

**YS**

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

**YS/T 1658.3-2023**

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**Methods for chemical analysis of crude nickel cobalt hydroxide  
- Part 3: Determination of fluorine content - Ion selective  
electrode method**

粗氢氧化镍钴化学分析方法 第3部分：氟离子含量的测定 离子选择性电极法  
(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 a YS/T standard: a recommended industry standard of China's non-ferrous metals industry.

### 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.

## 1 Scope

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

This document is applicable to the determination of fluorine content in crude nickel cobalt hydroxide. The determination range is 0.050%-1.00%.

## 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 decomposed by fusion with sodium hydroxide and sodium peroxide. The fusion mass is leached with water, and the fluorides are separated from iron, copper, lead, etc. by filtration (or by allowing to stand and then pipetting the supernatant). In a medium of trisodium citrate-potassium nitrate-triethanolamine at pH 6.5 to 7.0, the fluoride ion concentration in the test solution is determined using an electrode potentiometer.

## 5 Reagents

Unless otherwise specified, only reagents of guaranteed grade can be used in the analysis.

### 5.1

Water, Grade 2 or superior as defined in GB/T 6682.

### 5.2

Sodium hydroxide.

### 5.3

Sodium peroxide.

### 5.4

Nitric acid (1+4).

**5.5****Trisodium citrate-potassium nitrate solution**

Weigh 294 g of trisodium citrate dihydrate ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ ) and 20 g of potassium nitrate into a 250-mL beaker, and dissolve in water; adjust the pH to 5 to 6 with nitric acid (5.4), transfer to a 1,000-mL volumetric flask, dilute to the scale with water, and mix well.

**5.6****Triethanolamine buffer solution**

Pipette 64 mL of hydrochloric acid ( $\rho = 1.19 \text{ g/mL}$ ) and 200 mL of triethanolamine solution (1+1) into a 500-mL beaker, adjust the pH to 6.5 to 7.0 with hydrochloric acid (1+1) and ammonia solution (1+1), transfer to a 500-mL volumetric flask, dilute to the scale with water, and mix well.

**5.7****Fluoride ion standard stock solution**

Weigh 2.2110 g of sodium fluoride (primary titration reagent,  $w_{\text{NaF}} \geq 99\%$ , dried at  $105^\circ\text{C}$  for 2 h) into a 50-mL beaker, dissolve in water, transfer to a 1,000-mL volumetric flask, and mix well. Transfer the solution to a dry plastic bottle for storage. This solution contains 1 mg of fluoride ion per mL. Alternatively, a commercially available certified reference standard solution may be used.

**5.8****Fluoride ion standard solution A**

Pipette 10.00 mL of fluoride ion standard stock solution (5.7) into a 100-mL volumetric flask, dilute to the scale with water, and mix well. Transfer the solution to a dry plastic bottle. This solution contains 100  $\mu\text{g}$  of fluoride ion per mL.

**5.9****Fluoride ion standard solution B**

Pipette 10.00 mL of fluoride ion standard solution A (5.8) into a 100-mL volumetric flask, dilute to the scale with water, and mix well. Transfer the solution to a dry plastic bottle. This solution contains 10  $\mu\text{g}$  of fluoride ion per mL.

**5.10****Phenol red solution (2 g/L)**

Weigh 0.1 g of phenol red into a 50-mL beaker, add 6 mL of NaOH solution (2 g/L), dilute to 50 mL with water, and mix well.