

YS

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

YS/T 1658.7-2023

**Methods for chemical analysis of crude nickel cobalt hydroxide
- Part 7: Determination of manganese content - Potentiometric
titration method**

粗氢氧化镍钴化学分析方法 第7部分：锰含量的测定 电位滴定法
(English Translation)

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Foreword

This document was issued on 29 December 2023 by the Ministry of Industry and Information Technology of the People's Republic of China, and has applied since 1 July 2024.

It is the first edition of this part.

It is a YS/T standard: a recommended industry standard of China's non-ferrous metals industry, and the method a Chinese laboratory or trading partner will refer to.

Introduction

YS/T 1658, Methods for chemical analysis of crude nickel cobalt hydroxide, consists of seven parts: Part 1, nickel content by the dimethylglyoxime gravimetric method; Part 2, chromium, phosphorus and manganese contents by inductively coupled plasma atomic emission spectrometry; Part 3, fluorine content by the ion selective electrode method; Part 4, copper, aluminium, lithium, zinc, cadmium, lead and arsenic contents by inductively coupled plasma atomic emission spectrometry; Part 5, moisture content by the gravimetric method; Part 6, insoluble substance in hydrochloric acid by the gravimetric method; Part 7, manganese content by potentiometric titration.

Part 2 and Part 7 both determine manganese: Part 2 by an instrumental method together with other elements, Part 7 by titration over the range 1.00 % to 30.00 %.

1 Scope

This document describes the method for the determination of manganese content in crude nickel cobalt hydroxide.

This document is applicable to the determination of manganese content in crude nickel cobalt hydroxide. The determination range is 1.00% to 30.00%.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this document. For dated references, only the edition cited applies. For undated references, the latest edition (including any 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 are defined in this document.

4 Principle

The test portion is first decomposed by hydrochloric acid and nitric acid. In a saturated sodium pyrophosphate solution, the pH is adjusted to within the range of 6 to 7, and the solution is titrated with potassium permanganate standard volumetric solution until a potentiometric jump is reached, which is taken as the end point.

5 Reagents and materials

Unless otherwise specified, the reagents used in this document shall be of analytical pure grade or superior.

5.1

Water, Grade 3 or superior as specified in GB/T 6682.

5.2

Hydrochloric acid ($\rho=1.19$ g/mL).

5.3

Nitric acid ($\rho=1.42$ g/mL).

5.4

Sulfuric acid (1+1).

5.5

Ammonia solution (1+1).

5.6**Saturated sodium pyrophosphate solution**

Weigh 300 g of sodium pyrophosphate into a 1,000-mL beaker, and add 900 mL of water. Allow it to stand, then take the upper saturated solution for use.

5.7**Manganese standard solution**

Weigh 1.0000 g of manganese metal ($w_{\text{Mn}} \geq 99.95\%$) (to the nearest 0.0001 g) into a 400-mL beaker, add 20 mL of sulfuric acid (5.4), heat to dissolve until the solution is clear. Leave the solution to cool and transfer it into a 1,000-mL volumetric flask, then dilute to the scale with water, and shake well. This solution contains 1 mg of manganese per 1 mL.

5.8

Potassium permanganate standard stock solution [$c(1/5 \text{ KMnO}_4)$ approx. 0.1 mol/L]: Weigh 3.20 g of potassium permanganate and dissolve in 1,000 mL of water, boil for 1 h, leave it to cool and stand for 7 d. Filter through No.3 sintered glass funnel into a brown glass bottle, mix well and store in dark for later use.

5.9

Potassium permanganate standard volumetric solution [$c(1/5 \text{ KMnO}_4)$ approx. 0.01 mol/L]:

a) Preparation: Pipette 100 mL of potassium permanganate standard stock solution (5.8) into a 1,000-mL brown volumetric flask, dilute to the scale with water, mix well and store in dark for later use.

b) Standardization: Pipette three aliquots of 10.00 mL of manganese standard solution (5.7) (denoted as V_1) into 250-mL beakers separately, add 50 mL saturated sodium pyrophosphate solution (5.6). Adjust the pH of the solution to 6-7 with sulfuric acid (5.4) or ammonia solution (5.5) while stirring. On the potentiometric titrator, titrate with potassium permanganate standard volumetric solution until potential jump occurs, which serves as the end point. Record the volume of potassium permanganate standard volumetric solution consumed (denoted as V_2). The solution shall be standardized immediately before use.

Carry out three parallel standardizations. The range of the volumes of potassium permanganate standard volumetric solution consumed shall not exceed 0.05 mL, and the mean value shall be taken. Otherwise, re-standardize.

Perform a blank determination alongside the standardization, and record the volume of

potassium permanganate standard volumetric solution consumed (denoted as V_0).

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