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Method for determination of silicon dioxide content in arenaceous quartz

石英砂中二氧化硅含量测定方法

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Method for determination of silicon dioxide content in arenaceous quartz

1 Scope

This Standard specifies the principle, instruments, sample requirements, test procedures and test results of the determination of silicon dioxide content in arenaceous quartz.

This Standard applies to the determination of silicon dioxide content in arenaceous quartz for carbon production.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Principle

The sample is decomposed with hydrofluoric acid and nitric acid. Sulfuric acid emits smoke to drive off silicon and fluorine. The sample is burned to constant weight at high temperature. The silicon dioxide content is calculated according to the difference between the masses of the sample before and after the hydrofluoric acid treatment. The chemical reaction is as follows:

$$SiO_2 + 4HF \longrightarrow SiF_4 \uparrow + 2H_2O$$

4 Instruments

- **4.1** Balance, with a division of 0.1 mg.
- **4.2** Platinum crucible, 30 mL.

- **4.3** Automatic temperature control drying oven.
- **4.4** Muffle furnace: The maximum operating temperature is not less than 1000 °C, and it can automatically control the temperature.

5 Reagents

- **5.1** Nitric acid, ρ : 1.40 g/cm³, analytically pure.
- **5.2** Sulfuric acid (1:3), analytically pure.
- **5.3** 40 % hydrofluoric acid, analytically pure.

6 Preparation of samples

PUT the sample in an oven at (120 °C \pm 5 °C) for 2 h to dry; TAKE it out to cool. USE the quarter method to divide 50 g \sim 60 g of the sample. USE a grinder to pulverize it to a particle size of less than 150 μ m. PACK the prepared sample into the sample bag and put it in the drying oven for later use.

7 Test procedures

7.1 Sample mass

WEIGH 0.5 g of sample, accurate to ±0.001 g.

7.2 Determination

- **7.2.1** PLACE the sample in a platinum crucible (4.2) that has been made to constant weight in advance; BAKE it at $120^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 30 min; TAKE it out; COOL it to room temperature in a desiccator; WEIGH it as m_1 .
- **7.2.2** PLACE the crucible in a fume hood; slowly ADD 5 mL of nitric acid (5.1) along the wall of the crucible; ADD 3 to 4 drops (about 1 mL) of sulfuric acid (5.2); gradually ADD 5 mL ~ 10 mL of hydrofluoric acid (5.3); PUT the crucible on a low-temperature electric furnace; HEAT to completely decompose the sample.
- **7.2.3** If the sample is not decomposed, continue to add 5 mL \sim 10 mL of hydrofluoric acid (5.3) and 3 mL of nitric acid (5.1) until the test solution is completely clear.
- **7.2.4** Continue to evaporate until the sulfuric acid smoke is exhausted and then take it down.

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