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**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 273.15-2012

**Chemical analysis methods and physical properties of cryolite. Part 15:
X-ray fluorescence spectrometric method for the determination of
elements content using pressed powder tablets
(English Translation)**

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1 Scope

This Part of YS/T 273 specifies the X-ray fluorescence spectrometric method for the determination of contents of fluorine, aluminum, sodium, silicon, iron, sulfur, phosphorus and calcium (respectively represented by F, Al, Na, SiO₂, Fe₂O₃, SO₄²⁻, P₂O₅, CaO) and other elements in cryolite using pressed powder tables.

This Part is applicable to the determination of fluorine, aluminum, sodium, silicon, iron, sulfur, phosphorus, calcium and other elements in cryolite. The measurement range is shown in Table

1.

2 Normative References

The following documents are essential to the application of this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this

Document.

YS/T 273.13-2006 Chemical analysis methods and physical properties of cryolite - Part

13: Preparation and storage of test samples

3 Principle of Method

The test material and dispersant are mixed and ground in a certain proportion; and then pressed to form a sample tablet. Measurements are performed by an X-ray fluorescence spectrometer.

Calculate the content of each element in the specimen based on the calibration curve of the intensity of each element in the standard sample tablet.

4 Reagents

4.1

Acetone

Analytically pure.

4.2

Absolute ethanol

Analytically pure.

4.3

Boric acid

Analytically pure.

5 Instruments

5.1

Wavelength dispersive X-ray fluorescence spectrometer.

5.2

Vibrating grinding mill and tungsten carbide grinding disc

The mortar should be able to grind a specimen of about 30g.

5.3

Tablet machine and mold

The pressing thickness shall be at least 4mm; the pressure shall be about 35t; and the inner diameter of the mold shall match the X-fluorometer sample cup.

6 Specimens

The specimen shall meet the requirements of 3.3 in YS/T 273.13-2006.

7.1 Number of measurements

The same specimen shall be measured twice independently and the average value shall be taken.

7.2 Preparation of specimen tablets

7.2.1

Mixing and grinding

Weigh about 10g of dry specimen and put it into a tungsten carbide grinding disc; add 10 drops of acetone (4.1) or absolute ethanol (4.2) to prevent the specimen from agglomerating; and grind it to less than 45 μ m with a vibrating mill.

7.2.2

Press tablet

Pour an appropriate amount of specimen (7.2.1) into the mold; edge it with boric acid; use a tablet machine to pressurize it to about 35t; and keep it for 30 s. After taking out the sample tablet and trim it, use the ear wash ball to blow off the attached powder sample;

and then measure it on an X-fluorometer. When measuring, only take the edge of the sample tablet to avoid contamination of the measurement surface.

NOTE 1: In order to eliminate particle effects to the greatest extent during tableting analysis, the sample preparation conditions shall be consistent with those used when establishing the calibration curve.

NOTE 2: The extruded sample tablet shall be smooth and strong; no powder shall fall off after trimming;

and boric acid shall not be mixed into the measurement surface, otherwise, it cannot be tested.

7.3.1 Background correction: For constant elements, choose one or two backgrounds to

measure.

7.3.2 Instrument drift correction: Calibrate instrument drift by measuring and monitoring

samples.

7.3.3 Drawing of calibration curve

Preparation of calibration sample tablets: Select the cryolite standard sample as the calibration sample to draw the calibration curve. Each element shall have a standard series

with a sufficient content range and a certain gradient. The preparation process shall be carried out according to

7.2.

7.4 Determination

7.4.1

Preheat the X-ray fluorescence instrument to make it stable; and adjust the tube voltage and tube current according to the X-ray tube model. Select the operating parameters according to the model of the X-ray fluorescence spectrometer.

7.4.2

Measure the monitoring sample

Set the name of the monitoring sample and measure the

X-ray intensity of the analyzed elements in the monitoring sample. The reference intensity of the analyzed elements in the monitoring sample shall be measured at the same start-up as the standard sample to ensure the effectiveness of drift correction.

7.4.3 Measure the unknown sample: Start the quantitative analysis program; measure the

monitoring sample; and perform instrument drift correction. Measure the standard samples prepared in the same batch as the unknown samples. Enter the name of the unknown sample;

measure the unknown sample; and the analysis results shall meet the repeatability requirements specified in Table 2.

8 Analysis Results

Based on the X-ray measurement intensity of the unknown sample, the results are calculated by computer software.

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