YS

NONFERROUS INDUSTRY STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

ICS 77.120.60

H 13

YS/T 928.4-2013

Methods for Chemical Analysis of Nickel Cobalt Manganese Composite Hydrogenoxide - Part 4: Determination of Iron, Calcium, Magnesium, Copper, Zinc, Silicon, Aluminum, Sodium Contents - Inductively Coupled Plasma Atomic Emission Spectrometric

镍、钴、锰三元素氢氧化物化学分析方法 第 4 部分：铁、钙、镁、铜、锌、硅、铝、钠量的测定 电感耦合等离子体原子发射光谱法

Issued on: October 17, 2013 **Implemented on: March 1, 2014**

Issued by: Ministry of Industry and Information Technology of the People's Republic of China

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1 Scope

This Part of YS/T 928 specifies the method for the determination of iron, calcium, magnesium, copper, zinc, silicon, aluminum and sodium content in nickel cobalt manganese composite hydrogenoxide.

This Part is applicable to the determination of iron, calcium, magnesium, copper, zinc, silicon, aluminum and sodium content in nickel cobalt manganese composite hydrogenoxide. See Table 1 for the determination scope.

2 Method Summary

Use hydrochloric acid to dissolve the test portion and adopt an inductively coupled plasma atomic emission spectrometer to directly conduct the determination. In accordance with the working curve method, calculate the concentration of each element. Express the determination results in mass fraction.

3 Reagents

All reagents used in this Part are of top-grade purity, and only pure water with an electrical resistivity of not less than 18.2 MΩ • cm shall be used in the analysis.

3.1 Hydrochloric acid (ρ = 1.19 g/mL).

3.2 Nitric acid (ρ = 1.42 g/mL).

3.3 Hydrochloric acid (1 + 1).

3.4 Nitric acid (1 + 1).

3.5 Aluminum standard stock solution: weigh-take 0.2000 g of pure aluminum ($w_{Al} \geq 99.99\%$) in a 300 mL beaker, slowly add 30 mL of hydrochloric acid (3.3), use a watch glass to cover it; at a low temperature, dissolve it, then, cool it. Use water to wash the watch glass and beaker wall. Transfer to a 1,000 mL volumetric flask, use water to dilute to the scale and mix it well. 1 mL of this solution contains 200 μg of aluminum.

3.6 Magnesium standard stock solution: weigh-take 0.3316 g of dried magnesium oxide ($w_{MgO} \geq 99.99\%$) in a 300 mL beaker, slowly add 30 mL of hydrochloric acid (3.3), use a watch glass to cover it; at a low temperature, dissolve it, then, cool it. Use water to wash the watch glass and beaker wall. Transfer to a 1,000 mL volumetric flask, use water to dilute to the scale and mix it well. 1 mL of this solution contains 200 μg of magnesium.

3.7 Calcium standard stock solution: weigh-take 0.2800 g of dried calcium oxide ($w_{CaO} \geq 99.99\%$) in a 300 mL beaker, slowly add 30 mL of hydrochloric acid (3.3), use a watch glass to cover it; at a low temperature, dissolve it, then, cool it. Use water to wash the watch glass and beaker wall. Transfer to a 1,000 mL volumetric flask, use water to dilute to the scale and mix it well. 1 mL of this solution contains 200 μg of calcium.

3.8 Copper standard stock solution: weigh-take 0.2000 g of pure copper ($w_{Cu} \geq 99.99\%$) in a 300 mL beaker, slowly add 30 mL of nitric acid (3.4), use a watch glass to cover it; at a low temperature, dissolve it and drive out nitrogen oxides, then, remove and cool it. Use water to wash the watch glass and beaker wall. Transfer to a 1,000 mL volumetric flask, use water to dilute to the scale and mix it well. 1 mL of this solution contains 200 μg of copper.

3.9 Iron standard stock solution: weigh-take 0.2000 g of pure iron ($w_{Fe} \geq 99.99\%$) in a 300 mL beaker, slowly add 30 mL of hydrochloric acid (3.3), use a watch glass to cover it; at a low temperature, dissolve it, then, remove and cool it. Use water to wash the watch glass and beaker wall. Transfer to a 1,000 mL volumetric flask, use water to dilute to the scale and mix it well. 1 mL of this solution contains 200 μg of iron.

3.10 Zinc standard stock solution: weigh-take 0.2000 g of pure zinc ($w_{Zn} \geq 99.99\%$) in a 300 mL beaker, slowly add 30 mL of nitric acid (3.4), use a watch glass to cover it; at a low temperature, dissolve it and drive out nitrogen oxides, then, remove and cool it. Use water to wash the watch glass and beaker wall. Transfer to a 1,000 mL volumetric flask, use water to dilute to the scale and mix it well. 1 mL of this solution contains 200 μg of zinc.

3.11 Sodium standard stock solution: weigh-take 0.5084 g of high-purity sodium chloride that has been dried at 110 °C and place it in a 300 mL beaker, slowly add 100 mL of water, use a watch glass to cover it; at a low temperature, dissolve it, then, cool it. Use water to wash the watch glass and beaker wall. Transfer to a 1,000 mL volumetric flask, use water to dilute to the scale, mix it well, and immediately transfer it into a dry plastic bottle. 1 mL of this solution contains 200 μg of sodium.

3.12 Silicon standard stock solution: weigh-take 0.4279 g of silica ($w_{SiO_2} \geq 99.99\%$) and place it in a platinum crucible, add 3 g of anhydrous sodium carbonate, mix it well, cover it, then, transfer it to a 400 °C muffle furnace. Raise the temperature to 900 °C, conduct melting for 1 h, then, remove and cool it. Use water to wash the outer wall of the crucible, place it in a 400 mL polytetrafluoroethylene plastic beaker and add 100 mL of hot water. At a low temperature, dissolve it, cool it, then, transfer to a 1,000 mL volumetric flask. Use water to dilute to the scale, mix it well, and immediately transfer it into a dry plastic bottle. 1 mL of this solution contains 200 μg of silicon.

3.13 Aluminum, magnesium, calcium, copper, iron and zinc standard solutions: accurately transfer-take 10.00 mL of the standard stock solutions (3.5, 3.6, 3.7, 3.8, 3.9 and 3.10) in a 200 mL volumetric flask. Use water to dilute to the scale and mix it well. 1 mL of this solution contains 10 μg of aluminum, magnesium, calcium, copper, iron and zinc.

3.14 Sodium standard solution: accurately transfer-take 10.00 mL of the standard stock solution (3.11) in a 200 mL volumetric flask. Use water to dilute to the scale, mix it well, and immediately transfer it into a dry plastic bottle. 1 mL of this solution contains 10 μg of sodium.

3.15 Silicon standard solution: accurately transfer-take 10.00 mL of the standard stock solution (3.12) in a 200 mL volumetric flask. Use water to dilute to the scale, mix it well, and immediately transfer it into a dry plastic bottle. 1 mL of this solution contains 10 μg of silicon.

4 Instrument

In terms of the inductively coupled plasma atomic emission spectrometer, the spectral lines of the various element are shown in Table 2; the working parameters of the instrument for sodium determination are shown in Table 3; the working parameters of the instrument for the determination of other elements are shown in Table 4.

Table 2 -- Element Spectral Lines

Element	Ca	Cu	Fe	Mg
Wavelength/nm	393.3	324.7	259.9	279.5
Element	Zn	Al	Na	Si
Wavelength/nm	206.2	167.0	588.9	212.4

with the condition parameters provided in Table 4, use water and silicon standard solution to determine the intensity value of silicon, and the instrument automatically fits the working curve.

5.4.2 Accurately transfer-take 0 mL, 2.00 mL, 5.00 mL, 10.00 mL, 20.00 mL and 30.00 mL of the standard solution (3.14) in a 200 mL volumetric flask. Use water to dilute to the scale, mix it well, and immediately transfer into a dry plastic bottle. On ICP spectrometer, in accordance with the condition parameters provided in Table 3, use water and sodium standard solution to determine the intensity value of sodium, and the instrument automatically fits the working curve.

5.4.3 Accurately transfer-take 0 mL, 2.00 mL, 5.00 mL, 10.00 mL, 20.00 mL and 30.00 mL of the standard solution (3.13) in a 200 mL volumetric flask. Use water to dilute to the scale and mix it well. On ICP spectrometer, in accordance with the condition parameters provided in Table 4, use water and aluminum, iron, calcium, magnesium, copper and zinc standard solutions to determine the intensity values of aluminum, iron, calcium, magnesium, copper and zinc, and the instrument automatically fits the working curve.

6 Calculation and Expression of Analysis Results

In accordance with Formula (1), calculate the mass fraction w_x of the element being determined and express it as a percentage.

$$w_x = \frac{(\rho - \rho_0) \times V \times 10^{-6}}{m} \times 100\% \qquad \cdots\cdots (1)$$

Where,

w_x---respectively the mass fraction of iron, calcium, magnesium, copper, zinc, silicon, aluminum and sodium, expressed in (%);

ρ---the concentration of the element in the test solution, expressed in (μg/mL);

ρ_0---the concentration of the element in the blank test solution, expressed in (μg/mL);

V---the total volume of the test solution, expressed in (mL);

m---the mass of the test portion, expressed in (g).

The result shall retain two decimal places.

7 Precision

7.1 Repeatability

Under repeatability conditions, obtain the determined values of two independent test results. Within the average value range provided in Table 5, the absolute difference between the two test results does not exceed the repeatability limit (r), and the circumstances of exceeding the

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