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Methods for chemical analysis of bauxite - Part 29:

Determination of available alumina and reactive silica

铝土矿石化学分析方法 第 29 部分：有效氧化铝和可反应硅的测定

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Methods for chemical analysis of bauxite - Part 29:

Determination of available alumina and reactive silica

1 Scope

This document describes the method for the determination of available alumina and reactive silicon in bauxite ore.

This document is applicable to the determination of available alumina and reactive silicon in bauxite ore. The determination range for available alumina is 30.00% ~ 60.00%; the determination range for reactive silicon is 0.10% ~ 10.00%.

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

Bauxite ore is dissolved in sodium hydroxide solution at a specific temperature and time. A certain volume of the solution is aliquoted and determined by the EDTA volumetric method. The alumina content obtained is the effective alumina content in the bauxite ore. The residue is treated with acid; a certain volume of the treated solution is aliquoted and mixed with the previous solution. It is determined by silicate molybdenum blue spectrophotometry. The silica content obtained is the reactive silicon content in the bauxite ore.

5 Reagents

Unless otherwise specified, only reagents of confirmed analytical grade and grade II water meeting the requirements of GB/T 6682 are used in the analysis.

5.1 Hydrochloric acid (1 + 11).

5.2 Hydrochloric acid (1 + 5).

5.3 Aqueous ammonia (1 + 5).

5.4 Sodium hydroxide solution (50 g/L, calculated as Na₂O): Weigh 64.516 g of sodium hydroxide into a 2000 mL beaker; dissolve in water; cool to room temperature; use water to dilute it to 1000 mL; mix thoroughly; transfer to a polyethylene bottle.

5.5 Acetic acid-sodium acetate buffer solution (pH approximately 5.7): Weigh 121 g of anhydrous sodium acetate into a 2000 mL beaker; dissolve in 700 mL of water; then add 9.6 mL of glacial acetic acid ($\rho = 1.05$ g/mL); use water to dilute to 1000 mL.

5.6 Ammonium molybdate solution (100 g/L).

5.7 Sulfuric acid-oxalic acid-ferrous ammonium sulfate mixed solution: Weigh 10 g of ferrous ammonium sulfate into a 2000 mL beaker; add 400 mL of water to dissolve it. Then add 60 mL of sulfuric acid (1 + 1). Weigh 10 g of oxalic acid into a 2000 mL beaker; add 300 mL of water to dissolve it. After cooling, mix with the previous solution and use water to dilute to 1000 mL.

5.8 Zinc standard titration solution (0.02942 mol/L): Weigh 2.3945 g of standard zinc oxide (calcined in a high-temperature furnace at $800\text{ }^{\circ}\text{C} \pm 50\text{ }^{\circ}\text{C}$ to constant weight); moisten with a small amount of water; dissolve in 30 mL of hydrochloric acid (1 + 1); cool it; transfer to a 1000 mL volumetric flask; use water to dilute to the mark; mix thoroughly.

5.9 Prepare and calibrate the disodium ethylenediaminetetraacetic acid (EDTA) standard titrant solution ($c \approx 0.05$ mol/L) as follows:

- a) Weigh 19.8 g of disodium ethylenediaminetetraacetic acid into a 500 mL beaker. Dissolve the solution in 200 mL of water. Transfer the solution to a 1000 mL volumetric flask; use water to dilute to the mark; mix thoroughly.
- b) Pipette three 15.00 mL portions of the zinc standard titrant solution (5.8) into separate 250 mL conical beakers. Add 80 mL of water and 2 drops of xylenol orange indicator (5.12). Adjust the solution to an orange color with aqueous ammonia (5.3). Add 15 mL of buffer solution (5.5) and 1 drop of bromocresol green indicator (5.11). Titrate with the EDTA standard titrant solution. The endpoint is reached when the solution changes from purple to green. Perform a

blank test at the same time.

5.10 Silicon dioxide standard solution: Weigh 0.1000 g of silicon dioxide ($w_{\text{SiO}_2} \geq 99.99\%$), that has been pre-calcined at 1000 °C for 2 h and cooled to room temperature, into a platinum crucible; add 3 g of anhydrous sodium carbonate; mix thoroughly. Cover the crucible with 1 g of anhydrous sodium carbonate; place it in a 950 °C furnace to melt for 12 minutes. Remove and cool it; place in a plastic cup. Leach with 200 mL of boiling water. Rinse the crucible and lid. Cool to room temperature; transfer to a 1000 mL volumetric flask; use water to dilute to the mark; mix it; store in a plastic bottle. 1 mL of this solution contains 0.1 mg of silica.

6 Instruments

6.1 Dissolution apparatus: Microwave digester with 100 mL polytetrafluoroethylene digestion tubes.

Note: Other dissolution apparatus may be selected based on sample characteristics.

6.2 Spectrophotometer.

7 Sample

Grind the sample to pass through a standard sieve with a pore size of 75 μm ; dry it in

an oven at $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 2 h; place it in a desiccator; cool it to room temperature for later use.

8 Test steps

8.1 Test material

Weigh 1.00 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.

8.4 Determination

8.4.1 Place the sample (8.1) in a digestion tube; add 20.0 mL of sodium hydroxide solution (5.4); shake well; transfer to the dissolution apparatus (6.1); keep it at $143\text{ }^{\circ}\text{C}$ for 30 min.

Note: The holding temperature and time can be adjusted according to different samples.

8.4.2 Transfer the contents of the dissolution apparatus (8.4.1) to a 250 mL volumetric flask with water; use water to dilute to the mark; mix thoroughly. Filter the test solution through quantitative filter paper; transfer the entire contents of the volumetric flask into a funnel; set aside the residue; place the filtrate in a 250 mL beaker.

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