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Methods for Chemical Analysis and Physical Properties Test of Cryolite – Part 16: Determination of Lithium Content – Flame Atomic Absorption Spectrometric Method

冰晶石化学分析方法和物理性能测定方法

第 16 部分：锂含量的测定 火焰原子吸收光谱法

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Foreword

YS/T 273 *Chemical analysis methods and physical properties of cryolite* consists of the following 17 parts:

--- Part 1: Determination of moisture content by gravimetric method;

--- Part 2: Determination of ignition loss;

--- Part 3: Determination of fluoride content;

--- Part 4: Determination of aluminum by the EDTA volumetric 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 sulphate content by barium sulphate gravimetric method;

--- Part 9: Determination of phosphorous pentoxide content – Molybdenum blue photometric method;

--- Part 10: Determination of alumina content by gravity 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;

--- Part 14: X-ray fluorescence spectrometric method for the determination of elements content;

--- Part 15: X-ray fluorescence spectrometric method for the determination of elements content using pressed powder tablets;

--- Part 16: Determination of lithium content – Flame atomic absorption spectrometric method;

--- Part 17: Determination of elements content – Inductively coupled plasma atomic emission spectrometric method.

This Part is Part 16 of YS/T 273.

Methods for Chemical Analysis and Physical Properties Test of Cryolite – Part 16: Determination of Lithium Content – Flame Atomic Absorption Spectrometric Method

1 Scope

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

This Part applies to the determination of lithium content in cryolite. Measuring range: 0.010%~1.50%.

2 Method Summary

Use perchloric acid to remove all fluorine from the test material; and then dissolve it with hydrochloric acid and water. The test solution is measured at the wavelength of 670.8 nm of the atomic absorption spectrometer using an air-acetyl block flame to measure the absorbance of lithium; and the lithium content is calculated.

3 Reagents

Unless otherwise stated, only reagents and laboratory Class-III water determined to be of analytically pure are used in the analysis.

3.1 Cryolite, spectrally pure.

3.2 Perchloric acid (ρ≈1.67 g/mL).

3.3 Hydrochloric acid (1+1).

3.4 Lithium standard storage solution: Weigh 5.3228 g of lithium carbonate (spectrally pure) into a 500 mL beaker; cover with a watch plate; slowly add 125 mL of nitric acid (1+9); heat until completely dissolved; boil for a few minutes; and cool to room temperature. Transfer the solution into a 1000mL volumetric flask and dilute with water to the mark; mix well. 1mL of this solution contains 1.0mg of lithium.

3.5 Lithium standard solution: Pipette 10.00 mL of lithium standard storage solution (3.4) into a 100 mL volumetric flask; dilute to volume with water; and mix well. 1 mL of this solution

contains 100μg of lithium.

4 Apparatus

Atomic absorption spectrometer with lithium hollow cathode lamp.

Under the best working conditions of the instrument, any instrument that can meet the following indicators can be used:

--- Sensitivity: In a solution consistent with the matrix of the measurement test material solution; the characteristic concentration of lithium shall be no greater than 0.028μg/mL;

--- Precision: Use the standard solution with highest concentration to measure the absorbance for the most 10 times; and the standard deviation shall not exceed 1.0% of the average absorbance.

Use the standard solution with lowest concentration (not the solution with zero concentration) to measure the absorbance 10 times; and its standard deviation shall not exceed 0.5% of the average absorbance of the standard solution with highest concentration;

--- Standard curve linearity: In standard solutions with equal-difference concentration, the ratio of the absorbance difference on the highest segment to the absorbance difference on the lowest segment shall be no less than 0.7.

5 Specimen

The specimen is ground and mixed, and passed through a 75μm standard sieve; baked in an oven at 110°C ± 5°C for 2 h; and cooled to room temperature in a desiccator.

6 Test Procedures

6.1 Test material

Weigh 0.50g of specimen (5), accurate to 0.0001g.

6.2 Parallel test

Do two experiments in parallel and take the average.

6.3 Blank test

Weigh 0.50 g of cryolite (3.1) and conduct a blank test together with the test material.

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