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Refined nickel - Determination of Silicon, manganese, phosphorus, iron, copper, cobalt, magnesium, aluminum, zinc, chromium - Inductively coupled plasma atomic emission spectrometric method

精炼镍 硅、锰、磷、铁、铜、钴、镁、铝、锌、铬含量的测定 电感耦合等离子体发射光谱法

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Table of Contents

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WARNING - Personnel using this standard shall have practical experience in regular laboratory work. This standard does not address all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated in relevant national regulations.

1 Scope

This standard specifies the method for the determination of silicon, manganese, phosphorus, iron, copper, cobalt, magnesium, aluminum, zinc and chromium content in refined nickel by using an inductively coupled plasma atomic emission spectrometric method.

This method is applicable to the analysis of silicon, manganese, phosphorus, iron, copper, cobalt, magnesium, aluminum, zinc and chromium content in refined nickel. The measuring range of each element is shown in Table 1.

2 Method principle

A certain amount of the sample is weighed and decomposed by the nitric acid; an inductively coupled plasma atomic emission spectrometer is used to measure the spectral intensity of the spectral line of the element to be measured in the solution; the spectral line is corrected by subtracting the background to eliminate the influence of the matrix nickel; the elemental content is obtained from the calibration curve drawn according to the standard solution.

3 Reagents

Unless otherwise stated, the reagents used in this standard are all of analytical grade, and the water used is first-grade water.

3.1 Nitric acid (ρ=1.42 g/mL).

3.2 Nitric acid (1+1).

3.3 Hydrofluoric acid (ρ=1.15 g/mL).

3.4 Silicon standard stock solution: Heat high-purity silica (the content is greater than 99.9%) at a temperature of 1100 °C until its mass is constant, and cool to room temperature in a desiccator; weigh 2.1393 g and put it into a platinum crucible, add about 5 g of sodium carbonate, heat and melt for 15 minutes. Take it out and cool it slightly, place it in a 500 mL beaker, dissolve the melt with water, and wash out the platinum crucible; then, transfer the solution into a 1000 mL volumetric flask, wash the beaker with water and pour the washing liquid into the volumetric flask, add water to the scale of the volumetric flask, mix well, and store in a plastic bottle. This solution contains 1000 μg of silicon in 1 mL.

3.5 Manganese standard stock solution: Weigh 1.0000 g of high-purity manganese metal (the content is greater than 99.9%), put it into a (300 mL) beaker with a glass cover, add 30 mL of nitric acid, and heat it carefully to decompose it. After the solution cools to room temperature, transfer it to a 1000 mL volumetric flask, clean the beaker with water, and pour the washing liquid into the volumetric flask. Add water to the scale of the volumetric flask and mix well. This solution contains 1000 μg of manganese in 1 mL.

3.6 Phosphorus standard storage solution: Weigh 4.3936 g of the standard dipotassium hydrogen phosphate dried at 105 ℃~110 ℃, dissolve it in water, transfer the solution to a 1000 mL volumetric flask, dilute it with water to the mark, and mix well. This solution contains 1000 μg of phosphorus in 1 mL.

3.7 Iron standard stock solution: Weigh 1.0000 g of high-purity iron powder (the content

is above 99.95%), add 30 mL hydrochloric acid (1+1) to dissolve it with slight heat, transfer the solution to a 1000 mL volumetric flask, dilute it with water to the mark, and shake well. 1 mL of this solution contains 1000 μg of Fe.

3.8 Copper standard stock solution: Weigh 1.0000 g of high-purity copper (the content is greater than 99.9%), put it into a 300 mL beaker, add 30 mL of nitric acid, and heat it at low temperature to decompose it. After the solution cools to room temperature, transfer it to a 1000 mL volumetric flask, dilute to volume with water, and mix well. This solution contains 1000 μg of copper in 1 mL.

3.9 Cobalt standard stock solution: Weigh 1.0000 g of high-purity cobalt (the content is greater than 99.9%), pour into a 500 mL beaker, then add 40 mL of nitric acid, and heat to completely dissolve; boil slightly to discharge nitrogen oxides, cool and pour it into a 1000 mL volumetric flask containing 160 mL of nitric acid. Dilute to volume with water and mix well. This solution contains 1000 μg of cobalt in 1 mL.

3.10 Magnesium standard stock solution: Weigh 1.0000 g of metallic magnesium (the content is above 99.9%), add 60 mL of hydrochloric acid (1+5) to dissolve, cool and transfer the solution to a 1000 mL volumetric flask, dilute to the mark with water, and shake well. This solution contains 1000 μg of magnesium in 1 mL.

3.11 Aluminum standard stock solution: Weigh 1.0000 g of pure metal aluminum (the content is above 99.9%), add 30 mL of hydrochloric acid (1+1), drop a small amount of nitric acid, heat to dissolve, and drive out the nitrogen compounds; cool and transfer to a 1000 mL volumetric flask, dilute to volume with water, and shake well. 1 mL of this solution contains 1000 μg of Al.

3.12 Zinc standard stock solution: Weigh 1.0000 g of pure metallic zinc (the content is above 99.9%), add 30 mL of hydrochloric acid (1+1) and slowly heat to dissolve, transfer the solution to a 1000 mL volumetric flask, dilute to the mark with water, and shake well. 1 mL of this solution contains 1000 μg of Zn.

3.13 Chromium standard stock solution: Weigh 1.0000 g of pure metal chromium (the content is above 99.9%), add 30 mL of hydrochloric acid (1+1) and heat to dissolve, then transfer the solution to a 1000 mL measuring bottle, dilute to the mark with water, and mix well. This solution contains 1000 μg of chromium in 1 mL.

4 Instruments

4.1 Single-scale pipettes and single-scale volumetric flasks.

4.2 Analytical balance, capable of weighing accurately to 0.0001 g.

4.3 Inductively coupled plasma atomic emission spectrometer

6 Analysis steps

6.1 Specimen

Weigh 0.2 g of the sample (Chapter 5), accurate to 0.0001 g.

6.2 Blank test

Perform a blank test along with sample analysis.

6.3 Pretreatment of specimens

Place the specimen (6.1) into a 200 mL Erlenmeyer flask, add 15 mL of nitric acid (3.2), and heat at low temperature until the specimen is completely dissolved. Remove the flask and cool to room temperature, transfer the solution into a 200 mL volumetric flask, dilute to volume with water, and mix well. This solution is the solution to be tested.

NOTE: If the content of P and Al elements in the sample is <0.01%, a 100 mL volumetric flask can be selected.

6.4 Determination

On the adjusted inductively coupled plasma spectrometer, according to the optimized working conditions of the instrument and the wavelengths given in Table 2, measure the spectral line intensity, from which the spectral line background is subtracted, of the sample solution (6.3) and blank test solution (6.2). The mass concentration of the corresponding element in the liquid to be tested is obtained on the working curve drawn by the instrument according to (6.5).

6.5 Drawing of working curve

6.5.1 Mix standard solution: Respectively pipette 60.00 mL of cobalt standard stock solution (3.9), 40.00 mL of iron standard stock solution (3.7), 20.00 mL each of silicon standard stock solution (3.4), manganese standard stock solution (3.5) and copper standard stock solution (3.8), 2.00 mL each of phosphorus standard stock solution (3.6), magnesium standard stock solution (3.10), aluminum standard stock solution (3.11), zinc standard stock solution (3.12) and chromium standard stock solution (3.13) in a 1000 mL volumetric flask, dilute with water to the mark and mix well. This solution contains 60 μg of cobalt, 40 μg of iron, 20 μg each of silicon, manganese, and copper, and 2 μg each of phosphorus, zinc, chromium, aluminum, and magnesium per milliliter.

6.5.2 Preparation of working curve series calibration solutions: Pipette 0 mL, 1.00 mL, 5.00 mL, 10.00 mL, 25.00 mL, 50.00 mL of mixed standard solutions (6.5.1) into a set of 100 mL volumetric flasks, add 7.5 mL of nitric acid (3.2), dilute to volume with water, and shake well. The concentrations of each element in the calibration curve standards

Appendix A

(Normative)

Operation of determining instrument performance specifications

A.1 Overview

The purpose of the performance tests given in this appendix is to use different types of instruments to appropriately determine the detection limit (DL) and background equivalent concentration (BEC) of the inductively coupled plasma spectrometer, and to perform appropriate spectral line background correction. Different operating conditions are allowed to be used for different models of instruments, but the final measurement results shall meet the requirements of this standard.

A.2 Detection limit of background equivalent concentration

Prepare 3 solutions, containing the concentration of the test substance at 0 concentration level, 10 times the detection limit, and 1000 times the detection limit. These solutions contain acids and matrix elements with similar concentrations to the samples to be tested.

Spray the 1000 times detection limit solution and wait 10 seconds after the solution enters the plasma to ensure stable atomization. Set operating and instrument conditions for the element to be measured.

Carefully position the selected wavelength at the highest peak and select an appropriate photomultiplier tube (if not automatically selected) to ensure that the measured light intensity has 4 significant digits. Set the integration time to 3 s.

A.2.1 Detection limit

Spray the blank solution for about 10 seconds and measure 10 times with the preset integration time.

Spray the 10 times detection limit solution for about 10 seconds and measure 10 times with the preset integration time.

According to the intensity readings obtained from the blank test solution and 10 times the detection limit solution, calculate the average value $\overline{X}_1$, $\overline{X}_b$ and the standard deviation S_b of the blank solution.

The net average intensity ($\overline{X}_{n1}$) of the 10 times detection limit solution according to

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