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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 18882.2-2017

Replacing GB/T 18882.2-2008

Chemical analysis methods for mixed rare earth oxide of ion-adsorption type rare earth ore - Part 2: Determination of the aluminium oxide content

离子型稀土矿混合稀土氧化物化学分析方法 第2部分：三氧化二铝量的测定

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Standardization Administration of the PRC

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1 Scope

China's national method for determining aluminium oxide in the mixed rare earth oxide produced from ion-adsorption rare earth ore, by two alternative routes: inductively coupled plasma atomic emission spectrometry and titration. Ion-adsorption deposits are the weathered granite clays of southern China, and they are the world's main source of the heavy rare earths - the elements that no other deposit type supplies in quantity. In these ores the rare earths are not locked in a mineral lattice but held on clay surfaces, from which they are stripped by leaching with a salt solution and then precipitated as a mixed oxide.

That is what makes them valuable and also what makes them analytically awkward: the same leach that takes the rare earths off the clay also takes aluminium off it, and aluminium follows the rare earths through precipitation. Aluminium oxide in the product is therefore the characteristic contaminant of this ore type, and it matters commercially because it is inert dilution in a material sold on rare earth content, and technically because it interferes with the solvent extraction circuit that separates the individual elements downstream. The standard gives two methods for the same determination so that a laboratory can choose according to its equipment and the level present. The ICP-OES route dissolves the sample in a mixed acid attack including hydrofluoric and perchloric acid and measures aluminium against matrix-matched calibration, which is faster and better at low levels; the titration route is the classical alternative, independent of an emission instrument. Issued on 14 October 2017 and in force since 1 May 2018, it replaces GB/T 18882.2-2008.

This Part of GB/T 18882 specifies the determination method of aluminum oxide content in mixed rare earth oxide of ion-absorpted type rare earth ore. This Part applies to the determination of aluminum oxide content in mixed rare earth oxide of ion-absorpted type rare earth ore, including two methods: Method

2 Method 1: Inductively coupled plasma atomic emission

spectrometry (ICP-OES)

2.1 Principle of the method Decompose the test sample by nitric acid and hydrofluoric acid; after the perchloric acid has smoked out, dissolve it in hydrochloric acid to make it clear; use approximate matrix matching to eliminate the rare earth matrix interference; directly excite by argon plasma light source; perform the spectrum measurement.

2.2 Reagents and materials Unless otherwise specified, all reagents used in this Part are analytical reagents that meet national or industry standards, and the water used is grade-II water.

2.2.4 Nitric acid (rho =

2.2.5 Hydrogen peroxide (30%).

2.2.6 Nitric acid (1+1).

2.3.1 Inductively coupled plasma atomic emission spectrometer, resolution <

0.006 nm (at 200 nm).

2.3.2 Light source: argon plasma light source.

2.4 Test sample

2.4.1 The particle size of the test sample is less than

0.074 mm.

2.4.2 Pre-dry the sample at 105 °C ~ 110 °C for 2 h; then, place it in a desiccator to cool to room temperature.

2.5 Procedure

2.5.1 Test material Weigh 0.1g of test sample (2.4), accurate to 0.000 1 g.

2.5.2 Number of determinations Independently perform two determinations; take the average value.

2.5.3 Blank test Carry out a blank test together with the test material (2.5.1).

2.5.4 Preparation of analytical solution Place the test materials (2.5.1) in a 150 mL polytetrafluoroethylene beaker; add a small amount of water; add 2 mL of nitric acid (2.2.6); heat to decompose for 3 min ~ 5 min; add 2 mL of hydrofluoric acid (2.2.2) and continue to decompose for 3 min ~ 5 min; add 3 mL of perchloric acid (2.2.3) to smoke and evaporate to nearly dry; remove and cool slightly; add 10 mL of hydrochloric acid (2.2.7) and add 3 ~ 5 drops of hydrogen peroxide (2.2.5); heat to dissolve until clear; transfer the test solution into a 100 mL volumetric flask; use water to dilute to the mark; mix well; test.

2.5.5 Series standard configuration According to Table 2, transfer the standard stock solution into six 100 mL volumetric flasks; use water to dilute to the mark; mix well; test. Unless otherwise specified, all reagents used in this Part are analytical reagents that meet national or industry standards, and the water used is grade-II water.

3.2.2 Hydrofluoric acid (rho =

3.2.3 Hydrogen peroxide (30%).

3.2.5 Nitric acid (rho =

3.2.6 Oxalic acid solution (100 g/L).

3.2.7 Sodium hydroxide solution (200 g/L).

3.2.8 Hydrochloric acid (1+1).

3.2.9 Hydrochloric acid (1+4).

3.2.10 Ammonia water (1+1).

3.2.11 EDTA (Ethylene Diamine Tetraacetic Acid) solution (about

0.05 mol/L): Weigh 20 g of EDTA and dissolve it in a small amount of water; transfer it into a 1 000 mL volumetric flask; use water to fix the volume; mix well.

3.2.12 Cresol red indicator (2 g/L): Weigh

0.2 g of cresol red and dissolve it in 100 mL of ethanol solution (1+1).

3.2.13 Phenolphthalein ethanol solution (10 g/L).

3.2.14 Acetic acid-sodium acetate buffer solution (pH 5.5): Weigh 200 g of sodium acetate and dissolve it in a small amount of water; transfer it into a 1 000 mL volumetric flask; add 10 mL of glacial acetic acid (w >= 99.5%); use water to fix the volume; mix well.

3.2.15 Xylenol orange indicator (4 g/L): Weigh

0.4 g of xylenol orange and 20 g of potassium nitrate; dissolve in a small amount of water; transfer to a 100 mL volumetric flask; use water to fix the volume; mix well. Store in a brown bottle.

3.2.16 Sodium fluoride solution (40 g/L).

3.2.17 Aluminum standard solution: Weigh 1.000 0 g of metallic aluminum (spectrally pure, remove all surface oxides before use) into a 500 mL beaker; add 50 mL of water; then, add 40 mL of hydrochloric acid (3.2.1); dissolve at low temperature until clear (add hydrochloric acid and water in between); cool. Transfer to a 1 000 mL volumetric flask; use hydrochloric acid (5+95) to dilute to the mark; mix well. This standard solution contains 1 mg of aluminum in 1 mL.

3.2.18 Zinc sulfate solution (about

0.1 mol/L): Weigh 30 g of zinc sulfate ($ZnSO_4·7H_2O$) and dissolve it in an appropriate amount of water; transfer it into a 1 000 mL volumetric flask; use water to fix the volume; mix well.

3.2.19 Zinc sulfate standard solution.

3.2.19.1 Preparation: Weigh

2.4 g of zinc sulfate ($ZnSO_4·7H_2O$) and dissolve it in a small amount of water; transfer it into a 1 000 mL volumetric flask; use water to fix the volume; mix well.

3.2.19.2 Calibration: Pipette

10.00 mL of aluminum standard solution (3.2.17) into a 250 mL conical flask; add 20 mL of LEDTA solution (3.2.11); add 1 ~ 2 drops of phenolphthalein ethanol solution (3.2.13); use sodium hydroxide solution (3.2.7) to neutralize until red appears. Use hydrochloric acid (3.2.9) to neutralize until colorless and add 1 drop in excess; add 20 mL of acetic acid-sodium acetate buffer solution (3.2.14); boil slightly at low temperature for 1 min ~ 2 min; remove and cool. Perform the following operations according to the analytical steps