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

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**Methods for chemical analysis and physical properties test of
cryolite - Part 9: Determination of phosphorous pentoxide
content - Molybdenum blue photometric method**

冰晶石化学分析方法和物理性能测定方法 第9部分：五氧化二磷含量的测定 钼蓝
分光光度法

(English Translation)

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Table of Contents

Foreword...	3
1 Scope...	5
2 Method Summary...	5
3 Reagents...	5
4 Apparatus...	6
5 Specimen...	6
6 Test Procedures...	6
7 Calculation of Test Results...	8
8 Precision...	8
9 Quality Assurance and Control...	9
10 Test Report...	9

1 Scope

This Part specifies the method for determination of phosphorous pentoxide in cryolite.

This Part applies to the determination of phosphorous pentoxide in cryolite. The determination range: 0.0030%~0.50%.

2 Method Summary

The test material is melted by a mixed solvent of anhydrous sodium carbonate and boric acid, and acidified by sulfuric acid. Use sodium hydroxide to adjust the acidity. When the pH value is ≤ 0.3 , add molybdic acid to form phosphomolybdenum heteropoly acid with phosphorus.

After being reduced by ascorbic acid to phosphomolybdenum blue, measure its absorbance at a wavelength of 815nm with a spectrophotometer. The content of phosphorus pentoxide is calculated using the standard curve method.

3 Reagents

Unless otherwise stated, use only reagents confirmed to be of analytically pure and distilled water or water of equivalent purity.

3.1

Sodium carbonate anhydrous.

3.2

Boric acid.

3.3

Sulfuric acid (1+1).

3.4 Acidic solution of ammonium molybdate (25 g/L): Weigh 25g of ammonium molybdate

tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ and dissolve it in 200 mL of hot water. After cooling, add about 434mL of sulfuric acid (3.3); dilute to 1L with water; and mix well. This solution is stored in polyethylene bottles.

3.5 Ascorbic acid solution (20 g/L).

3.6 Sodium hydroxide solution (200 g/L).

3.7 Phosphorus pentoxide standard storage solution: Weigh 0.1944g of sodium dihydrogen

phosphate monohydrate $[\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}]$, dissolve it in water and transfer it to a 1L volumetric flask; dilute to the mark and mix well. 1 mL of this solution contains 100 μg of phosphorus pentoxide.

3.8 Phosphorus pentoxide standard solution: Pipette 50.00mL of phosphorus pentoxide

standard storage solution (3.7) into a 500 mL volumetric flask; dilute to the mark with water; and mix well. 1 mL of this solution contains 10 μg of phosphorus pentoxide.

3.9 Phenolphthalein ethanol solution (10 g/L).

4 Apparatus

4.1

Platinum dish

Diameter 70 mm, height 35 mm, with platinum lid.

4.2

High temperature furnace

Capable of controlling the temperature at $850^\circ\text{C} \pm 20^\circ\text{C}$.

4.3

Water bath

Capable of controlling the temperature at $80^\circ\text{C} \pm 2^\circ\text{C}$.

4.4

Spectrophotometer.

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.

6.1 Test material

Weigh the specimen (5) according to Table 1, accurate to 0.0001 g.

6.2 Parallel test

Do two experiments in parallel and take the average.

6.3 Blank test

Do a blank test along with the test material.

6.4 Determination

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