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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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**Ferrosilicon - Determination of aluminum, calcium, manganese,
chromium, titanium, copper, phosphorus and nickel content -
Inductively coupled plasma atomic emission spectrometric method**

硅铁 铝、钙、锰、铬、钛、铜、磷和镍含量的测定 电感耦合等离子体原子发射
光谱法

(English Translation)

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1 Scope

This standard specifies methods for the determination of aluminum, calcium, manganese, chromium, titanium, copper, phosphorus and nickel content by inductively coupled plasma atomic emission spectrometry (ICP-AES).

This standard applies to the determination of aluminum, calcium, manganese, chromium, titanium, copper, phosphorus and nickel content in ferrosilicon. The measurement range of each element is as shown in Table 1.

Table 1 -- Elements and measurement range

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard;

however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable.

Analytical element Determination range (mass fraction) /%

4.6.1.1 Aluminum standard stock solution, 1.00 mg/mL.

Weigh 1.0000 g of metal aluminum (> 99.95%) in a 250 mL polytetrafluoroethylene beaker.

Add 30 mL of hydrochloric acid (1 + 1). Heat it at low temperature to dissolve it. Cool it to room temperature. Transfer it into a

1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly.

Note: It may also use the following standard method to prepare aluminum standard stock solution. Weigh 1.000 g of metal aluminum (> 99.95%) in a polytetrafluoroethylene beaker. Add 30 mL of sodium hydroxide solution (200 g/L). Heat it at low temperature to dissolve it. Add 100 mL of water. Add hydrochloric acid (1 + 1) to acidify it and add 10 mL in excess. Cool it to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly.

4.6.1.2 Aluminum standard stock solution, 50.00 µg/mL.

Divide 25.00 mL of aluminum standard stock solution (4.6.1.1) into a 500 mL volumetric flask. Add 20 mL of hydrochloric acid (4.4). Use water to dilute it to the mark. Mix it uniformly.

4.6.2 Calcium standard stock solution

4.6.2.1

Calcium standard stock solution, 1.00 mg/mL.

Weigh 2.4973 g of calcium carbonate (> 99.99%) which was pre-dried at 105 °C

± 5 °C and cooled to room temperature in a desiccator in a 250 mL beaker. Add

20 mL of water. Mix it uniformly. Cover the watch glass. Carefully add hydrochloric acid (1 + 1) to dissolve the calcium carbonate. Add 20 mL of hydrochloric acid (1 + 1). Boil off the carbon dioxide. Cool it to room temperature.

Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark.

Mix it uniformly.

4.6.2.2

Calcium standard stock solution, 50.00 µg/mL.

Divide 25.00 mL of calcium standard stock solution (4.6.2.1) into a 500 mL volumetric flask. Add 20 mL of hydrochloric acid (4.4). Use water to dilute it to the mark. Mix it uniformly.

4.6.3 Manganese standard stock solution

4.6.3.1

Manganese standard stock solution, 1.00 mg/mL.

Weigh 1.0000 g of electrolytic manganese (> 99.95%) in a 250 mL beaker. Add

30 mL of nitric acid (1 + 1). Heat to dissolve it. Boil off the nitrogen oxides. Cool it to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark.

Mix it uniformly.

Weigh 0.5000 g of metallic copper (> 99.95%) in a 250 mL beaker. Add 20 mL of nitric acid (1 + 1). Dissolve it at low temperature. Heat to boil it to remove nitrogen oxides. Cool it to room temperature. Transfer it to a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly.

Note: If necessary, the oxide or basic salt on the surface of the metallic copper is removed in advance by the use of nitric acid.

4.6.6.2 Copper standard stock solution, 50.00 µg/mL.

Divide 50.00 mL of copper standard stock solution (4.6.6.1) into a 500 mL volumetric flask. Add 20 mL of hydrochloric acid (4.4). Use water to dilute it to the mark. Mix it uniformly.

4.6.7 Phosphorus standard stock solution

4.6.7.1

Phosphorus standard stock solution, 0.50 mg/mL.

Weigh 2.1968 g of potassium dihydrogen phosphate (> 99.95%) which has been dried to a constant amount at 105 °C ± 5 °C and cooled to room temperature. Use a small amount of water to dissolve it. Transfer it to a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly.

4.6.7.2

Phosphorus standard stock solution, 50.00 µg/mL.

Divide 50.00 mL of phosphorus standard stock solution (4.6.7.1) into a 500 mL volumetric flask. Add 20 mL of hydrochloric acid (4.4). Use water to dilute it to the mark. Mix it uniformly.

4.6.8 Nickel standard stock solution

4.6.8.1

Nickel standard stock solution, 0.50 mg/mL.

Weigh 0.5000 g of metallic nickel (> 99.95%) in a 250 mL beaker. Add 20 mL of nitric acid (1 + 1). Heat to dissolve it. Boil off the nitrogen oxides. Cool it to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly.

4.6.8.2

Nickel standard stock solution, 50.00 µg/mL.

Divide 50.00 mL of nickel standard stock solution (4.6.8.1) into a 500 mL volumetric flask. Add 20 mL of hydrochloric acid (4.4). Use water to dilute it to the mark. Mix it uniformly.

4.6.9 Yttrium standard stock solution, 200.0 µg/mL.

Weigh 0.2540 g of cerium oxide (> 99.95%) which was previously calcined at 750 °C ± 5 °C for 30 min and cooled to room temperature into a 250 mL beaker.

7.2 Sample quantity

Weigh 0.5000 g (accurate to 0.0002 g) of specimen.

7.3 Blank test

A blank test is performed in parallel with the sample.

Note: For the blank test, it shall use an appropriate amount of pure iron (4.7) instead of the specimen. The amount of iron in the specimen varies within ±5%, which has no significant impact on the spectral intensity of the element to be tested. Weigh 0.12 g of pure iron, which is equivalent to the specimen containing 24% of iron (about 70% ~ 75% silicon). According to the iron content (or silicon content) of the specimen, it may adjust the amount of pure iron weighed. For example, when the amount of silicon is 65%, it may weigh 0.15 g of pure iron (about 65% ~ 70% of silicon).

7.4.1 Preparation of sample solution

Place the sample in a 200 mL Teflon beaker. Use a small amount of water to wet it. Add 10 mL of nitric acid (4.1). Use a plastic tube to carefully add about 5 mL of hydrofluoric acid (4.2), until the reaction is stopped. Add 5 mL of hydrochloric acid (4.4). Slowly heat it until the sample is completely dissolved.

Add about 8 mL of perchloric acid (4.3). Heat it at low temperature to a perchloric acid smoke. Use water to rinse the beaker's wall. Continue to heat to produce the smoke, until about 2 mL ~ 3 mL of solution is left. Slightly cool it.

Add 20 mL of hydrochloric acid (4.5). Heat to dissolve the salts. Cool it to room temperature. Transfer the test solution to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. For measurement by internal standard method, add 5.00 mL of hydrazine standard solution (4.6.9) before dilution.

Note: Perchloric acid shall maintain a low temperature when smoking (the reference temperature is to control the surface temperature of the electric furnace at about 200 °C). It shall not emit thick white smoke; otherwise it will cause loss of chromium.

7.4.2 Preparation of calibration solution

Use the pure iron (4.7) which is equivalent to the amount of iron in the sample to replace the sample. Operate according to 7.4.1. Before finally diluting it to

100 mL, add the standard solution (4.6) of each analytical element.