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NONFERROUS METALS INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

YS/T 1658.2-2023

Methods for chemical analysis of crude nickel cobalt hydroxide - Part 2: Determination of chromium, phosphorus and manganese contents - Inductively coupled plasma atomic emission spectrometry

粗氢氧化镍钴化学分析方法 第2部分：铬、磷、锰含量的测定 电感耦合等离子体原子发射光谱法

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1 Scope

This document describes the methods for the determination of the chromium, phosphorus and manganese contents of crude nickel cobalt hydroxide.

It applies to the determination of those three contents. The measuring ranges of Table 1 are: chromium from 0.0010 % to 0.10 %, phosphorus from 0.10 % to 2.00 %, and manganese from 0.050 % to 1.00 %.

2 Normative references

The contents of the following documents constitute indispensable provisions of this document through normative reference in the text. For dated references, only the edition corresponding to that date applies. For undated references, the latest edition, including all amendments, applies.

GB/T 6682 Water for analytical laboratory use - Specification and test methods · YS/T 1460-2021 Crude nickel cobalt hydroxide

3 Terms and definitions

No terms and definitions need to be defined for the purposes of this document.

4 Principle

The test portion is dissolved in hydrochloric acid. In a dilute hydrochloric acid medium the emission intensities of chromium, phosphorus and manganese are measured on an inductively coupled plasma atomic emission spectrometer. The mass concentrations of the three elements are obtained by the standard addition method, and the result is expressed as a mass fraction.

5 Reagents

Unless otherwise stated, only reagents confirmed to be of analytical grade are used.

5.1 Water complying with grade two of GB/T 6682 or better. 5.2 Hydrochloric acid (1+1).

5.3 Manganese standard stock solution: 10.0000 g of manganese metal of mass fraction not less than 99.99 % is dissolved in 20 mL of hydrochloric acid (5.2) by gentle heating in a 250 mL beaker, cooled and made up to 1 000 mL. One millilitre contains 10.00 mg of

manganese. A commercial certified solution may be used instead.

5.4 Chromium standard stock solution: 0.1000 g of chromium metal of mass fraction not less than 99.99 %, prepared in the same way and made up to 1 000 mL. One millilitre contains 100 micrograms of chromium.

5.5 Phosphorus standard stock solution: 42.6349 g of diammonium hydrogen phosphate, prepared in the same way and made up to 1 000 mL. One millilitre contains 10.00 mg of phosphorus.

5.6 Mixed manganese and phosphorus standard solution: 10.00 mL of the manganese stock solution (5.3) and 10.00 mL of the phosphorus stock solution (5.5) are made up to 100 mL with 4 mL of hydrochloric acid (5.2). One millilitre contains 1 000 micrograms of manganese and 1 000 micrograms of phosphorus.

5.7 Chromium standard solution: 10.00 mL of the chromium stock solution (5.4) is made up to 100 mL with 4 mL of hydrochloric acid (5.2). One millilitre contains 10 micrograms of chromium.

6 Apparatus

6.1 Inductively coupled plasma atomic emission spectrometer. Under the optimum working conditions of the instrument, the standard solution of the lowest concentration of each element is measured eleven times in succession and the relative standard deviation of the emission intensity shall not exceed 2.0 %.

6.2 The recommended analytical lines of Table 2 are 283.56 nm for chromium, 213.61 nm for phosphorus and 257.61 nm for manganese.

7 Samples

7.1 Sampling and sample preparation follow 7.4 of YS/T 1460-2021.

7.2 Before analysis the sample is dried at 105 degrees Celsius plus or minus 2 degrees for four hours, cooled to room temperature in a desiccator and weighed immediately.

8 Test procedure

8.1 Test portion. The sample is weighed to 0.0001 g according to Table 3: 5.00 g with 40 mL of hydrochloric acid for chromium, and 1.00 g with 20 mL of hydrochloric acid for phosphorus and manganese.

8.2 Duplicate test. Two determinations are carried out in parallel. 8.3 Blank test. A blank is run with the test portion.

8.4.1 The test portion is placed in a 300 mL beaker, moistened with a little water, treated with the corresponding volume of hydrochloric acid (5.2), covered with a watch glass and

heated gently until completely dissolved; it is then cooled to room temperature, transferred to a 100 mL volumetric flask, made up to the mark with water and mixed.

8.4.2 For a chromium mass fraction between 0.0010 % and 0.010 %, 10.00 mL of the test solution is placed in each of a set of 100 mL flasks, to which 0, 0.50, 1.00, 2.50, 5.00 and 10.00 mL of the chromium standard solution (5.7) are added in turn, each with 4 mL of hydrochloric acid, and made up to the mark. Above 0.010 % and up to 0.10 %, the additions are 0, 0.50, 1.00, 3.00, 5.00 and 6.00 mL of the chromium stock solution (5.4).

8.4.3 For manganese and phosphorus, 10.00 mL of the test solution is placed in each of a set of 100 mL flasks, to which 0, 0.05, 0.10, 0.50, 1.00, 2.00 and 3.00 mL of the mixed standard solution (5.6) are added in turn, each with 4 mL of hydrochloric acid, and made up to the mark.

8.4.4 The emission intensities of the series of standard solutions are measured at the analytical lines recommended in Table 2, and the working curve is drawn with mass concentration on the horizontal axis and emission intensity on the vertical axis. The linear correlation coefficient of the working curve shall not be less than 0.999. The curve is extrapolated backwards, and the point where it meets the horizontal axis gives the mass concentration of the element in the test solution.

9 Processing of test data

The content of the element is expressed as a mass fraction and calculated by formula (1) from the mass concentrations read on the self-calibration curve for the sample and for the blank, the volume of the test solution, the total volume to which it was made up, the mass of the test portion and the volume taken.

The result is given to two decimal places; to three decimal places when the mass fraction is below 0.10 %, and to four decimal places when it is below 0.010 %.

10 Precision

10.1 Repeatability. Under repeatability conditions the absolute difference between two independent test results shall not exceed the repeatability limit r, and the cases in which it does shall not exceed 5 %. The limit is obtained from Table 4 by linear interpolation or extrapolation: for chromium, r is 0.0003 at 0.0029 %, 0.001 at 0.010 %, 0.003 at 0.051 % and 0.005 at 0.088 %; for phosphorus, 0.01 at 0.12 %, 0.02 at 0.30 % and 0.03 at 0.76 % and 1.36 %; for manganese, 0.004 at 0.068 %, 0.02 at 0.21 % and 0.04 at 1.05 %.

10.2 Reproducibility. Under reproducibility conditions the absolute difference shall not exceed the reproducibility limit R, obtained from Table 5 in the same way: for chromium, 0.0010, 0.003, 0.005 and 0.008 at the four levels; for phosphorus, 0.03, 0.04, 0.06 and 0.11; for manganese, 0.010, 0.04 and 0.11. The raw data of the precision test are given in Annex A.

11 Test report

The test report shall state the object of the test, the number of this document, the analytical result and how it is expressed, any departure from the basic analytical procedure, any anomaly observed, and the date of the test.