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# NONFERROUS METAL INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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YS/T 273.5-2006

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# Chemical analysis methods and physical properties of cryolite - Part 5: Determination of sodium by flame atomic absorption spectrometric method

冰晶石化学分析方法和物理性能测定方法 第 5 部分: 火焰原子吸收光谱法测定钠含量

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#### Foreword

YS/T 273 "Chemical analysis methods and physical properties of cryolite" is divided into 13 parts:

- -- Part 1: Determination of moisture content Gravimetric method;
- -- Part 2: Determination of ignition loss;
- -- Part 3: Determination of fluoride content;
- -- Part 4: Determination of aluminum content EDTA titration method;
- -- Part 5: Determination of sodium by flame atomic absorption spectrometric method:
- -- Part 6: Determination of silica content by the molybdenum blue photometric;
- -- Part 7: Determination of iron content by orthophenantholine photometric method;
- -- Part 8: Determination of sulfate content Barium sulfate gravimetric method;
- -- Part 9: Determination of phosphorus pentoxide content Molybdenum blue photometric method;
- -- Part 10: Determination of alumina content by gravimetric method;
- -- Part 11: Determination of sulphur content by X-ray fluorescence spectrometric method;
- -- Part 12: The atomic absorption spectrophotometric method for the determination of calcium oxide content:
- -- Part 13: Preparation and storage of test samples.

This Part is Part 5.

This part is a revision of YS/T 273.5-1994. In addition to editorial modification, two chapters on Precision AND Quality Assurance and Control have been added. Other changes include:

- -- The measuring wavelength of the instrument is changed from the most sensitive 589.0 nm to the hypo-sensitive 589.6 nm;
- -- The length of the working curve is shortened, and the concentration of the highest concentration standard solution in the working curve is reduced from 10.0  $\mu$ g/mL to 5.00  $\mu$ g/mL;

# Chemical analysis methods and physical properties of cryolite - Part 5: Determination of sodium by flame atomic absorption spectrometric method

#### 1 Scope

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

This Part applies to the determination of sodium content in cryolite. Determination range: 20%~35%.

### 2 Method summary

After the test portion is dissolved with sulfuric acid, it is heated to remove fluorine, and the precipitate is dissolved with hydrochloric acid and water; the sodium content of the test solution is determined at the wavelength of 589.0 nm or 589.6 nm of an atomic absorption spectrometer by the use of an air-acetylene fuel-lean flame.

# 3 Reagents

- **3.1** Sulfuric acid ( $\rho$  1.84 g/mL).
- **3.2** Hydrochloric acid ( $\rho$  1.19 g/mL).
- **3.3** Sodium standard solution: Weigh 3.089 g of anhydrous sodium sulfate that has been dried at 120 °C and cooled in a desiccator, place it in a 250 mL beaker, and add 100 mL of water to dissolve; then, add 10 mL of hydrochloric acid (3.2), transfer the solution to a 1000 mL volumetric flask, dilute to volume with water, mix well, and transfer it to a polyethylene bottle. 1 mL of this solution contains 1.000 mg of sodium.

# 4 Apparatus

- **4.1** Platinum dish: The diameter is 80 mm, and the height is 35 mm.
- **4.2** Atomic absorption spectrometer: It is equipped with a sodium hollow cathode lamp.

Any instrument that can meet the following indicators under the best working conditions can be used.

- -- Characteristic concentration: In a solution consistent with the matrix of the measured sample, the characteristic concentration of sodium shall not be greater than 0.07 ug/mL.
- -- Precision: Use the standard solution with the highest concentration to measure the absorbance 10 times, and the standard deviation shall not exceed 1.0% of the average absorbance; use the standard solution with the lowest concentration (not the "zero" concentration standard solution) to measure the absorbance 10 times, and the standard deviation shall not exceed 0.5% of the average absorbance of the highest concentration standard solution.
- -- Working curve linearity: Divide the working curve into 5 equal segments according to concentration, and the ratio of the absorbance difference in the highest segment to the absorbance difference in the lowest segment is not less than 0.85.

#### 5 Sample

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

#### 6 Analysis procedure

#### 6.1 Test portion

Weigh 0.5 g of test sample (5), accurate to 0.0001 g.

#### 6.2 Number of determinations

Two determinations shall be carried out independently and the average value shall be taken.

#### 6.3 Blank test

Do a blank test along with the sample test.

#### 6.4 Determination

**6.4.1** Place the test portion (6.1) in the platinum dish (4.1), add 5 mL of sulfuric acid (3.1), and slowly dissolve it in a sand bath (15 min~20 min) to remove fluorine; then, heat it to evaporate until the thick smoke emits; remove the test portion and cool it to room temperature; add 3 mL of hydrochloric acid (3.2) and 30 mL of water, and heat in a low-temperature sand bath until all salts are dissolved. After cooling, transfer the solution to a 500 mL volumetric flask, dilute to volume with water, and mix well.

**6.4.2** According to Table 1, take the test solution (6.4.1), place it in a 500 mL volumetric

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