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Methods for chemical analysis of fire smelting nickel substrate material - Part 10: Determination of nickel, chromium, manganese, cobalt, copper and phosphorus contents - Inductively coupled plasma atomic emission spectrometric method

火法冶炼镍基体料化学分析方法 第 10 部分：镍、铬、锰、钴、铜、磷量的测定 电感耦合等离子体原子发射光谱法

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Foreword

YS/T 953 "Methods for chemical analysis of fire smelting nickel substrate material" is divided into 11 parts:

- Part 1: Determination of nickel content - Dimethylglyoxime spectrophotometric method and dimethylglyoxime gravimetric method;
- Part 2: Determination of silicon content - Molybdosilicate blue spectrophotometric method and perchloric acid dehydration gravimetric method;
- Part 3: Determination of phosphorus content - Bismuth phosphomolybdate blue spectrophotometric method;
- Part 4: Determination of chrome content - Ammonium ferrous sulfate titration method;
- Part 5: Determination of manganese content - Potassium periodate spectrophotometric method;
- Part 6: Determination of cobalt content - 5-CI-PADAB spectrophotometric method and flame atomic absorption spectrometric method;
- Part 7: Determination of copper content - BCO spectrophotometric method and flame atomic absorption spectrometric method;
- Part 8: Determination of iron content - Titanium trichloride reduction-potassium dichromate titrimetric method;
- Part 9: Determination of carbon and sulfur contents - Infrared absorption method after high frequency combustion;
- Part 10: Determination of nickel, chromium, manganese, cobalt, copper and phosphorus contents - Inductively coupled plasma atomic emission spectrometric method;
- Part 11: Determination of Lead, Arsenic, Cadmium and Mercury contents - Inductively coupled plasma-mass spectrometric method.

This Part is Part 10 of YS/T 953.

This Part is drafted in accordance with the rules given in GB/T 1.1-2009.

This Part shall be under the jurisdiction of National Technical Committee 243 on Nonferrous Metals of Standardization Administration of China (SAC/TC 243).

Methods for chemical analysis of fire smelting nickel substrate material - Part 10: Determination of nickel, chromium, manganese, cobalt, copper and phosphorus contents - Inductively coupled plasma atomic emission spectrometric method

1 Scope

This Part of YS/T 953 specifies the inductively coupled plasma atomic emission spectrometric method for the determination of nickel, chromium, manganese, cobalt, copper and phosphorus contents in the fire smelting nickel substrate material.

This Part applies to the determination of nickel, chromium, manganese, cobalt, copper and phosphorus contents in the fire smelting nickel substrate material. The determination range is shown in Table 1.

Table 1 -- Determination range

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 20066 Steel and iron - Sampling and preparation of samples for the determination of chemical composition

3 Method summary

USE hydrochloric acid, nitric acid and hydrofluoric acid to dissolve the test portion; ADD perchloric acid to blow off the fluoride. If necessary, add yttrium as an internal standard; USE an inductively coupled plasma atomic emission spectrometer to directly measure. Calculate the mass concentration of each element according to the working curve method; EXPRESS the determination result in mass fraction.

4 Reagents

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

4.1 Hydrochloric acid (ρ=1.19 g/mL).

4.2 Nitric acid (ρ=1.42 g/mL).

4.3 Hydrofluoric acid (ρ=1.15 g/mL).

4.4 Perchloric acid (ρ=1.67 g/mL).

4.5 Standard stock solutions of chromium, cobalt, copper, manganese, phosphorous, nickel and yttrium: commercially-available certified reference solutions. Mass concentration is all 1000 μg/mL.

4.6 Mixed standard solution: Respectively PIPETTE 5.00 mL each of copper and phosphorus single-element standard stock solutions (4.5), 10.00 mL each of chromium, cobalt, and manganese single-element standard stock solutions (4.5), and 20.00 mL of nickel standard stock solution (4.5) INTO a 100 mL volumetric flask. ADD 5 mL of hydrochloric acid (4.1); USE water to dilute to the mark and mix well. 1 mL of this solution contains 50 μg each of copper and phosphorus, 100 μg each of chromium, cobalt, and manganese, and 200 μg of nickel.

4.7 Yttrium internal standard solution: PIPETTE 25.00 mL of yttrium single-element standard stock solution (4.5) into a 1000 mL volumetric flask; ADD 50 mL of hydrochloric acid (4.1); USE water to dilute to the mark and mix well. 1 mL of this solution contains 25 μg of yttrium.

Perform two determinations independently and take the average.

7.3 Blank test

Do a blank test along with the test portion.

7.4 Determination

7.4.1 Dissolution of test portion

PLACE the test portion (7.1) in a polytetrafluoroethylene beaker; ADD 5 mL of hydrochloric acid (4.1), 5 mL of nitric acid (4.2); HEAT to no longer react. ADD 2~3 drops of hydrofluoric acid (4.3); ADD 5 mL of perchloric acid (4.4); COVER with a watch glass; HEAT to completely dissolve the test portion; further heat to emit perchloric acid fumes; REMOVE it and cool it slightly. TRANSFER the solution to a 100 mL beaker; continue to heat until the perchloric acid fumes are exhausted; REMOVE it and cool it slightly. ADD 5 mL of hydrochloric acid (4.1); USE a small amount of water to rinse the wall of the beaker; COVER with a watch glass; HEAT to dissolve the salts until the solution is clear; REMOVE and cool. TRANSFER to the corresponding volumetric flask according to Table 3. If the internal standard method is used, use a pipette to add the corresponding volume of yttrium internal standard solution (4.7); USE water to dilute to the mark and mix well.

7.4.2 Measurement

On the inductively coupled plasma emission spectrometer, after the apparatus runs stably, under the selected apparatus working conditions, use the prepared standard series solution (7.5) to standardize or calibrate the working curve. The correlation coefficient of the working curve of each element shall be above 0.999. Otherwise it is necessary to re-standardize or re-prepare standard series solution for standardization. MEASURE the spectral intensity of the analysis test solution (7.4.1) and the blank test solution (7.3). According to the working curve, the apparatus automatically processes the data, calculates and outputs the mass concentration of each element.

7.5 Drawing of working curve

Respectively pipette 0.00 mL, 0.50 mL, 1.00 mL, 2.00 mL, 5.00 mL, 10.00 mL, 20.00 mL of mixed standard solution (4.6) into seven 100 mL volumetric flasks; ADD 5 mL of hydrochloric acid (4.1) to each. If the internal standard method is used, use a pipette to add 10.00 mL of yttrium internal standard solution (4.7); USE water to dilute to the mark and mix well. According to steps in 7.4.2, measure the spectral intensity and draw a working curve.

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