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YS/T 820.1-2012

Methods for chemical analysis of laterite nickel ores - Part 1: Determination of nickel content - Flame atomic absorption spectrometry

红土镍矿化学分析方法 第 1 部分:镍量的测定

火焰原子吸收光谱法

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Foreword

This part was drafted in accordance with the rules given in GB/T 1.1-2009.

YS/T 820-2012 *Methods for chemical analysis of laterite nickel ores* is divided into 26 parts:

-- Part 1: Determination of nickel content - Flame atomic absorption spectrometry;

-- Part 2: Determination of nickel content - Dimethylglyoxime spectrophotometry;

-- Part 3: Determination of total iron content - Potassium dichromate titration;

-- Part 4: Determination of phosphorus content - Phosphorus molybdenum blue spectrophotometry;

-- Part 5: Determination of cobalt content - Flame atomic absorption spectrometry;

-- Part 6: Determination of copper content - Flame atomic absorption spectrometry;

-- Part 7: Determination of calcium and magnesium content - Flame atomic absorption spectrometry;

-- Part 8: Determination of silica content - Potassium silicafluoride titrimetric method;

-- Part 9: Determination of scandium and cadmium contents - Inductively coupled plasma mass spectrometry;

-- Part 10: Determination of calcium, cobalt, copper, magnesium, manganese, nickel, phosphate and zinc content - Inductively coupled plasma atomic emission spectrometry;

-- Part 11: Determination of fluorine and chlorine contents - Ion chromatography;

-- Part 12: Determination of manganese content - Flame atomic absorption spectrometry;

-- Part 13: Determination of lead content - Flame atomic absorption spectrometry;

-- Part 14: Determination of zinc content - Flame atomic absorption spectrometry;

-- Part 15: Determination of cadmium content - Flame atomic absorption spectrometry;

-- Part 16: Determination of carbon and sulfur content - High frequency combustion with infrared absorption spectrometry;

Methods for chemical analysis of laterite nickel ores - Part 1: Determination of nickel content - Flame atomic absorption spectrometry

1 Scope

This part of YS/T 820 specifies the determination method of nickel content in laterite nickel ores.

This part applies to the determination of the nickel content in laterite nickel ores. The measuring range is 0.100%~3.00%.

2 Normative references

The following documents are essential for the application of this document. For the dated referenced documents, only the versions with the indicated dates are applicable to this document; for the undated referenced documents, only the latest version (including all the amendments) is 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 sample is decomposed with hydrochloric acid, nitric acid, hydrofluoric acid, and perchloric acid. Salts are dissolved with hydrochloric acid; in a dilute hydrochloric acid medium, at the wavelength of 232.0 nm of an atomic absorption spectrometer, with the use of an air-acetylene flame, the absorbance of nickel is measured, and then the nickel content is calculated.

4 Reagents

Unless otherwise stated, only reagents confirmed to be analytical grade and distilled or deionized water or water of equivalent purity are used in the analysis.

4.1 Hydrochloric acid (ρ1.19 g/mL).

4.2 Nitric acid (ρ1.42 g/mL).

4.3 Hydrofluoric acid (ρ1.15 g/mL).

4.4 Perchloric acid (ρ1.67 g/mL).

4.5 Nitric acid (1+1).

4.6 Nickel standard storage solution: Weigh 1.0000 g of metallic nickel (w_{Ni} ≥99.95%), place it in a 400 mL beaker, and add 50 mL of nitric acid (4.5) to dissolve it completely; heat and boil to drive off nitrogen oxides, then remove the beaker to stop heating, and cool to room temperature; transfer the solution to a 1000 mL volumetric flask, dilute it to the mark with water, and mix well. 1 mL of this solution contains 1 mg of nickel.

4.7 Nickel standard solution: Pipette 10.00 mL of nickel standard stock solution (4.6) into a 100 mL volumetric flask, add 5 mL of hydrochloric acid (4.1), dilute to the mark with water, and mix well. 1 mL of this solution contains 100 μg nickel.

5 Instruments

The atomic absorption spectrometer shall be equipped with a nickel hollow cathode lamp.

Under the working conditions of the instrument, any atomic absorption spectrometer that can achieve the following indicators can be used:

-- Characteristic concentration: In the solution that is basically consistent with the measuring test solution, the characteristic concentration of nickel shall not be greater than 0.08 μg/mL;

-- Precision: The absorbance is measured 10 times with the standard solution of the maximum concentration, and the standard deviation shall not exceed 1% of the average absorbance; the absorbance is measured 10 times with the standard solution of the minimum concentration (not “zero” standard solution), and the standard deviation shall not exceed 0.5% of the average absorbance of the standard solution with the maximum concentration;

-- Working curve linearity: The working curve is divided into five equal parts according to the concentration, and the ratio of the absorbance difference of the highest section to the absorbance difference of the lowest section shall not be less than 0.70.

6 Samples

6.1 Samples

7.4.3 Put the test solution (7.4.2) at the wavelength of 232.0 nm of the atomic absorption spectrometer, use an air-acetylene flame, carry out zero setting with water, and measure the absorbance of the test solution and the blank solution. Check the corresponding nickel concentration from the working curve.

7.5 Drawing of the working curve

7.5.1 Pipette 0 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL, 5.00 mL of nickel standard solution (4.7) into a set of 100 mL volumetric flasks, add 5 mL hydrochloric acid (4.1), dilute to the mark with water, and mix well.

7.5.2 Use the air-acetylene flame, and set the atomic absorption spectrometer to zero with water at the wavelength of 232.0 nm. Measure the absorbance of the series of standard solutions, subtract the absorbance of the "zero" concentration solution in the series of standard solutions, and draw the working curve with the concentration of nickel as the abscissa and the absorbance as the ordinate.

8 Calculation of analysis results

The nickel content is expressed as the mass fraction w_{Ni} of nickel, and the value is expressed in %, calculated according to formula (1):

$$w_{Ni} = \frac{(\rho_1 - \rho_2) \cdot V_0 \cdot V_2 \times 10^{-6}}{m \cdot V_1} \times 100 \times K \quad \cdots\cdots (1)$$

$$K = \frac{100}{100 - A} \quad \cdots\cdots (2)$$

where:

ρ_1 -- the concentration of nickel in the measuring solution obtained from the working curve, in micrograms per milliliter (μg/mL);

ρ_2 -- the concentration of nickel in the blank solution obtained from the working curve, in micrograms per milliliter (μg/mL);

V_0 -- the total volume of the test solution, in milliliters (mL);

V_1 -- the volume of the test solution taken, in milliliters (mL);

V_2 -- the volume of the measuring solution, in milliliters (mL);

m -- the mass of the sample, in grams (g);

K -- conversion factor, calculated according to formula (2);

A -- the mass fraction of the hygroscopic moisture content measured according to

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