

ICS 73.060

YS

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

YS/T 820.8-2012

**Methods for Chemical Analysis of Laterite Nickel Ores - Part 8:
Determination of Silica Content - Potassium Silica fluoride
Titrimetric Method**

红土镍矿化学分析方法 第8部分：二氧化硅量的测定 氟硅酸钾滴定法
(English Translation)

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1 Scope

This Part of YS/T 820 specifies the determination of silica content in the laterite nickel ores.

This Part is applicable to the determination of silica content in the laterite nickel ores.

The determination range is 5.00%~45.00%.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

YS/T 820.24-2012 Methods for Chemical Analysis of Laterite Nickel Ores - Part 24:
Determination Hygroscopic Moisture Content - Gravimetric Method

3 Method Summary

The testing material is melted by potassium hydroxide, leached in water, and acidified.

In the nitric acid solution, add potassium chloride and potassium fluoride to precipitate the silicic acid in the form of potassium fluorosilicate; filter and wash to remove the free acid; use boiling water to hydrolyze and generate hydrofluoric acid; use bromothymol blue-phenol red as indicator; titrate by sodium hydroxide standard solution; and calculate the content of silica.

4 Reagents

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

4.1

Potassium hydroxide.

4.2

Potassium chloride.

4.3

Hydrochloric acid (ρ 1.19g/mL).

4.4

Nitric acid (ρ 1.42g/mL).

4.5

Hydrochloric acid (1+9).

4.6

Potassium fluoride solution (200g/L)

Take 40g of potassium fluoride [KF·2H₂O] in a polyethylene beaker; add 150mL of water and 50mL of nitric acid (4.4); add potassium chloride (4.2) to saturation; and stand for 30min; filter by quick filter paper;

and store the filtrate in a polyethylene container.

4.7 Saturated potassium chloride-ethanol lotion: mix absolute ethanol and carbon

dioxide-free water according to (1+2), and add potassium chloride (4.2) to saturation.

4.8 Neutral water: in 2000mL carbon dioxide-free boiling water, add 1mL of bromothymol blue-phenol red mixed indicator solution (4.9); adjust to a bright blue-violet by sodium hydroxide standard titration solution (4.11).

4.9 Bromothymol blue-phenol red mixed indicator solution: take 0.1g of bromothymol

blue [C₂₇H₂₈O₅Br₂S] and phenol red [C₁₉H₁₄O₅S] indicators, respectively; and dissolve them in 20mL of absolute ethanol; add 30mL of hot water; and stir to complete dissolve and mix evenly.

4.10 Sodium hydroxide stock solution (cNaOH ~1.0mol/L): take 40g of sodium hydroxide

and dissolve it in carbon dioxide-free water; dilute to 1000mL; mix evenly; store in a polyethylene container; and place it tightly until the solution is clear.

4.11 Sodium hydroxide standard titration solution (cNaOH~0.1mol/L).

4.11.1 Preparation

Pipette 100mL of sodium hydroxide stock solution (4.10); dilute to 1000mL by carbon dioxide-free water; mix evenly; and store in a tubulated bottle equipped with soda asbestos tube.

4.11.2 Calibration

Take 0.50g (accurate to 0.0001g) of reference potassium hydrogen phthalate [KHC₈H₄O₄] (dried to constant weight at 105°C ~110°C); place it in a 250mL conical flask; add 100mL of carbon dioxide-free hot water and dissolve it. After cooling, add 4 drops of phenolphthalein indicator (10g/L); titrate by sodium hydroxide standard titration solution (4.11) to pink; and keep it for 30s without fading. Do a blank test at the same time. Three portions are calibrated in parallel, and the volume range of the

6.2 The number of determinations Perform 2 determinations independently, and take their average value.

6.3 Blank test

Do the blank test with the testing material.

6.4.1 Place the testing material (6.1) in a 30mL nickel crucible; add 4g of potassium

hydroxide (4.1); heat it on a hot plate to expel the moisture; cover the crucible lid with a small gap; and move it into a 650°C high temperature furnace to melt for 10min~15min. Take it out, cool to room temperature; place it in a 250mL polyethylene beaker; leaching the frit with a small amount of boiling water for several times. And then wash out the crucible with hydrochloric acid (4.5); control the volume not to exceed 50mL. Under constant stirring, quickly add 20mL of hydrochloric acid (4.3); and stir until the solution is clear.

6.4.2 Add 10mL of nitric acid (4.4); place the beaker in a cold-water bath till it reaches

room temperature; add 3g of potassium chloride (4.2) and a little filter paper pulp; and stir well. Slowly add 10mL of potassium fluoride solution (4.6) under stirring; continue stirring for

1min~2min; and stand for about 15min.

6.4.3 Use a plastic funnel with absorbent cotton and filter paper pulp (previously rinse

with saturated potassium chloride-ethanol lotion (4.7) for 2~3 times) to filter; and use saturated potassium chloride-ethanol lotion (4.7) to purge the polyethylene beaker.

Completely transfer the precipitate to the funnel and wash until the precipitate has no acid reaction.

NOTE: The acid-free reaction refers to taking 3mL~4mL of the filtrate for washing the precipitate; adding 1 drop of indicator solution (4.9); adding 1 drop of sodium hydroxide standard titration solution (4.11); showing bright blue-purple, then it indicates that it has been cleaned.

6.4.4 Place the precipitate and absorbent cotton in a polyethylene beaker; add 6 drops

of indicator solution (4.9); add 150 mL of boiling neutral water (4.8); stir evenly; and immediately use sodium hydroxide standard titration solution (4.11) to titrate till a stable bright blue-violet appears.