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Scandium oxide

氧化钪

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Scandium oxide

1 Scope

This standard specifies the requirements, test methods, inspection rules and packaging, marking, transportation, storage and quality certificates of scandium oxide.

This standard is applicable to scandium oxide produced by extraction method, ion exchange method or extraction chromatography method for use in laser, electric light source, atomic energy, electronics, metallurgy and other fields.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this document. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

GB/T 12690.2 Chemical analysis methods for non-rare earth impurity of rare earth metals and their oxides - Part 2: Determination of ignition loss content of rare earth oxides - Gravimetric method

GB/T 12690.3 Chemical analysis methods for non-rare earth impurity of rare earth metals and their oxides - Part 3: Determination of water content of rare earth oxides - Gravimetric method

GB/T 17803 Designation system for rare earth products

3 Requirements

3.1 Product designations

The products are divided into five designations according to chemical composition: Sc_2O_3-5N5, Sc_2O_3-5N, Sc_2O_3-4N, Sc_2O_3-3N5, Sc_2O_3-3N; the method of expressing product designations shall comply with GB/T 17803.

3.2 Chemical composition

4.1.1 Refer to Appendix A for the analysis method of total rare earth (REO). When the total amount of rare earths measured is more than 99%, the actual value of the total amount of rare earths is calculated by the subtraction method, that is (100% - Σ non-rare earth impurities).

4.1.2 Refer to Appendix B for the analysis method of rare earth impurity content.

4.1.3 Refer to Appendix C for the analysis method of non-rare earth impurity content.

4.1.4 The analysis method of ignition loss and moisture shall be carried out in accordance with the provisions of GB/T 12690.2 and GB/T 12690.3.

4.1.5 The absolute purity of scandium oxide (Sc_2O_3) is calculated as [100% - (Σ Rare earth impurities + Σ non-rare earth impurities)].

4.1.6 The relative purity (Sc_2O_3/REO) of scandium oxide (Sc_2O_3) is calculated as (100% - Σ rare earth impurities/REO).

4.2 Numerical rounding

It is performed according to the provisions of GB/T 8170.

4.3 Appearance quality

Under natural scattered light, visually check the appearance quality.

5 Inspection rules

5.1 Inspection and acceptance

5.1.1 The product is inspected by the supplier's quality inspection department, to ensure that the product meets the requirements of this standard; meanwhile it shall fill the product quality certificate.

5.1.2 The purchaser shall inspect the received product. If the inspection result does not conform to the provisions of this standard, it shall notice the supplier within 2 months from the date of receipt of the product; the supplier and the buyer shall negotiate and resolve it. If arbitration is needed, it can be entrusted to a unit recognized by both parties to conduct a joint sampling on the buyer's site.

5.2 Group-batching

Products shall be submitted for inspection in batches; each batch shall consist of products of the same designation.

5.3 Inspection items

c) The name of the product manufacturer;

d) Product name and designation;

e) Lot number;

f) Gross weight and net weight;

g) Packing date;

h) Sign or words of "moisture-proof".

6.1.2 The products are packed in double-layer plastic bags or plastic bottles, each bag (bottle) has a net weight of 0.1 kg, 0.25 kg, 0.5 kg, 1 kg. Then put the bag (bottle) in an iron drum (wooden box, carton or plastic box); the net weight of each iron drum (wooden box, carton or plastic box) is 0.5 kg, 1 kg, 5 kg, 10 kg.

6.2 Transportation and storage

The product shall be protected from rain and moisture during transportation; it shall be stored in a dry place and shall not be stacked in the open air.

6.3 Quality certificate

Each batch of products shall be accompanied by a quality certificate, which shall indicate:

a) Product name;

b) Supplier's name, address, telephone, fax;

c) Name, address, telephone, fax of the manufacturer of raw mineral products;

d) The name, address, telephone, fax of the product manufacturer;

e) Designation and batch number;

f) Quantity (net weight and number of pieces);

g) Various analysis inspection results and the supplier's quality inspection department's imprint;

h) Date of issuance;

i) This standard number or contract number;

j) Production date (indicate the year, month, day; if the production date has

Appendix A

(Informative)

Chemical analysis method of metal scandium and its oxide - Determination of the total amount of rare earths - Gravimetric method

A.1 Scope

This method specifies the determination method for the total amount of rare earths in scandium oxide.

This method is suitable for the determination of the total amount of rare earths in scandium oxide. Measuring range: 90.00% ~ 99.50%.

A.2 Principle of the method

The sample is dissolved in hydrochloric acid; the rare earth is precipitated by ammonia water to separate calcium and magnesium. The precipitate is dissolved by hydrochloric acid; the oxalic acid is used to precipitate rare earths. The total amount of rare earth oxide is determined by gravimetric method after precipitation and ignition.

A.3 Reagents

A.3.1 Hydrochloric acid (ρ = 1.19 g/mL), excellent grade pure.

A.3.2 Hydrochloric acid (1 + 1), excellent grade pure.

A.3.3 Ammonia (1 + 1), excellent grade pure.

A.3.4 Oxalic acid solution (50 g/L), excellent grade pure.

A.3.5 Oxalic acid solution (20 g/L), excellent grade pure.

A.3.6 Ammonia-ammonium chloride solution (20 g/L), which is adjust to pH 9 ~ 10 with ammonia water (A.3.3).

A.3.7 Cresol red indicator (1 g/L), prepared with ethanol.

A.4 Specimen

The oxide specimen is dried at 105 °C for 2 h, placed in a desiccator, cooled to room temperature, weighed immediately.

A.5 Analytical procedures

A.5.1 Sample

Appendix B

(Informative)

Metal scandium and its oxide chemical analysis method - Determination of lanthanum, cerium, praseodymium, neodymium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, lutetium, yttrium

B.1 Scope

This Appendix specifies the method for determining the amount of lanthanum oxide, cerium oxide, praseodymium oxide, neodymium oxide, samarium oxide, europium oxide, gadolinium oxide, terbium oxide, dysprosium oxide, holmium oxide, erbium oxide, thulium oxide, ytterbium oxide, lutetium oxide and yttrium oxide in scandium oxide.

This appendix applies to the determination of lanthanum oxide, cerium oxide, praseodymium oxide, neodymium oxide, samarium oxide, europium oxide, gadolinium oxide, terbium oxide, dysprosium oxide, holmium oxide, erbium oxide, thulium oxide, ytterbium oxide, lutetium oxide, yttrium oxide in scandium oxide. This Appendix contains two methods. Method 1 is inductively coupled plasma spectroscopy; the measurement range is (mass fraction): 0.0010% ~ 0.10%; Method 2 is inductively coupled plasma mass spectrometry; the measurement range is (mass fraction): 0.00003% ~ 0.010%. When the measurement ranges of method 1 and method 2 overlap, method 2 is used as the arbitration method.

This Appendix also applies to the determination of lanthanum, cerium, praseodymium, neodymium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, lutetium, yttrium in scandium.

B.2 Inductively coupled plasma spectroscopy (Method 1)

B.2.1 Principle of the method

The specimen is dissolved in hydrochloric acid; in a dilute hydrochloric acid medium, it is directly excited by an argon plasma light source, for the spectrum determination; the influence of the matrix on the determination is corrected by the matrix matching method.

B.2.2 Reagents

B.2.2.1 Hydrogen peroxide (30%), excellent grade pure.

B.2.2.2 Hydrochloric acid (1 + 1), excellent grade pure.

B.2.3 Apparatus

B.2.3.1 Inductively coupled plasma spectrometer, resolution < 0.006 nm (at 200 nm)>.

B.2.3.2 Light source: Argon plasma light source.

B.2.4 Specimen

B.2.4.1 Oxide specimens are dried at 105 °C for 2 hours, placed in a desiccator, cooled to room temperature, weighed immediately.

B.2.4.2 The surface oxide layer shall be removed from the metal specimen and weighed immediately after sampling.

B.2.5 Analytical procedures

B.2.5.1 Sample

B.2.5.1.1 Oxide sample

Weigh 1.000 g of specimen (B.2.5.1), accurate to 0.0001 g.

B.2.5.1.2 Metal sample

Weigh 0.862 g of sample (B.2.4.2), accurate to 0.0001 g.

B.2.5.2 Number of determinations

Weigh two sets of samples; perform parallel determination; take the average value.

B.2.5.3 Blank test

Do a blank test with the sample.

B.2.5.4 Preparation of analysis test solution

Place the sample (B.2.5.1) in a 100 mL beaker; add 10 mL of water; add 20 mL of hydrochloric acid (B.2.2.2); heat at low temperature to completely dissolve; evaporate to about 5 mL; cool to room temperature. Transfer to a 100 mL volumetric flask; use water to dilute to the mark; mix well; prepare for use.

B.2.5.5 Preparation of standard series solutions

Transfer the scandium oxide matrix solution (B.2.2.5) and each rare earth oxide standard solution (B.2.2.6 ~ B.2.2.8) into seven 50 mL volumetric flasks according to Table B.1; add 5 mL of hydrochloric acid (B.2.2.2); use water to dilute to the mark; mix well; prepare a standard series solution; use the standard

C.3.3 Hydrochloric acid (1 + 19), excellent grade pure.

C.3.4 Nitric acid (1 + 1), excellent grade pure.

C.3.5 Hydrofluoric acid, excellent grade pure.

C.3.6 Perchloric acid, excellent grade pure.

C.3.7 Scandium oxide matrix solution: Weigh 5.0000 g of scandium oxide [w (Sc_2O_3/ΣREO) ≥ 99.9995%, w (ΣREO) ≥ 99.5%] that has been burned at 900 °C for 1 h; place it in 200 mL beaker; add 40 mL of hydrochloric acid (C.3.2); heat at low temperature to complete dissolution; evaporate to about 10 mL; cool to room temperature; transfer to a 50 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 100 mg of scandium oxide.

C.3.8 Silicon standard storage solution: Weigh 0.2139 g of spectrally pure silica (spectrally pure) that has been pre-fired at 950 °C for 2 h and placed in a desiccator to cool to room temperature; place it in a platinum crucible (premium grade) which contains 5 g of anhydrous sodium carbonate; mix well; melt at 1000 °C for 20 min; take out and cool it slightly; use hot water to extract it; make its volume reach to 1000 mL. 1 mL of this solution contains 100 μg of silicon. Store it in a plastic bottle. Use pure water to dilute this solution into 1 mL of silicon standard solution containing 10 μg and 1 μg, respectively.

C.3.9 Iron standard storage solution: Pipette 1.4298 g of iron trioxide (spectrally pure) dried at 110 °C for 1 h into a 200 mL beaker; add 50 mL of hydrochloric acid (C.3.2) to heat to dissolve; cool to room temperature; transfer it into a 1000 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 1 mg iron. Use hydrochloric acid (5 + 95) to dilute this solution into 1 mL of iron standard solutions containing 100 μg, 10 μg, 1 μg, respectively.

C.3.10 Calcium standard storage solution: Pipette 2.4951 g of calcium carbonate (spectrally pure) baked at 110 °C for 1 h into a 200 mL beaker; add 50 mL of hydrochloric acid (C.3.2) to heat to dissolve; cool to room temperature; transfer it into a 1000 mL volumetric flask; use water to dilute it to the mark; mix well. 1 mL of this solution contains 1 mg of calcium. Use hydrochloric acid (C.3.3) to dilute this solution into 1 mL of calcium standard solutions containing 100 μg, 10 μg, 1 μg, respectively.

C.3.11 Zirconium standard storage solution: Weigh 3.5328 g of zirconium oxychloride ($ZrOCl_2 \cdot 8H_2O$) (spectrally pure) into a 200 mL beaker; add 5 mL of nitric acid (C.3.4) and 50 mL of water; after heating to dissolve, transfer it into a 1000 mL volumetric flask; use water to dilute it to the mark; mix well. 1 mL of this solution contains 1 mg of zirconium. Use hydrochloric acid (C.3.3) to dilute this solution into 1 mL of zirconium standard solutions containing 100 μg, 10 μg, 1 μg, respectively.

this solution into 1 mL of sodium standard solution containing 100 μg, 10 μg, 1 μg, respectively.

C.3.18 Nickel standard storage solution: Weigh 1.2730 g of nickel oxide (spectrally pure) that has been baked at 110 °C for 1 h, in a 200 mL beaker; add 50 mL of hydrochloric acid (C.3.2) to dissolve it; cool to room temperature; transfer it into a 1000 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 1 mg of nickel. Use hydrochloric acid (C.3.3) to dilute this solution into 1 mL nickel standard solutions containing 100 μg, 10 μg, 1 μg, respectively.

C.3.19 Standard storage solution of thorium: Weigh 2.3800 g of thorium nitrate [$Th(NO_3)_4 \cdot 4H_2O$] (spectrally pure) into a 200 mL beaker; add 100 mL hydrochloric acid (C.3.2) to dissolve it; cool to room temperature; transfer it in a 1000 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 1 mg thorium. Use hydrochloric acid (C.3.3) to dilute this solution into 1 mL of thorium standard solutions containing 100 μg, 10 μg, 1 μg, respectively.

C.3.20 Tantalum standard storage solution: Weigh 1.0000 g of metallic tantalum (spectrally pure) into a platinum crucible; add 6 mL ~ 8 mL of hydrofluoric acid (C.3.5); dissolve and evaporate at low temperature to near dryness; use (100 g/L) tartaric acid to extract it; dissolve at slight heat; cool to room temperature; transfer it into a 1000 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 1 mg of tantalum. Use hydrochloric acid (C.3.3) to dilute this solution into 1 mL of tantalum standard solutions containing 100 μg, 10 μg, 1 μg, respectively.

C.3.21 Argon [Φ (Ar) ≥ 99.99%].

C.4 Instruments

C.4.1 Inductively coupled plasma spectrometer, resolution < 0.006 nm.

C.4.2 Argon plasma light source.

C.5 Specimen

C.5.1 Oxide specimens are dried at 105 °C for 2h, placed in a desiccator, cooled to room temperature, weighed immediately.

C.5.2 The surface oxide layer shall be removed from the metal specimen and weighed immediately after sampling.

C.6 Analytical procedure

C.6.1 Sample

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