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

YS/T 509.6-2008

**Methods for chemical analysis of spodumene and lepidolite
concentrates. Determination of phosphorus pentoxide content.
The molybdenum blue photometric method
(English Translation)**

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Foreword

YS/T 509 Methods for Chemical Analysis of Spodumene and Lepidolite Concentrates

is divided into 11 parts:

---Part 1: Determination of Lithium Oxide Sodium Oxide and Potassium Oxide

Contents - Flame Atomic Absorption Spectrometric Method;

---Part 2: Determination of Rubidium Oxide and Caesium Oxide Contents - Flame

Atomic Absorption Spectrometric Method;

---Part 3: Determination of Silicon Dioxide Content - Gravimetric - Molybdenum

Blue Photometric Method;

---Part 4: Determination of Aluminum Oxide Content - EDTA Compleximetric

Method;

---Part 5: Determination of Ferric Oxide Content - O-phenanthroline Photometric

Method and EDTA Compleximetric Method;

---Part 6: Determination of Phosphorus Pentoxide Content - The Molybdenum

Blue Photometric Method;

---Part 7: Determination of Beryllium Oxide Content - Chromazurol S and

Cetyltrimethyl Ammonium Bromide Spectrophotometric Method;

---Part 8: Determination of Calcium Oxide and Magnesium Oxide Content - Flame Atomic Absorption Spectrometric Method;

---Part 9: Determination of Calcium Oxide and Magnesium Oxide Content - Flame Atomic Absorption Spectrometric Method;

---Part 10: Determination of Manganous Oxide Content - Persulfate Oxidation Photometric Method;

---Part 11: Determination of Loss on Ignition - Gravimetric Method.

This Part is Part 6.

This Part serves as a replacement of YS/T 509.7-2006 Methods for Chemical Analysis of Spodumene and Lepidolite Concentrates - The Molybdenum Blue Photometric Method for the Determination of Phosphorus Pentoxide Content (the former GB/T 3885.7-1983).

In comparison with YS/T 509.7-2006, this Part mainly has the following changes:

---"place it in a beaker" in 3.7 is modified into "place it in a 250 mL beaker";

Methods for Chemical Analysis of Spodumene and
Lepidolite Concentrates - Determination of
Phosphorus Pentoxide Content - The Molybdenum
Blue Photometric Method

1 Scope

This Part specifies the method of determining phosphorus pentoxide content in spodumene and lepidolite concentrates.

This Part is applicable to the determination of phosphorus pentoxide content in spodumene and lepidolite concentrates. The range of determination is 0.01% ~ 1.00%.

2 Method Summary

Use hydrofluoric acid and sulfuric acid to decompose the test piece; sulfuric acid emits smoke, which drives off silicon and fluorine, and is leached with water. Add ammonium molybdate to 0.5 mol/L ~ 0.65 mol/L H₂SO₄ medium to make phosphorus generate phosphomolybdenum heteropoly acid. Use ascorbic acid as a reducing agent; heat up

in boiling water to reduce phosphomolybdenum heteropoly acid to phosphorus molybdenum blue. At a wavelength of 700 nm of the spectrophotometer, measure its absorbance.

3 Reagents

Unless it is otherwise specified, only reagents that are confirmed to be analytically pure, and distilled water or water of equivalent purity are used in the analysis.

3.7 Phosphorus pentoxide standard stock solution: weigh-take 1.9175 g of potassium

Conduct a blank test on the accompanying sample.

6.4.1 Place the sample (6.1) in a platinum dish; add 10 mL of hydrofluoric acid (3.4)

and 5 mL of sulfuric acid (3.2). Place it on a sand bath and heat it up to decompose it; steam it, until it emits white smoke of sulfur trioxide for 3 min ~ 5 min. Make the remaining volume of the solution about 2 mL, then, remove it and cool it down.

6.4.2 Use water to wash the wall of the dish. Add 20 mL of water; heat it up on a sand

bath. When all salts are dissolved, remove it; cool it down to room temperature. Then, transfer it into a 100 mL volumetric flask; use water to dilute it to the scale; mix it well.

6.4.3 Transfer-take 25.00 mL of solution (6.4.2); place it in a 50 mL volumetric flask.

Add 5 mL of sulfuric acid (3.3) and 5 mL of ascorbic acid solution (3.6). Under constant shaking, add 4 mL of ammonium molybdate solution (3.5); mix it up. Use water to rinse the neck of the volumetric flask. Add water to make the volume of the color-developing solution about 45 mL; mix it well.

6.4.4 Place the volumetric flask in a boiling water bath to heat it up for 5 min. Then,

take it out. Use running water to cool it down to room temperature. Use water to dilute to the scale; mix it well.

6.4.5 Transfer part of the solution (6.4.4) into a 1 cm cuvette; take the blank of the accompanying sample as a reference. At a wavelength of 700 nm of the spectrophotometer, measure its absorbance. Through the working curve, find out the corresponding phosphorus pentoxide content.

6.5.1 Transfer-take 0 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL and 5.00 mL of phosphorus pentoxide standard solution (3.8). Respectively place them in a group of

50 mL volumetric flasks. Add 7 mL of sulfuric acid (3.3), 25 mL of water and 5 mL of

ascorbic acid solution (3.6). Under constant shaking, add 4 mL of ammonium molybdate solution (3.5); mix it well. Use water to rinse the neck of the volumetric flask. Add water to make the volume of the color-developing solution about 45 mL; proceed in accordance with the steps in 6.4.4.

6.5.2 Transfer part of the solution (6.5.1) into a 1 cm cuvette; take the reagent blank

as a reference. At a wavelength of 700 nm of the spectrophotometer, measure its absorbance. Use phosphorus pentoxide content as the x-coordinate; take its absorbance as the y-coordinate; draw a working curve.

7 Calculation of Analysis Result

The mass fraction w of phosphorus pentoxide, expressed in (%), shall be calculated in

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