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RARE EARTH INDUSTRY STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

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Chemical analysis methods of ion type rare earth ore - Determination of total rare earth ion phase

离子型稀土原矿化学分析方法 离子相稀土总量的测定

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Chemical analysis methods of ion type rare earth ore - Determination of total rare earth ion phase

1 Scope

This Standard specifies the method for determining total rare earth ion phase in ion type rare earth ore.

This Standard is applicable to the determination of total rare earth ion phase in ion type rare earth ore. It includes 3 methods: Method 1 Inductively Coupled Plasma Mass Spectrometry; Method 2 Inductively Coupled Plasma Atomic Emission Spectrometry and Method 3 EDTA Volumetric Method. The measurement range of Method 1 is: 0.010%~0.50%. The measurement range of Method 2 is: 0.020%~0.50%. The measurement range of Method 3 is: 0.020%~0.50%. Among them, Method 3 is not applicable to the determination of samples with an iron content greater than 10 after leaching or a copper content greater than 5 [Translator's note: This sentence of original Chinese is ambiguous and incomprehensible].

2 Method 1: Inductively Coupled Plasma Mass Spectrometry

2.1 Method principle

The test material is leached with ammonium sulfate solution. In dilute nitric acid medium, argon plasma is used as the ionization source. The mass fractions of fifteen rare earth elements are determined by mass spectrometry. The sum of the mass fractions is the total rare earth ion phase. Use the internal standard method to perform calibration.

2.2 Reagents and materials

2.2.1 Hydrogen peroxide (analytical grade).

2.2.2 Nitric acid [high purity (MOS)].

2.2.3 Nitric acid (1+1) (prepared with MOS grade nitric acid).

2.2.4 Ammonium sulfate solution (20 g/L): Weigh 20 g of ammonium sulfate into a 500 mL beaker. Dissolve in 300 mL of water until clear. Transfer to a 1000 mL volumetric flask. Dilute to volume with water. Mix well.

2.2.5 Lanthanum standard solution: Weigh 0.1000 g of lanthanum oxide [w (REO) >99.5%, w (La_2O_3/REO) >99.99%] calcined at 950°C for 1 h and place in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Add 2 mL of hydrogen peroxide (2.2.1).

Heat at low temperature until they are completely dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of lanthanum oxide.

2.2.6 Cerium standard solution: Weigh 0.1000 g of cerium oxide [w (REO) >99.5%, w (CeO_2/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Add 2 mL of hydrogen peroxide (2.2.1). Heat at low temperature until they are completely dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of cerium oxide.

2.2.7 Praseodymium standard solution: Weigh 0.1000 g of praseodymium oxide [w (REO) >99.5%, w (Pr_6O_{11}/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of praseodymium oxide.

2.2.8 Neodymium standard solution: Weigh 0.1000 g of neodymium oxide [w (REO) >99.5%, w (Nd_2O_3/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of neodymium oxide.

2.2.9 Samarium standard solution: Weigh 0.1000 g of samarium oxide [w (REO) >99.5%, w (Sm_2O_3/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of samarium oxide.

2.2.10 Europium standard solution: Weigh 0.1000 g of europium oxide [w (REO) >99.5%, w (Eu_2O_3/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature to dissolve until clear. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of europium oxide.

2.2.11 Gadolinium standard solution: Weigh 0.1000 g of gadolinium oxide [w (REO) >99.5%, w (Gd_2O_3/REO) >99.99%] calcined at 950 °C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature to dissolve until clear. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to volume with water. Mix well. 1 mL of this solution contains 1000 μg of gadolinium oxide.

2.2.12 Terbium standard solution: Weigh 0.1000 g of terbium oxide [w (REO) >99.5%, w (Tb_4O_7/REO) >99.99%] calcined at 950°C for 1 h and place in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Add 2 mL of hydrogen peroxide (2.2.1). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100

mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of terbium oxide.

2.2.13 Dysprosium standard solution: Weigh 0.1000 g of dysprosium oxide [w (REO) >99.5%, w (Dy_2O_3/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature to dissolve until clear. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of dysprosium oxide.

2.2.14 Holmium standard solution: Weigh 0.1000 g of holmium oxide [w (REO) >99.5%, w (Ho_2O_3/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of holmium oxide.

2.2.15 Erbium standard solution: Weigh 0.1000 g of erbium oxide [w (REO) >99.5%, w (Er_2O_3/REO) >99.99%] calcined at 950°C for 1 h and place in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature to dissolve until clear. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of erbium oxide.

2.2.16 Thulium standard solution: Weigh 0.1000 g of thulium oxide [w (REO) >99.5%, w (Tm_2O_3/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature until dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of thulium oxide.

2.2.17 Ytterbium standard solution: Weigh 0.1000 g of ytterbium oxide [w (REO) >99.5%, w (Yb_2O_3/REO) >99.99%] calcined at 950°C for 1 h and place in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Add 2 mL of hydrogen peroxide (2.2.1). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of ytterbium oxide.

2.2.18 Lutetium standard solution: Weigh 0.1000 g of lutetium oxide [w (REO) >99.5%, w (Lu_2O_3/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of lutetium oxide.

2.2.19 Yttrium standard solution: Weigh 0.1000 g of yttrium oxide [w (REO) >99.5%, w (Y_2O_3/REO) >99.99%] calcined at 950°C for 1 h in a 100 mL beaker. Add 10 mL of nitric acid (2.2.3). Add 2 mL of hydrogen peroxide (2.2.1). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1000 μg of yttrium oxide.

for 1 h.

3.2.18 Lutetium oxide: [w (REO) >99.5%, w (Lu_2O_3/REO) >99.99%], calcined at 950℃ for 1 h.

3.2.19 Yttrium oxide: [w (REO) >99.5%, w (Y_2O_3/REO) >99.99%], calcined at 950℃ for 1 h.

3.2.20 Cerium standard stock solution: Weigh 0.4000 g of cerium oxide (3.2.6) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Add 2 mL of hydrogen peroxide (3.2.1). Heat at low temperature until they are completely dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 4 mg of cerium oxide.

3.2.21 Samarium standard stock solution: Weigh 0.500 g of samarium oxide (3.2.9) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 5 mg of samarium oxide.

3.2.22 Europium standard stock solution: Weigh 0.300 g of europium oxide (3.2.10) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 3 mg of europium oxide.

3.2.23 Gadolinium standard stock solution: Weigh 0.4000 g of gadolinium oxide (3.2.11) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 4 mg of gadolinium oxide.

3.2.24 Terbium standard stock solution: Weigh 0.2000 g of terbium oxide (3.2.12) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 2 mg of terbium oxide.

3.2.25 Dysprosium standard stock solution: Weigh 0.3500 g of dysprosium oxide (3.2.13) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 3.5 mg of terbium oxide.

3.2.26 Holmium standard stock solution: Weigh 0.4000 g of holmium oxide (3.2.14) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric

flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 4 mg of holmium oxide.

3.2.27 Erbium standard stock solution: Weigh 0.3000 g of erbium oxide (3.2.15) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 3 mg of erbium oxide.

3.2.28 Thulium standard stock solution: Weigh 0.2000 g of thulium oxide (3.2.16) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 2 mg of thulium oxide.

3.2.29 Ytterbium standard stock solution: Weigh 0.3000 g of ytterbium oxide (3.2.17) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 3 mg of ytterbium oxide.

3.2.30 Lutetium standard stock solution: Weigh 0.2000 g of lutetium oxide (3.2.18) and place in a 100 mL beaker. Add 10 mL of nitric acid (3.2.3). Heat at low temperature until they are dissolved. Remove and cool. Transfer the solution to a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 2 mg of lutetium oxide.

3.2.31 Mixed standard stock solution of rare earth oxides: According to Table 5, weigh the corresponding amount (≥50 mg) of single rare earth oxides (3.2.5~3.2.19) into a 250 mL beaker. Add 20 mL of hydrochloric acid (3.2.2) and 1 mL of hydrogen peroxide (3.2.1) and heat to dissolve completely. Cool. Transfer to three 500 mL volumetric flasks respectively. According to Table 5, transfer the corresponding amount (<50 mg) of standard stock solution (3.2.20~3.2.29). Combine into the 500 mL volumetric flask with the same serial number. Add 40 mL of hydrochloric acid (3.2.2). Dilute to the scale with water. Mix well. 1 mL of this solution contains 2 mg of rare earth oxides.

3.2.32 Argon [φ (Ar) >99.99%]

The test material is leached with ammonium sulfate solution and titrated with disodium ethylenediaminetetraacetate (EDTA) standard solution at pH 5.5 using xylenol orange as indicator.

4.2 Reagents and materials

4.2.1 Hydrochloric acid (analytical grade).

4.2.2 Acetylacetone (1+9).

4.2.3 Hydrochloric acid (1+4).

4.2.4 Ammonia water (1+4).

4.2.5 Ammonium sulfate solution (20 g/L): Weigh 20 g of ammonium sulfate into a 500 mL beaker. Dissolve in 300 mL of water until clear. Transfer to a 1000 mL volumetric flask. Dilute to the scale with water. Mix well.

4.2.6 Sulfosalicylic acid solution (100 g/L): Weigh 100 g of sulfosalicylic acid solution into a 500 mL beaker. Dissolve in 300 mL of water until clear. Transfer to a 1000 mL volumetric flask. Dilute to the scale with water. Mix well.

4.2.7 Acetylacetone (1+9): Pipette 10 mL of acetylacetone (4.2.2) into a 100 mL volumetric flask. Dilute to the scale with water. Mix well.

4.2.8 Hexamethylenetetramine solution (200 g/L): Weigh 200 g of hexamethylenetetramine into a 500 mL beaker. Dissolve in 300 mL of water until clear. Transfer to a 1000 mL volumetric flask. Add 25 mL of hydrochloric acid (4.2.1). Dilute to the scale with water. Mix well. Each 100 mL contains 2.5 mL of hydrochloric acid.

4.2.9 Xylenol orange indicator (2 g/L): Weigh 0.2000 g of xylenol orange indicator into a 250 mL beaker. Dissolve it in 100 mL (1 +1) ethanol solution.

4.2.10 Congo red test paper.

4.2.11 Zinc standard stock solution: Weigh 0.2000 g of metallic zinc [w (Zn) >99.9%] into a 250 mL beaker. Dissolve in 20 mL hydrochloric acid (4.2.1) until clear. Cool. Transfer to a 200 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 1 mg of zinc.

4.2.12 Zinc standard solution: Pipette 10 mL of zinc standard stock solution into a 100 mL volumetric flask. Dilute to the scale with water. Mix well. 1 mL of this solution contains 0.1 mg of zinc.

4.2.13 EDTA standard solution (0.002 mol/L)

Preparation: Weigh 1.5 g of disodium ethylenediaminetetraacetate dried at 80°C for 2 h and dissolve it in 100 mL of water in a 250 mL beaker. Transfer to a 2000 mL

volumetric flask. Dilute to the scale with water. Mix well. Store in a plastic bottle.

Calibration: Pipette 20.00 mL of zinc standard solution (4.2.12) into a 300 mL conical flask. Add 50 mL of water. Add a small piece of Congo red test paper. Use hydrochloric acid (4.2.3) and ammonia water (4.2.4) to adjust the test paper to purple-red. The pH value of the solution is 5~5.5. Add 5 mL of hexamethylenetetramine solution (4.2.8). Add 2 drops of xylenol orange indicator (4.2.9). Titrate with EDTA standard solution (4.2.13) until the solution changes from purple-red to bright yellow, which is the end point. Two people measure 8 portions in parallel. The extreme difference of the volume of EDTA standard solution (4.2.12) consumed should not exceed 0.10 mL. Take the average value.

Calculate the concentration of EDTA solution according to formula (3):

$$c = \frac{\rho \times V_1}{(V_2 - V_3)M} \qquad \cdots\cdots(3)$$

Where,

c - EDTA solution concentration, in moles per liter (mol/L);

ρ - mass concentration of zinc standard solution, in milligrams per milliliter (mg/mL);

V_1 - volume of zinc standard solution pipetted, in milliliters (mL);

V_2 - volume of EDTA standard solution consumed during calibration, in milliliters (mL);

V_3 - volume of EDTA standard solution consumed in blank determination, in milliliters (mL);

M - molar mass of zinc, in grams per mole (g/mol).

4.3 Specimens

Grind the specimen to a particle size of less than 1 mm. Dry it in a drying oven at 105°C for 1 h. Cool it to room temperature in a desiccator.

4.4 Analysis steps

4.4.1 Test material

Weigh 20.0 g of specimen (4.3) to an accuracy of 0.0001 g.

4.4.2 Number of measurements

Carry out two independent measurements. Take the average value.

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------ The End ------