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Methods for chemical analysis of bauxite - Part 16: Determination of phosphorus pentoxide content - Molybdenum blue spectrophotometric method

铝土矿石化学分析方法 第 16 部分：

五氧化二磷含量的测定 钼蓝光度法

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Methods for chemical analysis of bauxite - Part 16: Determination of phosphorus pentoxide content - Molybdenum blue spectrophotometric method

1 Scope

This Part specifies the method for the determination of phosphorus pentoxide content in bauxite ores.

This Part applies to the determination of phosphorus pentoxide content in bauxite ore. The determination range is 0.01% ~ 5.00%.

2 Principles of the method

The decomposition of the analytical specimen can be performed, by any one of the following methods:

a) Treatment with a mixed acid of hydrochloric acid, nitric acid, sulfuric acid.

This method is suitable for trihydrate bauxite ore or monohydrate bauxite ore. The residue after dissolving the specimen, after volatilizing silica, is required to be less than 1% of the specimen amount.

b) It is sintered with sodium peroxide; melted for a short period of time; the melt is dissolved with sulfuric acid.

This method is suitable for diaspore. The residue, after dissolution of the specimen, is required to be greater than 1% of the specimen amount.

Silica is dehydrated, salts dissolved, filtered and residue calcined; silica is evaporated with hydrofluoric acid and sulfuric acid; it is melted with sodium carbonate and sodium tetraborate; it is dissolved with sulfuric acid AND incorporated into the main solution. The molybdate is added, to form a phosphomolybdate complex with phosphate; the molybdenum blue is reduced by ascorbic acid; the absorbance of the solution is measured, at about 710 nm in a spectrophotometer.

of this solution, into a 250 mL volumetric flask. Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 20 μg of phosphorus pentoxide.

4 Instruments

Spectrophotometer.

5 Specimen

Use a mortar to grind the specimen, to make it pass through a 150 μm sieve. Place the ground specimen, at 110 °C ± 5 °C, to dry it for 2 hours. Place it in a desiccator. Cool it to room temperature for later use.

6 Analytical procedures

6.1 Sample

Weigh about 1.0 g of specimen (5), accurate to 0.0001 g.

6.2 Number of measurements

Make two measurements independently. Take the average value.

6.3 Blank test

Carry out a blank test, together with the sample.

6.4 Measurement

6.4.1 Sample decomposition

6.4.1.1 Acid decomposition

Put the sample (6.1) into a 400 mL beaker. Use water to wet it. Add 60 mL of mixed acid (3.7). Cover a watch glass. Heat it to decompose it, at 80 °C (when the Fe_2O_3 content in the specimen is greater than 15%, the heating time shall be extended). When the brown smoke disappears, wash the watch glass and beaker walls. Remove the watch glass. Heat and evaporate it, until the smoke of sulfuric acid is emitted. Then cover the watch glass. Heat the solution on an electric heating plate, to keep the temperature of the solution at 210 °C ± 10 °C. Heat and reflux vigorously for 60 min. In another beaker, which is filled with sulfuric acid (ρ_{20} 1.84 g/mL), insert a thermometer AND immerse it in sulfuric acid for 10 mm, to measure the temperature.

6.4.1.2 Decomposition by alkali sintering

Put the sample (6.1) into a dry crucible. Add 10 g of sodium peroxide (3.1). After mixing well, put it into a muffle furnace. Sinter it at 490 °C for 45 min. Take it out. Rotate the crucible, to make the melt evenly adhere to the inner wall of the crucible. Cool it down. Place the crucible side in a 400 mL beaker. Cover a watch glass. Carefully add 140 mL of sulfuric acid solution (3.4), from rear side of the crucible. Then add 20 mL of sulfuric acid solution (3.3). Heat to leach out the melt in the crucible. When the specimen, in the crucible, is completely leached out, take out the crucible. Wash the watch glass and the beaker wall. Remove the watch glass. Heat to evaporate the solution, until the sulfuric acid smoke is emitted. Then cover the watch glass. Heat it on the electric hot plate, to keep the temperature of the solution at 210 °C ± 10 °C. Carry out strong reflux for 60 min.

6.4.2 Dissolution and filtration

Cool the solution of 6.4.1.1 or 6.4.1.2 to room temperature. Carefully add 130 mL of water. Heat to 80 °C ~ 90 °C, while stirring. Keep the temperature, for at least 40 min, to completely dissolve the salts. Use medium-speed filter paper to filter it, while it is still hot. Collect the filtrate in a 250 mL volumetric flask. Use water to wash the beaker. Use a glass rod with a rubber tip to scrub the beaker. Quantitatively transfer the residue onto the filter paper. Wash the filter paper and residue, by 5 mL ~ 10 mL of hot water, each time. Retain the filtrate.

6.4.3 Treatment of residues

Transfer the filter paper and residue of 6.5.2, into a crucible, which was pre-burned to a constant weight. Slowly ash the filter paper. Then gradually heat it to 650 °C, in a muffle furnace, for 30 min. Take out the platinum crucible. After cooling, add a few drops of water to wet the residue. Add 5 drops of sulfuric acid solution (3.3). Add 10 mL of hydrofluoric acid (3.5). Carefully evaporate to dryness, in a fume hood, to drive out the dioxide silicon and sulfuric acid. Weigh the crucible after cooling. Measure the mass of the residue.

Add 0.7 g ± 0.1 g of sodium carbonate and sodium tetraborate mixed flux (3.2). Melt it in muffle furnace above 1000 °C, for 5 min. Rotate the melt. Melt for 1 min ~ 2 min. After taking out the crucible and cooling, add 10 mL of sulfuric acid solution (3.4). Heat at low temperature, until the melt dissolves. Incorporate the solution into the remaining filtrate (6.4.2). Use water to rinse the crucible clean. Cool it to room temperature. Use water to dilute it to the mark. Mix well. This solution is the test solution.

6.4.4 Treatment of the test solution

When the P_2O_5 content is within 0.01% ~ 1.0%, directly use a pipette to quantify the test solution. When the P_2O_5 content is within 1% ~ 5%, pipette 10 mL of the test solution into a 100 mL volumetric flask. Use water to dilute it to the mark.

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