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

YS/T 254.1-2011

**Methods for chemical analysis of beryllium concentrate and
beryl. Part 1: Determination of beryllium oxide content.
Phosphate gravimetry**

铍精矿、绿柱石化学分析方法 第1部分：氧化铍量的测定磷酸盐重量法
(English Translation)

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1 Scope

This Part of YS/T 254 specifies the method for determining the content of beryllium oxide in beryllium concentrate and beryl.

This Part is applicable to the determination of beryllium oxide in beryllium concentrate and beryl. The determination range is 6.00%~14.00%.

2 Method summary

The test material is melted with potassium hydrofluoride. Beryllium is converted into beryllium fluoride salt. After sulfuric acid fumes and melts, it is converted into beryllium sulfate. Leach with water. Acidify with hydrochloric acid. Use hydrogen peroxide and disodium ethylenediaminetetraacetate to complex impurity elements. In the presence of ammonium acetate, diammonium hydrogen phosphate is used to precipitate beryllium twice at a pH value of 5.2~5.8. The precipitate is burned into beryllium pyrophosphate at 1000°C. Weigh to constant.

3 Reagents

3.1

Potassium hydrofluoride.

3.2

Sulfuric acid ($\rho=1.84$ g/mL).

3.3

Hydrochloric acid (rho1.19 g/mL).

3.4

Hydrogen peroxide (rho1.10 g/mL).

3.5

Ammonia (rho0.90 g/mL).

3.6

Hydrochloric acid solution (1+3).

3.7

Ammonia (1+1).

3.8 Diammonium hydrogen phosphate solution (132.5 g/L): Weigh 132.5 g of

diammonium hydrogen phosphate. Dissolve in 500 mL of water. Filter. Transfer to a

5.3.3 Move the platinum dish into a 700°C high temperature furnace. Melt it until it becomes transparent. Take out. Cool down.

5.3.4 Wipe the outer wall of the platinum dish. Dip the melt into a 400 mL beaker with hot water. Add 5 mL of hydrochloric acid (3.3). Heat and boil to make the solution clear.

5.3.5 Add 3 mL of hydrogen peroxide (3.4), 10 mL of disodium

ethylenediaminetetraacetate solution (3.9), and 8 mL of diammonium hydrogen phosphate solution (3.8) into the beaker. Neutralize with ammonia water (3.5) until obvious precipitation appears. Slowly add 20 mL of ammonium acetate solution (3.10) while stirring. Heat and boil for 2 min ~ 3 min.

5.3.6 Place the beaker in a boiling water bath to keep it warm. Convert the precipitate into crystalline precipitate. Cool down. Leave for more than 4 h.

5.3.7 Filter with slow quantitative filter paper. Wash the beaker and sediment 4~5 times with hot ammonium nitrate solution (3.11).

5.3.8 Use 20 mL of hot hydrochloric acid solution (3.6) to dissolve the precipitate from the funnel into the original beaker. Wash the filter paper several times with hot water.

Dilute the solution with water to a volume of approximately 100 mL.

5.3.9 Add 1 mL of hydrogen peroxide (3.4), 5 mL of disodium

ethylenediaminetetraacetate solution (3.9), and 4 mL of diammonium hydrogen phosphate solution (3.8) into the beaker. Neutralize with ammonia water (3.5) until obvious

precipitation appears. Slowly add 20 mL of ammonium acetate solution (3.10) while stirring. Heat and boil for 2 min ~ 3 min.

5.3.10 Place the beaker in a boiling water bath to keep it warm. Convert the precipitate into crystalline precipitate. Cool down. Leave for more than 4 h.

5.3.11 Filter with slow quantitative filter paper. Wipe the beaker clean with half a piece of filter paper. Wash with hot ammonium nitrate solution (3.11) until there is no chloride ion [check with silver nitrate (3.12)].

5.3.12 Place the precipitate together with the filter paper in a constant weight crucible (porcelain or corundum). Carefully dry and ashes. Place it in a high temperature furnace of 1000°C and burn it for 2 h. Take out. Cool slightly. Place in a desiccator to cool to room temperature. Weigh. Then put the crucible into a high temperature furnace of

1000°C. Burn it for 1 h. take out. Cool slightly and then place in a desiccator to cool to room temperature. Weigh to constant.

6 Calculation and presentation of analysis results

The content of beryllium oxide is calculated as the mass fraction of beryllium oxide w_{BeO} . Values are expressed in %. Calculate according to formula (1):

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