YS

NONFERROUS METAL INDUSTRY STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

ICS 71.100.10
H 21

YS/T 273.12-2006

Chemical analysis methods and physical properties of cryolite - Part 12: The atomic absorption spectrophotometric method for the determination of calcium oxide content

冰晶石化学分析方法和物理性能测定方法

第 12 部分：火焰原子吸收光谱法测定氧化钙含量

Issued on: May 25, 2006 **Implemented on: December 1, 2006**

Issued by: National Development and Reform Commission of PRC

Table of Contents

Chemical analysis methods and physical properties of cryolite – Part 12: The atomic absorption spectrophotometric method for the determination of calcium oxide content

1 Scope

This Part specifies the method for determination of calcium oxide content in cryolite.

This Part applies to the determination of calcium oxide in cryolite. Determination range (mass fraction): ≤1.25%.

2 Method summary

Use perchloric acid to de-fluorinate the test portion; heat until the smoke of perchloric acid has evaporated, then use hydrochloric acid and water to dissolve it. In the presence of lanthanum chloride, measure the calcium content using an air-acetylene fuel-rich flame at the wavelength of 422.7 nm of the atomic absorption spectrometer.

3 Reagents

3.1 Perchloric acid (ρ1.67 g/mL).

3.2 Hydrochloric acid (1+1): guaranteed reagent.

3.3 Sodium solution (7.5 mg/mL): Transfer 9.5 g of high-purity sodium chloride and use water to dissolve it in a 500 mL volumetric flask.

3.4 Sodium solution (1.5 mg/mL): Transfer 20 mL of sodium solution (3.3) into a 100 mL volumetric flask; use water to dilute to the mark; mix well.

3.5 Lanthanum chloride solution (200 g/L): Take 100 g of $LaCl_3 \cdot nH_2O$; use water to dilute it in a 500 mL volumetric flask.

3.6 Aluminum solution (4 mg/mL): Weigh 4.00 g of pickled high-purity aluminum; place it in a 500 mL beaker; cover the watch plate; add a total of 120 mL of hydrochloric acid (3.2) in batches; add a drop of mercury to help dissolution; after the violent reaction stops, slowly heat until it is completely dissolved; cool; transfer the solution to a 1 000

mL volumetric flask; use water to dilute to the mark; mix well.

3.7 Aluminum solution (0.8 mg/mL): Pipette 20 mL of aluminum solution (3.6) into a 100 mL volumetric flask; use water to dilute to the mark; mix well.

3.8 Calcium standard stock solution: Weigh 1.248 6 g of calcium carbonate that has been previously dried at 105 °C; place it in a 250 mL beaker; cover it with a watch plate; add 50 mL of water; then add 10 mL of hydrochloric acid (3.2); heat slightly; after the reaction is complete, cool; transfer to a 500 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 1.00 mg of calcium.

3.9 Calcium standard solution: Pipette 25.00 mL of calcium standard stock solution (3.8) into a 500 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 0.05 mg of calcium.

4 Instruments and apparatuses

Atomic absorption spectrometer, hollow cathode lamp with calcium.

Under the best working conditions of the instrument, anything that can achieve the following indicators can be used:

- Characteristic concentration: In a solution consistent with the matrix of the measured sample, the characteristic concentration of calcium shall not be greater than 0.24 ug/mL.

- Precision: Use the standard solution of the highest concentration to measure the absorbance for 10 times, where the standard deviation shall not exceed 1.0% of the average absorbance. Use the standard solution of the lowest concentration (not the “zero” concentration standard solution) to measure the absorbance for 10 times, where the standard deviation shall not exceed 0.5% of the average absorbance of the standard solution of the highest concentration.

- Linearity of working curve: Divide the working curve into five segments according to the concentration, and the ratio of the absorbance difference of the highest segment to the absorbance difference of the lowest segment shall not be less than 0.7.

5 Test sample

The test sample shall comply with the requirements of 3.3 in YS/T 273.13.

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------