

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

SN/T 2763.4-2012

Chemical analysis of nickel laterite ore - Part 4: Determination of nickel, cobalt, aluminum, magnesium and phosphorus content - Inductively coupled plasma atomic emission spectrometric method

红土镍矿化学分析方法 第4部分：镍、钴、铝、镁、磷含量的测定 电感耦合等离子体原子发射光谱法

(English Translation)

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1 Scope

This Part of SN/T 3323 specifies inductively coupled plasma atomic emission spectrometry (ICP-AES), the testing method to determine the lead, chromium, cadmium contents in mill scale.

This Part is applicable to the determination of lead, chromium, cadmium contents in mill scale. The lower limit of determination for each element is shown in Table 1.

Table 1 -- Lower limit of determination for each element

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6379.2, Measurement methods and results - Accuracy (trueness and precision)

- Part 2: Determine the standard methods of measurement repeatability and reproducibility of the basic method

GB/T 6682, Water for analytical laboratory use - Specification and test methods

GB/T 12806, Laboratory glassware - One-mark volumetric flasks

GB/T 12808, Laboratory glassware - One-mark pipettes

3 Principle

The specimen is decomposed by hydrochloric acid. Use the inductively coupled plasma

Element Mass fraction % atomic emission spectrometer for testing. Use the matrix matching method to eliminate the matrix effects.

4 Reagents and materials

Unless otherwise stated, all reagents are analytically pure and above. Water is grade two water specified in GB/T 6682.

4.1

Hydrochloric acid ($\rho = 1.19 \text{ g/mL}$): Guaranteed reagent.

4.2

High-purity iron powder

The iron mass fraction is greater than 99.98%.

4.3

Hydrochloric acid (1+1)

Add 1 volume of hydrochloric acid (4.1) to 1 volume of water. Mix well.

4.4

Lead, chromium, cadmium standard solution (certified reference material):

1000 $\mu\text{g/mL}$.

4.5 35mg/mL iron matrix solution: Weigh 3.5g of high-purity iron powder (4.2). Add

40mL of hydrochloric acid solution (4.3). Heat to dissolve. Use water to set volume constant in a 100mL volumetric flask.

5.1.1 Performance indicator

The spectrometer can also be sequential, multi-channel or full-spectral. In either type, it shall meet the following performance indicator:

The correlation coefficient (r) of the standard curve of each element to be tested is greater than 0.995.

5.1.2 Working state of instrument

Start the ICP-AES instrument. According to the operating procedures of the instrument, warm up the instrument and adjust various conditions and measurement parameters of the instrument, so that the performance of the instrument meets the requirements of

5.1.1.

5.1.3 Confirmation of spectral lines for determination

Choose the appropriate spectral lines according to the instrument conditions. The selection of the spectral lines shall follow the following rules:

volumetric flask. Use water to set the volume constant. Shake well. Determine on the inductively coupled plasma atomic emission spectrometer with selected operating parameters.

7.4 Preparation of working curve

Add 10mL of iron matrix solution (4.5), 8mL of HCl (4.1) into a set of 100mL volumetric flasks. Respectively pipette a certain amount of standard solutions of each element into the volumetric flask. Use water to dilute to the scale. Shake well. The content of the test element shall be within the range of the working curve made. The quantity of the series of standard solution shall be decided by the precision requirements.

It requires not to be less than 4. See Annex B for the solution concentration of the recommended calibration curve.

7.5.1 Measurement of calibration curve

Adjust the instrument according to the provisions in 5.1.2. Measure the spectral intensity of each test element in the series solutions of calibration curve (7.4) at the analysis line in turn. The instrument automatically returns to establish a linear equation.

Its correlation coefficient (r) shall comply with 5.1.1 regulations.

7.5.2 Determination of test solution

Introduce the test solution (7.3) into the plasma in turn. Respectively determine the spectrum strength of each test element and internal standard elements at the same analysis line. The instrument shall automatically calculate the corresponding concentration of each element according to the calibration curve.

8 Result calculation

Calculate the content of each element according to formula (1), in mass fraction.

Where,

X_i - The content of each test element i (mass fraction), %;

c_i - The mass concentration of test element found from the working curve, in micrometers per milliliter ($\mu\text{g}/\text{mL}$);

V - The constant volume of test solution, in milliliters (mL);

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