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NONFERROUS METAL INDUSTRY STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

ICS 71.040.40

CCS H 30

YS/T 575.27-2023

Methods for chemical analysis of bauxite - Part 27:

**Determination of elements content - Inductively coupled
plasma atomic emission spectrometric method**

铝土矿石化学分析方法 第 27 部分：元素含量的测定 电感耦合等离
子体原子发射光谱法

Issued on: December 20, 2023

Implemented on: July 01, 2024

Issued by: Ministry of Industry and Information Technology of PRC

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Methods for chemical analysis of bauxite - Part 27:

Determination of elements content - Inductively coupled plasma atomic emission spectrometric method

1 Scope

This document describes the method for determining the element content in bauxite ores (expressed as oxides: K_2O , Na_2O , CaO , MgO , Li_2O , ZnO , Fe_2O_3 , V_2O_5 , TiO_2 , Ga_2O_3 , MnO , SiO_2).

This document applies to the determination of element content in bauxite ores. The determination range is shown in Table 1.

2 Normative references

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

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

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

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Principle

The sample is melted with sodium hydroxide or lithium metaborate and leached with hydrochloric acid or nitric acid. The sample solution is introduced into an inductively coupled plasma atomic emission spectrometer, excited with an argon plasma light source, to measure the emission intensity of each element at a selected wavelength. Matrix effects on the measurement are corrected using the matrix matching method. The mass concentration of each element is calculated based on a standard working curve; the results are expressed as mass fraction.

5 Reagents or materials

Unless otherwise specified, only reagents of confirmed analytical grade are used in the analysis.

5.1 Water, GB/T 6682, Grade II.

5.2 Sodium hydroxide (superior purity).

5.3 Anhydrous lithium metaborate (superior purity).

5.4 Hydrochloric acid (1 + 1).

5.5 Nitric acid (1 + 1).

5.6 Hydrochloric acid (1 + 3).

5.7 Aluminum matrix solution (5 mg/mL): Weigh 5.00 g of metallic aluminum ($w_{\text{Al}} \geq 99.99\%$) into a 1000 mL beaker; cover with a watch glass; add a total of 200 mL of hydrochloric acid (5.4) in portions. Slowly heat until it is completely dissolved. Add a few drops of hydrogen peroxide; boil for several minutes; remove from the flask; cool it; transfer to a 1000 mL volumetric flask; use water to dilute it to the mark; shake well.

5.8 Sodium hydroxide matrix solution I: Weigh 3.0 g of sodium hydroxide (5.2) into a 300 mL beaker. Add 50 mL of water until completely dissolved. Cool it; transfer to a 200 mL plastic volumetric flask; use water to dilute it to the mark; shake well.

5.9 Sodium hydroxide matrix solution II: Weigh 1.5 g of sodium hydroxide (5.2) into several 300 mL beakers. Add 25 mL of water to each beaker until completely dissolved. Cool it; transfer to several 100 mL plastic volumetric flasks; set aside.

5.10 Lithium metaborate matrix solution I: Weigh 1.0 g of anhydrous lithium metaborate (5.3) into a 300 mL beaker; cover with a watch glass; add 50 mL of water; add 10 mL of nitric acid (5.5); slowly heat until completely dissolved; remove and cool it; transfer to a 200 mL volumetric flask; use water to dilute it to the mark; shake well.

5.11 Lithium metaborate matrix solution II: Weigh 0.5 g of anhydrous lithium metaborate (5.3) in several portions; place in several 300 mL beakers. Cover with a watch glass. Add 25 mL of water and 5 mL of nitric acid (5.5) to each beaker. Slowly heat until completely dissolved. Remove and cool it; transfer to several 100 mL volumetric flasks for later use.

5.12 Standard stock solution (1000 µg/mL): Prepare it according to Appendix A. 1 mL of this standard stock solution contains 1000 µg of each of potassium oxide, calcium oxide, magnesium oxide, sodium oxide, lithium oxide, zinc oxide, iron oxide, vanadium pentoxide, titanium dioxide, gallium oxide, manganese oxide, silicon dioxide.

5.13 Mixed standard solution I: Pipette 5.00 mL of each standard stock solution (5.12) as needed; place it into a 100 mL volumetric flask. Maintain the same acidity as the standard stock solution; use water to dilute it to the mark; shake well. The concentration of this mixed solution is 50 µg/mL (dilute when needed).

5.14 Mixed standard solution II: Pipette 40.00 mL of mixed standard solution I (5.13); place it into a 100 mL volumetric flask. Maintain the same acidity as the standard stock solution; use water to dilute it to the mark; shake well. The concentration of this mixed solution is 20 µg/mL (dilute when needed).

5.15 Mixed standard solution III: Pipette 10.00 mL of mixed standard solution I (5.13); place it into a 100 mL volumetric flask. Maintain the same acidity as the standard stock solution; use water to dilute it to the mark; shake well. The concentration of this mixed solution is 5 µg/mL (dilute when needed).

6 Instruments

6.1 Inductively coupled plasma atomic emission spectrometer (equipped with a high salt resistant atomizer): The instrument resolution is less than 0.006 nm (at 200 nm).

6.2 Platinum dish: 100 mL.

6.3 Silver crucible: 30 mL.

6.4 Oven: Controllable temperature is $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

6.5 High temperature furnace: Controllable temperature is $750\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$, $850\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$.

7 Sample

Grind the sample to pass through a 75 μm standard sieve; dry it in an oven (6.4) at $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 2 h; take it out and place it in a desiccator; cool it to room temperature and set aside.

8 Test steps

8.1 Test material

Weigh 0.20 g of sample (7), accurate to 0.0001 g.

8.2 Parallel test

Perform two parallel tests and take the average value.

8.3 Blank test

Perform a blank test with the sample (8.1).

8.4 Preparation of analytical solution

8.4.1 Sample dissolution method I

8.4.1.1 Applicable to the determination of potassium oxide, calcium oxide, magnesium oxide, lithium oxide, zinc oxide, iron oxide, vanadium pentoxide, titanium dioxide, gallium oxide, manganese oxide, silicon dioxide in bauxite ore.

8.4.1.2 Place the sample (8.1) in a silver crucible (6.3); add 3.0 g of sodium hydroxide (5.2). Place the silver crucible in a high-temperature furnace (6.5); heat from room temperature to approximately $750\text{ }^{\circ}\text{C}$. Maintain the melt for 20 minutes; remove the sample. Rotate the silver cru. Cool it. Place the silver crucible in a glass short-necked funnel with a diameter of not less than 9 cm. Insert the funnel into a 200 mL volumetric flask, which was pre-filled with 50 mL of hydrochloric acid (5.4). Add a small amount of boiling water to the silver crucible. After a vigorous reaction, combine the leaching solution with the volumetric flask. While hot, shake the solution in the volumetric flask several times, until all the melt is leached. Rinse the silver crucible with a small amount of hydrochloric acid (5.6). Finally, rinse the silver crucible and funnel with hot water. Cool to room temperature; use water to dilute it to the mark; shake well.

8.4.2 Sample dissolution method II

8.4.2.1 Applicable to the determination of potassium oxide, sodium oxide, calcium

oxide, magnesium oxide, zinc oxide, iron oxide, vanadium pentoxide, titanium dioxide, gallium oxide, manganese oxide in bauxite.

8.4.2.2 Place the sample (8.1) in a platinum dish, which is pre-filled with 1.0 g of anhydrous lithium metaborate (5.3). Stir thoroughly. Place the sample in a high-temperature furnace (6.5); heat from room temperature to approximately 850 °C. Maintain the temperature for 30 minutes; remove the sample. Cool the sample slightly. Add 30 mL of water and 10 mL of nitric acid (5.5). Heat the sample at low temperature until the melt is completely dissolved. Remove the sample; cool the sample to room temperature; transfer the sample to a 200 mL volumetric flask; use water to dilute the sample to the mark; shake well.

8.4.2.3 Pipette 5.00 mL of solution (8.4.2.2) into a 100 mL volumetric flask; add 10 mL of nitric acid (5.5); use water to dilute it to the mark; shake well.

8.5 Preparation of series standard solutions

8.5.1 Sample dissolution method I

8.5.1.1 When the mass fraction of the element to be analyzed is within 0.0020% ~ 0.020%, pipette 0 mL, 0.20 mL, 0.50 mL, 1.00 mL, 5.00 mL of mixed standard solution III (5.15) into a 100 mL volumetric flask, which contains sodium hydroxide matrix solution II (5.9). Add 5 mL of aluminum matrix solution (5.7) and 20 mL of hydrochloric acid (5.4); use water to dilute it to the mark; mix well.

8.5.1.2 When the mass fraction of the element to be measured is > 0.020% ~ 0.20%, pipette 0 mL, 1.00 mL, 2.00 mL, 5.00 mL, 10.00 mL of mixed standard solution II (5.14) into a series of 100 mL volumetric flasks containing sodium hydroxide matrix solution II (5.9). Add 5 mL of aluminum matrix solution (5.7) and 20 mL of hydrochloric acid (5.4) to each flask. Use water to dilute it to the mark; mix thoroughly.

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