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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 6730.84-2023

**Iron ores - Determination of total rare earth content -
Inductively coupled plasma atomic emission spectrometry**

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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Table of Contents

1 Scope
2 Normative references
3 Introduction...
4 Principles...
5 Reagents and materials...
5.9 Nitric acid, rho~
6 Instruments and equipment
6.4 Analytical balance: Sensitivity
7 Sampling and specimen preparation
8 Analytical procedures
8.2 Specimen size Weigh approximately
8.3 Blank test and verification test
8.4 Determination
8.4.2 Preparation of analytical solution
9 Result calculation and representation...

1 Scope

Part 84 of China's national series on the analysis of iron ores, determining total rare earth content by inductively coupled plasma atomic emission spectrometry. The method exists because of one deposit. Bayan Obo in Inner Mongolia is mined as an iron ore and is simultaneously the largest rare earth deposit in the world, supplying a large share of global production as a co-product of iron - so for that ore, and the tailings and process streams that come from it, the rare earth content is a valuation rather than an impurity check. The range the method covers, up to fifteen per cent, is far above anything found in ordinary iron ore and reflects exactly that. Determining the rare earths as a total rather than individually is what a process control or a valuation needs, and ICP-AES handles the group well because their emission lines are strong. This part applies to iron ore, iron concentrate, sinter and pellet products over a mass fraction range of 0.10 to 15.00 per cent.

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~

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 [$w(\text{Y}_2\text{O}_3/\text{REO}) \geq 99.99\%$, $w(\text{REO}) \geq 99.5\%$] 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 [$w(\text{La}_2\text{O}_3/\text{REO}) \geq 99.99\%$, $w(\text{REO}) \geq 99.5\%$] 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

(CeO₂/REO) $\geq$ 99.99%, w (REO) $\geq$ 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 (Pr₆O₁₁/REO) $\geq$ 99.99%, w (REO) $\geq$ 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 (Nd₂O₃/REO) $\geq$ 99.99%, w (REO) $\geq$ 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 (Sm₂O₃/REO) $\geq$ 99.99%, w (REO) $\geq$ 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 (Eu₂O₃/REO) $\geq$ 99.99%, w (REO) $\geq$ 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 (Gd₂O₃/REO) $\geq$ 99.99%, w (REO) $\geq$ 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 (Tb₄O₇/REO) $\geq$ 99.99%, w (REO) $\geq$ 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 (Dy₂O₃/REO) $\geq$ 99.99%, w (REO) $\geq$ 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