# GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

## GB/T 1873-1995

# Phosphate rock and concentrate - Determination of silicon dioxide content - Gravimetric and volumetric methods

磷矿石和磷精矿中二氧化硅含量的测定

重量法和容量法

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# Phosphate rock and concentrate - Determination of silicon dioxide content - Gravimetric and volumetric methods

## Part I -- Perchloric Acid Dehydration Gravimetric Method (Arbitration Method)

## 1 Scope

This Standard specifies the determination of silicon dioxide content by perchloric acid dehydration gravimetric method.

This Standard applies to the determination of silicon dioxide content that is greater than 1 % in phosphate rock and concentrate products.

## 2 References

The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. At the time of publication, the editions indicated are valid. All standards are subject to revision. The parties who are using this Standard shall explore the possibility of using the latest version of the following standards.

GB/T 6682-92 Water for laboratory use - Specifications

## 3 Method summary

The sample is melted with sodium hydroxide, leached with hydrochloric acid. Perchloric acid is evaporated and smoked to dehydrate the silicic acid. Filter, burn, and weigh. Desiliconize with hydrofluoric acid. Weigh the residue. Calculate the silicon dioxide content from its vector.

## 4 Reagents and solutions

The water used in this Standard shall meet the specifications of Grade 3 water in GB/T 6682; the reagents listed, unless otherwise specified, refer to analytical

COVER the watch glass, and EVAPORATE to a wet salt form.

**6.4** REMOVE the beaker, COOL, ADD 20 mL of hydrochloric acid solution (4.4), RINSE the watch glass and beaker wall with hot water until the solution volume is about 50 mL, and HEAT to dissolve the soluble salts. LET IT STAND for a while, FILTER it with slow quantitative filter paper. First WASH the beaker and precipitate 4 ~ 6 times with hot hydrochloric acid solution (4.6), then WASH it with hot water until there are no chloride ions. INSPECT with silver nitrate solution (4.10).

**6.5** PLACE the precipitate together with the filter paper into a platinum crucible. After careful drying and ashing, PLACE it in a high-temperature furnace and BURN at 950 °C for 60 min. TAKE OUT and COOL in a desiccator for 30 min, WEIGH. REPEAT burning for 20 min until the mass is constant.

**6.6** ADD several drops of water to the platinum crucible to wet the precipitate, ADD 3 ~ 5 drops of sulfuric acid solution (4.8) and 3 ~ 5 mL of hydrofluoric acid (4.9), and slowly HEAT to evaporate to nearly dry. REPEAT the process once more and continue to HEAT until all white sulfur trioxide smoke is emitted. PLACE it in a high-temperature furnace and BURN at 950 °C for 60 min. TAKE OUT and COOL in a desiccator for 30 min, WEIGH. REPEAT burning for 20 min until the mass is constant.

## 7 Expression of analysis results

The silicon dioxide ($SiO_2$) content (X) expressed in mass percentage is calculated according to formula (1):

$$X = \frac{(m_1 - m_2) - (m_3 - m_4)}{m} \times 100 \quad \cdots\cdots (1)$$

where:

$m_1$ - the mass of precipitation and platinum crucible before hydrofluoric acid treatment, g;

$m_2$ - the mass of precipitation and platinum crucible after hydrofluoric acid treatment, g;

$m_3$ - the mass of precipitation and platinum crucible before hydrofluoric acid treatment of the blank test, g;

$m_4$ - the mass of precipitation and platinum crucible after hydrofluoric acid treatment of the blank test, g;

$m$ - the mass of the sample, g.

## 11 Method summary

The sample is melted with sodium hydroxide, leached with water, and acidified. Add potassium chloride and potassium fluoride to the nitric acid solution to precipitate silicic acid in the form of potassium fluosilicate. Filter and wash to remove free acid. Hydrolyze with boiling water to generate hydrofluoric acid. Use bromothymol phenol blue-phenol red as an indicator. Titrate with sodium hydroxide standard titration solution. Then the silicon dioxide content can be determined.

## 12 Reagents and solutions

The water used in this Standard shall meet the specifications of Grade 3 water in GB/T 6682; the reagents listed, unless otherwise specified, refer to analytical reagents.

**12.1** Potassium hydroxide (GB/T 2306).

**12.2** Hydrochloric acid (GB/T 622).

**12.3** Hydrochloric acid solution: 1 + 9.

**12.4** Nitric acid (GB/T 626).

**12.5** Potassium chloride (GB/T 646).

**12.6** Potassium fluoride solution: 200 g/L. WEIGH 40 g of potassium fluoride (GB/T 1271) and PLACE in a polyethylene beaker, ADD 150 mL of water and 50 mL of nitric acid (12.4), ADD solid potassium chloride (12.5) to saturate, LEAVE it for 30 min, FILTER with quick filter paper, and STORE the filtrate in a polyethylene bottle.

**12.7** Potassium chloride-ethanol washing solution I: WEIGH 50 g of potassium chloride (12.5) and DISSOLVE in 800 mL of water and 200 mL of ethanol (GB/T 679), ADD a few drops of 1 g/L methyl red indicator solution, and ADJUST with sodium hydroxide standard titration solution (4.9) until the color turns yellow.

**12.8** Potassium chloride-ethanol washing solution II: WEIGH 50 g of potassium chloride (4.5) and DISSOLVE in 500 mL of water and 500 mL of ethanol (GB/T 679).

**12.9** Sodium hydroxide (GB/T 629) standard titration solution: $c$(NaOH) = 0.1 mol/L. Preparation and calibration are performed according to GB/T 601.

**12.10** Neutral boiling water: ADD a few drops of indicator liquid (12.11) to the

boiling water, and ADJUST with sodium hydroxide standard titration solution (12.9) until the color turns bright purple.

**12.11** Mixed indicator liquid (bromothymol blue-phenol red): WEIGH 0.09 g of bromothymol blue (HG/T 3-1222) and 0.11 g of phenol red, DISSOLVE in 20 mL of ethanol (GB/T 679) and 20 mL water, ADJUST with sodium hydroxide standard titration solution (12.9) until the color turns bright purple, and DILUTE to 100 mL with water.

## 13 Sample

The sample is passed through a 125 pm test sieve (GB 6003), dried at 105 ~ 110 °C for more than 2 h, placed in a desiccator and cooled to room temperature.

## 14 Analysis steps

**14.1** WEIGH 0.1 ~ 0.15 g of sample to the nearest 0.0001 g, PLACE it in a silver crucible, and ADD 2 g of potassium hydroxide (12.1). CARRY OUT a blank test at the same time.

**14.2** COVER the crucible lid, leaving a gap. PLACE it in a high temperature furnace, slowly INCREASE the temperature from low temperature to 650 ~ 700 °C and keep it for 10 min. TAKE OUT the crucible and turn it, COOL it a little, PLACE it in a 250 mL polyethylene beaker, ADD 15 ~ 20 mL of boiling water, and immediately COVER the watch glass. STIR with a polyethylene rod. WASH the crucible with a small amount of water and hydrochloric acid solution (12.3), and CONTROL the volume to 30 ~ 40 mL. With constant stirring, quickly ADD 8 mL of hydrochloric acid (12.2), STIR the solution until it is clear, and COOL.

NOTE: For samples containing high aluminum, it shall avoid introducing sodium salts to interfere with the determination. For samples with low aluminum content, it may pipette 25.0 mL of sample solution A prepared from GB/T 1871.1, Part 1, 7.1.1.3 into a 250 mL polyethylene beaker, and then perform in accordance with 6.3.

**14.3** ADD 10 mL of nitric acid (12.4), PLACE the beaker in a cold water bath, add about 3 g of solid potassium chloride (12.5), and carefully STIR until it is saturated and there is a small amount of potassium chloride that is not dissolved. With stirring, slowly ADD 10 mL of potassium fluoride solution (12.6). Continue stirring for 1 min, and leave it for about 20 min.

**14.4** FILTER (or suction filter) using a polyethylene funnel (or wax-coated funnel) and fast filter paper with filter paper pulp. WASH the beaker and precipitate with potassium chloride-ethanol washing solution I (12.7) 2 to 3 times each.

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