curve. The corresponding manganese monoxide mass concentration.

6.3.6 Drawing the working curve

6.3.6.1 Transfer 0 mL,

16.00 mL of manganese monoxide standard solution. (6.2.8) Place them in a set of 25mL volumetric flasks, dilute to the mark with hydrochloric acid (6.2.3), and mix well.

6.5 Precision

6.5.1 Repeatability (r) The measured values of the independent test results obtained under repeatability conditions, within the range of the average values given in Table 1, represent the absolute differences between the two test results. For differences not exceeding the repeatability limit (r), the number of cases exceeding the repeatability limit (r) should not exceed 5%. The repeatability limit (r) is determined by linear interpolation based on the data in Table 1. It can be obtained by the method of extension or extrapolation.

7 Determination of magnesium oxide content

7.1 Method Overview The sample is dissolved in a PTFE-sealed dissolution container with hydrochloric acid at a constant temperature, followed by the addition of a strontium salt release agent (dissolution method I); alternatively, the sample can be... The sample was placed in a microwave digester and dissolved under high temperature and pressure with sulfuric acid, followed by the addition of a releasing agent, lanthanum salt (dissolution method II). An air-acetylene flame was then used for atomic digestion. The absorbance was measured at a wavelength of 285.2 nm using an absorption spectrometer. The matrix matching method was employed to eliminate the influence of the matrix and solvent on the measurement results.

7.2 Reagents Unless otherwise specified, only reagents confirmed to be of superior purity and Class II water conforming to GB/T 6682 shall be used in the analysis.

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