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Iron ores - Determination of total rare earth content - Inductively coupled plasma atomic emission spectrometric method

铁矿石 稀土总量的测定 电感耦合等离子体原子发射光谱法

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Warning - Personnel using this document shall have practical experience in regular laboratory work. This document does not identify all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions, which are stipulated in relevant national regulations.

1 Scope

This document describes a method for the determination of total rare earth content, by inductively coupled plasma atomic emission spectrometry.

This document is applicable to the determination of the total rare earth content in iron ore, iron concentrate, sinter, pellet mineral products. Determination range (mass fraction): 0.10% ~ 15.00%.

2 Normative references

The contents of the following documents constitute essential provisions of this document through normative references in the text. Among them, for dated reference documents, only the version corresponding to the date applies to this document; for undated reference documents, the latest version (including all amendments) applies to this document.

GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions

GB/T 6379.2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 6730.1 Iron ores - Preparation of pre-dried test samples for chemical analysis

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

5.8 Hydrochloric acid, 5 + 95.

5.9 Nitric acid, $\rho \approx 1.42$ g/mL.

5.10 Nitric acid, 1 + 1.

5.11 Hydrogen peroxide, 30%.

5.12 Sodium hydroxide lotion, 20 g/L.

Weigh 2 g of sodium hydroxide. Dissolve it in 100 mL of water.

5.13 Triethanolamine, 5 + 95.

Measure 5 mL of triethanolamine. Add 95 mL of water. Mix well.

5.14 Yttrium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of yttrium oxide [w (Y_2O_3/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of hydrochloric acid (see 5.7). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.15 Lanthanum oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of lanthanum oxide [w (La_2O_3/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of hydrochloric acid (see 5.7). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.16 Cerium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of cerium oxide [w (CeO_2/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of nitric acid (see 5.10). Heat at low temperature. Dropwise add hydrogen peroxide (see 5.11), until it is completely dissolved. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.17 Praseodymium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of praseodymium oxide [w (Pr_6O_{11}/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of hydrochloric acid (see 5.7). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.18 Neodymium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of neodymium oxide [w (Nd_2O_3/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of hydrochloric acid (see 5.7). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.19 Samarium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of samarium oxide [w (Sm_2O_3/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of hydrochloric acid (see 5.7). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.20 Europium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of europium oxide [w (Eu_2O_3/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of hydrochloric acid (see 5.7). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.21 Gadolinium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of gadolinium oxide [w (Gd_2O_3/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of hydrochloric acid (see 5.7). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.22 Terbium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of terbium oxide [w (Tb_4O_7/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of nitric acid (see 5.10). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

5.23 Dysprosium oxide standard storage solution, 1 mg/mL.

Weigh 0.1000 g of dysprosium oxide [w (Dy_2O_3/REO) ≥ 99.99%, w (REO) ≥ 99.5%] that has been burned at 950 °C for 1 hour. Place it in a 100 mL beaker. Add 10 mL of hydrochloric acid (see 5.7). Heat at low temperature to dissolve it completely. Cool to room temperature. Transfer to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

6 Instruments and equipment

Unless otherwise specified, use usual laboratory equipment. Single-marked volumetric flasks, graduated pipettes, single-marked pipettes shall comply with the requirements of GB/T 12806, GB/T 12807, GB/T 12808, respectively. Corundum crucibles, beakers, volumetric flasks, etc., which are used in the test, are soaked in hydrochloric acid solution (see 5.8) for more than 24 hours, rinsed with water, dried, prepared for use.

6.1 Inductively coupled plasma atomic emission spectrometer shall meet the following requirements:

a) The resolution is less than 0.008 nm (at 200 nm);

b) Comply with the emission spectrometer calibration procedures and technical indicators required in JJG 768.

6.2 Electric hot plate: Temperature control range 50 °C ~350 °C.

6.3 Muffle furnace: Temperature control range 500 °C ~800 °C.

6.4 Analytical balance: Sensitivity 0.1 mg.

6.5 Corundum crucible.

7 Sampling and specimen preparation

7.1 Laboratory specimen

Sampling and specimen preparation shall be carried out, in accordance with GB/T 10322.1. Generally, the specimen particle size is less than 100 μm. If the content of combined water or easy oxides in the specimen is high, the particle size shall be less than 160 μm.

The requirements for high combined water and easy oxidation content are in accordance with GB/T 6730.1.

7.2 Preparation of pre-dried specimens

Thoroughly mix the laboratory specimens. Take samples using the increment specimen reduction method. According to the provisions of GB/T 6730.1, dry the specimen at a temperature of 105 °C ± 2 °C. Cool it to room temperature in a desiccator for later use.

8 Analytical procedures

8.1 Number of measurements

According to Appendix B, the same pre-dried specimen shall be measured independently at least twice.

Note: "Independent" means that the results of the second and subsequent measurements are not affected by the results of the previous measurements. In this analytical method, this condition means that the same measured object is independently and repeatedly measured, in a short period of time, by the same operator, using the same equipment and the same test method, in the same laboratory, including the use of appropriate recalibration.

8.2 Specimen size

Weigh approximately 0.50 g of the pre-dried specimen (see 7.2), accurate to 0.0001 g. The specimen weighing operation shall be carried out as quickly as possible, to prevent the specimen from absorbing moisture again.

8.3 Blank test and verification test

8.3.1 Blank test

Carry out a blank test, along with the specimen analysis. All reagents shall be taken from the same reagent bottle.

8.3.2 Verification test

Along with the specimen, analyze the standard samples of the same type, for verification tests.

8.4 Determination

8.4.1 Decomposition of specimens

Place the specimen (see 8.2) in a corundum crucible, which contains 3 g ~ 4 g of sodium hydroxide (see 5.1) that has been pre-baked, to remove moisture. Mix evenly. Add 4 g of sodium peroxide (see 5.2) to cover it. Place it in a muffle furnace (See 6.3). Gradually increase the temperature from low temperature to 750 °C and melt, until red and transparent for 5 min ~ 10 min. Shake twice in this process. Take out and cool.

8.4.2 Preparation of analytical solution

8.4.2.1 Move the cooled corundum crucible into a 400 mL beaker, which contains 100 mL of triethanolamine (see 5.13), 1 g of EDTA (see 5.3), 1 g of ascorbic acid (see 5.4), 1 g of hydroxylamine hydrochloride (see 5.5). Wait until the violent reaction stops. heat to boiling. Remove and rinse the crucible. Boil the solution for several minutes. Cool it slightly. Use quantitative medium-speed filter paper to filter it. Use sodium hydroxide washing solution (see 5.12), to rinse the beaker and sediment 6 ~ 7 times. Use 5 mL of hydrochloric acid (see 5.7), to dissolve the precipitate on the filter paper. Filter it into the volumetric flask. After the precipitate is dissolved, use dilute hydrochloric acid (see

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