

ICS 77.120.99

CCS H 64

YS

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

YS/T 509.3-2008

**Methods for Chemical Analysis of Spodumene and Lepidolite
Concentrates - Determination of silicon dioxide content -
Gravimetric-molybdenum blue photometric method
(English Translation)**

Issued on: March 12, 2008

Implemented on: September 1, 2008

Issued by: National Development and Reform Commission of China

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Foreword

YS/T 509 "Methods for chemical analysis of spodumene and lepidolite concentrates" consists of the following 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 complexometric method;
- Part 5: Determination of ferric oxide content-0-phenanthroline photometric method and EDTA complexometric 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 met;

- Part 8: Determination of calcium oxide and magnesium oxide content - Flame atomic absorption spectrometric method;
- Part 9: Determination of fluorine content - Ion selective electrode 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 3.

This Part replaces YS/T 509.3-2006 "Methods for chemical analysis of spodumene and lepidolite concentrates The gravimetric-molybdenum blue photometric method for the determination of silicon dioxide content" (previous edition is GB/T 3885.3-1983).

Compared with YS/T 509.3-2006, the main changes in this Part are as follows:

- modified "dry ashing" in 6.4.7 to "after ashing on the electric furnace";

Methods for chemical analysis of spodumene and lepidolite concentrates - Determination of silicon dioxide content - Gravimetric-molybdenum blue photometric method

1 Scope

This Part specifies the determination methods for silicon dioxide content in spodumene, lepidolite concentrates.

This Part is applicable to the determination of silicon dioxide content in spodumene, lepidolite concentrates. Determination range: >50%.

2 Method summary

The test material is decomposed with potassium hydroxide-sodium hydroxide mixed flux. Use water to leach. Use hydrochloric acid for acidification. Add polyethylene oxide as coagulant. Use gravimetric-molybdenum blue photometric method to determine the amount of silicon dioxide.

The lepidolite concentrate needs to add boric acid to eliminate the interference

of fluorine.

3 Reagents

Unless otherwise stated, only the confirmed analytically-pure reagents and distilled water or water of comparable purity are used in the analysis.

5.1 Spodumene concentrate shall pass through a sieve with an aperture of 0.074mm. Lepidolite concentrate shall pass through a sieve with an aperture of 0.105mm.

5.2 The specimen is pre-baked at 105°C~110°C for 2h. Place in a desiccator to cool to room temperature.

6.1 Test material

Weigh 0.50g of specimen, to the nearest of 0.0001g.

6.2 Number of determinations

Make parallel determinations of two test materials. Take the average value.

6.3 Blank test

Along with spodumene and lepidolite concentrates, conduct blank test of test material respectively.

6.4.1 Place the test material (6.1) in a 30mL nickel crucible. Add 3 drops of

absolute ethanol (3.3) to moisten. Add 2g of potassium hydroxide (3.1) and 2g of sodium hydroxide (3.2). Put the lid on the crucible and leave a little gap.

Remove water on a low temperature electric furnace. Place in a high temperature furnace of 600°C~650°C. Melt for 10min~15min. Take out. Cool.

6.4.2 Use filter paper to wipe the outer wall of the crucible. Place in a 125mL

porcelain evaporating dish with handle. Cover the watch glass. Use hot water to leach the melt. Wash the crucible. Take it out.

6.4.3 In the test solution (6.4.2) of lepidolite concentrate, add 20mL of boric acid saturated solution (3.12). The test solution of spodumene concentrate does not need to add saturated solution of boric acid.

6.4.4 Add 20mL of hydrochloric acid (3.5). Place on a boiling water bath and evaporate to a volume of about 10mL~15mL. Remove. Cool.

6.4.5 Add 5mL of polyredoxane solution (3.15). Use a glass rod to stir well. Let stand for 5min~10min.

6.4.6 Use medium-speed quantitative filter paper to filter. The filtrate is accepted by a 250mL volumetric flask. Add a small amount of hot hydrochloric acid (3.7) to the porcelain evaporating dish. Use a wiper rod to wipe. Use hot hydrochloric acid to wash the evaporating dish. Wash the precipitate 7~8 times. Then use hot water to wash the precipitate. Use silver nitrate solution (3.11) to check that there is no chloride ion.

6.4.7 Place the precipitate and filter paper in a platinum crucible. After ashing on the electric stove, place in a high temperature furnace at 950°C~1000°C for 1.5h. Take out. Cool for a while. Place in a desiccator to cool to room temperature. Weigh. Then put the crucible into a high temperature furnace of 950°C~1000°C and burn for 0.5h. Take out. Cool for a while. Place in the desiccator to cool to room temperature. Weigh. Repeat the operation until the amount becomes constant.

6.4.8 Add 3~5 drops of water along the wall of the crucible to wet the precipitation. Add 4 drops of sulfuric acid (3.8), 10mL of hydrofluoric acid (3.4). Evaporate on a low-temperature electric stove until there is white smoke. Remove. Cool for a while. Add 5mL of hydrofluoric acid (3.4). Continue to heat until all white smoke is emitted.