

Foreword

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This document is drafted in accordance with the rules given in *GB/T 1.1—2020 Directive for standardization—Part 1: Rules for structure and drafting of standardization documents*.

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This document was proposed and prepared by SAC/TC 243 National Nonferrous Metals Standardization Technical Committee.

Crude nickel cobalt hydroxide

1 Scope

This document specifies the technical requirements, test methods, inspection rules and marking, packaging, transportation, storage, accompanying documents and purchase order of crude nickel cobalt hydroxide.

This document is applicable to the crude nickel cobalt hydroxide products obtained from lithium-ion battery wastes containing nickel and cobalt through pretreatment, leaching, impurity removal, precipitation and other hydrometallurgy enrichment processes, and these products can be used as raw materials for producing nickel-cobalt-manganese composite hydroxides, lithium nickel cobalt manganese oxide, chemical salts of nickel or cobalt and other related materials.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682, *Water for analytical laboratory use—Specification and test methods*

GB/T 8170, *Rules of rounding off for numerical values & expression and judgement of limiting values*

YS/T 1157.2, *Methods for chemical analysis of crude cobalt hydroxide—Part 2: Determination of nickel, copper, iron, manganese, zinc, lead, arsenic and cadmium contents—Inductively coupled plasma atomic emission spectrometry*

YS/T 1229.2, *Methods for chemical analysis of crude nickel hydroxide—Part 2: determination of cobalt content—Flame atomic absorption spectrometric method*

YS/T 1229.3, *Methods for chemical analysis of crude nickel hydroxide—Part 3: Determination of copper, cobalt, manganese, calcium, magnesium, zinc, iron, aluminum, lead, arsenic and cadmium contents—Inductively coupled plasma atomic emission spectrometry*

YS/T 1342.1, *Methods for chemical analysis of waste secondary battery—Part 1: Determination of nickel content—Dimethylglyoxime gravimetric method and flame atomic absorption spectrometry*

YS/T 1342.2, *Methods for chemical analysis of waste secondary battery—Part 2: Determination of cobalt content—Potentiometric titration method and flame atomic absorption spectrometry*

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YS/T 1342.3, *Methods for chemical analysis of waste secondary battery—Part 3: Determination of manganese content—Potentiometric titration method and flame atomic absorption spectrometry*

YS/T 1342.4, *Methods for chemical analysis of waste secondary battery—Part 4: Determination of lithium content—Flame atomic absorption spectrometry*

3 Terms and definitions

No terms and definitions are listed in this document.

4 Product classification

Products are classified into three grades on their chemical composition.

5 Technical requirements

5.1 Chemical composition

The chemical composition of product (dry basis) shall comply with the requirements given in Table 1.

Table 1 Chemical composition of product (dry basis)

Grade		Grade 1	Grade 2	Grade 3	
Chemical composition (mass fraction) /%	Main element, min.	Nickel + Cobalt (Ni + Co)	40.0	35.0	25.0
	Impurity elements, max.	Manganese (Mn)	10.0	20.0	25.0
		Copper (Cu)	0.5	1.0	2.0
		Aluminum (Al)	0.5	1.0	2.0
		Lithium (Li)	0.5	1.0	2.0
		Arsenic (As)	0.01	0.01	0.03
		Cadmium (Cd)	0.005	0.005	0.01
		Chromium (Cr)	0.005	0.005	0.01
		Lead (Pb)	0.005	0.005	0.01
		Fluorine (F)	0.5		
		Phosphorus (P)	1.0		
Hydrochloric acid insoluble matter, max.	1.0				

5.2 Moisture

The moisture content of the product shall not be more than 60%.

5.3 Appearance quality

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The product shall be wet block, dry mud or powder and without inclusions. The color of the same batch of products shall be consistent. A certain difference in color between the surface and inside is allowed, attributed to the oxidation on the surface.

5.4 Others

Once the buyer has other requirements for the product, it shall be agreed by the supplier and buyer through negotiation and indicated in the purchase order.

6 Test methods

6.1 When the nickel content is less than 1.00% in the product, it shall be determined as specified in YS/T 1157.2, and when the nickel content is not less than 1.00%, it shall be determined as specified in YS/T 1342.1.

6.2 When the cobalt content is less than 1.00% in the product, it shall be determined as specified in YS/T 1229.2, and when the cobalt content is not less than 1.00%, it shall be determined as specified in YS/T 1342.2.

6.3 When the manganese content is less than 0.10% in the product, it shall be determined as specified in YS/T 1229.3, and when the manganese content is not less than 0.10%, it shall be determined as specified in YS/T 1342.3.

6.4 The lithium content in the product shall be determined as specified in YS/T 1342.4.

6.5 The copper, aluminum, arsenic, cadmium and lead contents in the product shall be determined as specified in YS/T 1229.3.

6.6 The phosphorus and chromium contents in the product shall be determined in accordance with the rules given in annex A or the method negotiated by the supplier and buyer.

6.7 The hydrochloric acid insoluble matter content in the product shall be determined in accordance with the rules given in annex B or the method negotiated by the supplier and buyer.

6.8 The fluorine content in the product shall be determined in accordance with the rules given in annex C or the method negotiated by the supplier and buyer.

6.9 The moisture content in the product shall be determined in accordance with the rules given in annex D or the method negotiated by the supplier and buyer.

6.10 The appearance quality of product shall be inspected visually.

7 Inspection rules

7.1 Inspection and acceptance

7.1.1 The products shall be inspected by the supplier or the third-party quality inspection department. The supplier shall guarantee the quality of the products to comply with the provisions of this document and purchase order.

7.1.2 The buyer can inspect the received products according to the provisions of this document. If the inspection results do not comply with the provisions of this document and purchase order, the buyer shall inform the supplier within 30 days from the date of receipt of the products, which shall be settled by the supplier and buyer through negotiations. In case of any arbitration, samples shall be determined by the supplier and buyer through joint sampling at the buyer or negotiation.

7.2 Batching

The products shall be submitted for inspection in batches, and each batch shall consist of the same grade of products. The same mixture of products forms a batch.

7.3 Inspection items

Each batch of products shall be inspected for chemical composition, moisture and appearance quality.

7.4 Sampling and sample preparation

7.4.1 Each batch of products shall be randomly sampled and sampling quantity should not be less than 50% of the number of packages. Each bag shall be sampled when the number of packages is less than 10 bags. The sampling quantity of each bag shall not be less than 0.4% of total products quantity.

7.4.2 Sample from three points at the top, middle and bottom equally located in any diagonal line of the package bag side. Sample drill rod shall penetrate the package bag, rotate 180 degrees and pull it out. And ensure full loading of sample drill rod. The hammer can be used to assist sampling when materials are not easy to sample. Each drill rod sample shall be put in plastic bag and sealed in time.

7.4.3 The whole batch of samples are put in woven bags and sealed. All samples in each batch shall be fully mixed, and nearest 4.0 kg of sample shall be divided by grid method.

7.4.4 Samples are equally divided into 3 parts, which are used for moisture content determination according to the provision of annex D. Samples with moisture content difference within 0.5% can be used for chemical composition determination, otherwise, the samples need to be redivided by grid method. All samples are ground and pass through a standard sieve of 0.150 mm. Evenly mix and take five samples separately, and each sample is not less than 200 g.

7.4.5 Five samples are put in clean airtight containers with labels (indicating serial number, grade, place of origin, sampling and name of the person responsible for sample preparation, sampling date and analysis items), one for acceptance analysis, one for buyer, one for supplier, one signed and confirmed by both parties onsite for arbitration and one for standby. The arbitration sample shall be kept by the buyer for three months (six months for international

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trade). Disagreement between the supplier and buyer on the inspection results should be declared within the retention period of the arbitration sample.

7.5 Inspection results judgement

7.5.1 The inspection result shall be rounded as specified in GB/T 8170 and judged by the rounding value comparison method.

7.5.2 The batch of products shall be judged as unqualified if any of the chemical composition, moisture or appearance quality of products is not in conformity with this document.

8 Marking, packaging, transportation, storage and accompanying documents

8.1 Marking

The outer packaging of products shall be accompanied by product name, batch number, net weight, the supplier name, factory address, "rainproof" sign, etc.

8.2 Packaging

Products are packed in woven bags lined with aluminum plastic bags or PE bags and sealed. And the net weight of each bag is 0.8 t~1.2 t. It can also be packaged according to the specifications required by buyer.

8.3 Transportation

Products shall be handled carefully during transportation to prevent the package from breaking, and take measures to prevent rain, water, etc.

8.4 Storage

Products shall be stored in dry, ventilated and non-corrosive warehouses. Products shall not be mixed with acid, alkali, oil and other chemicals. And products shall be prevented from rain, corrosion, moisture, etc.

8.5 Accompanying documents

Each batch of products shall have accompanying documents, which shall include supplier information, product information, this document number, production date or packaging date, and should also include:

- a) Product quality guarantee:
 - Main performance and technical parameters of product;
 - Product characteristics (including manufacturing process and raw material characteristics);
 - Product quality responsibility;

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- The quality certification report obtained by the product and the analysis inspection results stamped by the technical supervision department of the supplier.
- b) Quality certification:
 - Inspection items and results or inspection conclusion;
 - Batch weight or batch number;
 - Inspection date;
 - Signature or stamp of inspector.
- c) Inspection report in the process of product quality control and inspection report for finished product;
- d) Product instructions: correct handling, use and storage methods, etc.;
- e) Others.

9 Purchase order content

The buyer may, according to their own needs, list the following contents in the purchase order form for the products listed in this document:

- a) Product name;
- b) Grade;
- c) Chemical composition (special requirements);
- d) Net weight;
- e) This document number;
- f) Others.

Annex A

(Normative)

Crude nickel cobalt hydroxide – Determination of phosphorus and chromium
– Inductively coupled plasma atomic emission spectrometry

A.1 Principle

Dissolve the test portion with hydrochloric acid. Determine the content of phosphorus by working curve method and the content of chromium by standard addition method on inductively coupled plasma atomic emission spectrometer (ICP-AES).

A.2 Reagents and materials

Unless otherwise specifies, this annex use only guaranteed reagents and only grade 1 water as specified in GB/T 6682.

A.2.1 Hydrochloric acid ($\rho=1.19$ g/mL).

A.2.2 Hydrochloric acid, diluted 1+1.

A.2.3 Phosphorus standard storage solution: weigh 0.4390 g of potassium dihydrogen phosphate [$\omega(\text{KH}_2\text{PO}_4) \geq 99.99\%$] in a 100 mL beaker, dissolve with water, transfer to a 100 mL volumetric flask, dilute to the mark with water and mix well. 1 mL of this standard solution contains 1 mg of phosphorus.

A.2.4 Chromium standard storage solution: weigh 0.2829 g potassium dichromate [$\omega(\text{K}_2\text{Cr}_2\text{O}_7) \geq 99.99\%$] in a 100 mL beaker, dissolve with water, Transfer into a 100 mL volumetric flask, dilute to the mark with water and mix well. 1 mL of this standard solution contains 1 mg of chromium.

A.2.5 Phosphorus standard solution: transfer 20.00 mL of phosphorus standard storage solution (A.2.3) to a 100 mL volumetric flask, add 10 mL of hydrochloric acid (A.2.2), dilute to the mark with water and mix well. 1 mL of this standard solution contains 200 μg of phosphonium.

A.2.6 Chromium standard solution A: transfer 10.00 mL of chromium standard storage solution (A.2.4) into a 200 mL volumetric flask, add 20 mL hydrochloric acid (A.2.2), dilute to the mark with water and mix well. 1 mL of this standard solution contains 50 μg of chromium.

A.2.7 Chromium standard solution B: transfer 10.00 mL of chromium standard storage solution (A.2.4) into a 100 mL volumetric flask, add 10 mL of hydrochloric acid (A.2.2), dilute to the mark with water and mix well. 1 mL of this standard solution contains 5 μg of chromium.

A.3 Apparatus

Inductively coupled plasma atomic emission spectrometer.

— Resolution: the optical resolution at 200 nm is not more than 0.010 nm; Optical resolution at 400 nm is not more than 0.020 nm.

— Instrument stability: the relative standard deviation of emission intensity is no more than 2.0% by using 1.0 µg/mL standard solution of phosphorus and chromium measured for 11 times.

Table A.1 Recommended analytical lines for elements

Element	the wavelength nm
P	178.221
Cr	283.563

A.4 Test procedures

A.4.1 Test portions

According to Table A.2, weigh the test sample (7.4.5) to the nearest 0.0001 g.

Table A.2 Sample quantity, beaker specification and hydrochloric acid volume table

Element to be determined	Quantity of sample material g	Beaker size mL	The volume of hydrochloric acid (A.2.2) mL
P	0.25	100	25
Cr	5.00	250	50

A.4.2 Parallel test

Carry out two tests in parallel and take the average value.

A.4.3 Blank test

Carry out a blank test with the test sample.

A.4.4 Test solution preparation

According to table A.2, place the test portion (A.4.1) in the corresponding beaker. Moisten with a small amount of water. Add hydrochloric acid (A.2.2) and cover with watch glass. Heat at low temperature until the sample is completely dissolved, and cool to room temperature. Transfer to a 250 mL volumetric flask. Dilute to the mark with water. Mix well and filter with quick qualitative filter paper.

A.4.5 Determination

A.4.5.1 Phosphorus content determination

Determine the excitation intensity of phosphorus in the blank test solution (A.4.3) and the test solution (A.4.4) by ICP-AES according to the wavelength recommended in Table A.1, and

find out the mass concentration of phosphorus in the blank reagent and the test solution from the working curve.

A.4.5.2 Chromium content determination

Transfer 25.00 mL of sample solution (A.4.4) to a series of 50 mL volumetric flasks. Add 0 mL, 1.00 mL, 2.00 mL, 3.00 mL and 4.00 mL of chromium standard solution in turn (choose chromium standard solution B (A.2.7) when the chromium content is not more than 0.0050%. Choose chromium standard solution A (A.2.6) when the chromium content is more than 0.0050%). Add 5 mL of hydrochloric acid (A.2.2) to each solution separately. Dilute to the mark with water, and mix well.

According to the wavelength recommended in Table A.1, from low to high, determine the excitation intensity of chromium in the above-mentioned solution series by ICP-AES. Draw the calibration curve of chromium with the mass concentration of chromium added in the test solution as the abscissa and the corresponding excitation intensity as the ordinate. The correlation coefficient of the calibration curve shall be more than 0.995. The reverse extension line of the calibration curve intersects with the abscissa, and the intersection point is the mass concentration of chromium in the test solution.

A.4.6 Phosphorus working curve drawing

Transfer 0.50 mL, 1.00 mL, 2.50 mL, 5.00 mL and 10.00 mL of phosphorus standard solution (A.2.5) to a series of 100 mL volumetric flasks. Add 10 mL of hydrochloric acid (A.2.2) separately. Dilute with water to the mark and mix well. According to the wavelength recommended in table A.1, from low to high, determine the excitation intensity of phosphorus in standard solution series by ICP-AES. Draw the calibration curve of phosphorus, with the mass concentration of phosphorus as the abscissa and the corresponding excitation intensity as the ordinate, and the correlation coefficient of the working curve shall be more than 0.999.

A.5 Test-data processing

A.5.1 Phosphorus test-data processing

The phosphorus content is expressed as the mass fraction of phosphorus (ω_p), given by the formula (A.1):

$$\omega_p = \frac{(\rho_2 - \rho_1) \cdot V_1 \times 10^{-6}}{m_1} \times 100\% \quad (\text{A.1})$$

Where:

ρ_2 is the mass concentration, in $\mu\text{g/mL}$, of phosphorus in the test solution found out from the standard curve;

ρ_1 is the mass concentration, in $\mu\text{g/mL}$, of phosphorus in the blank solution found out from the standard curve;

V_1 is the volume, in mL, of constant volume of test solution, in mL;

m_1 is the mass, in g, of the weighed test.

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The calculation result shall be expressed to two significant figures after the decimal point.

A.5.2 Chromium test-data processing

The chromium content is expressed as the mass fraction of chromium(ω_{Cr}), given by the formula (A.2):

$$\omega_{Cr} = \frac{\rho_3 \cdot V_2 \cdot V_4 \times 10^{-6}}{m_2 \cdot V_3} \times 100\% \quad (\text{A.2})$$

Where:

ρ_3 is the mass concentration, in $\mu\text{g/mL}$, of chromium in found out from the standard curve of the test solution;

V_2 is the volume, in mL, of constant volume of test solution;

V_4 is the volume, in mL, of constant volume of respectively transferred test solution;

m_2 is the mass, in g, of the weighed test portion;

V_3 is the volume, in mL, of transferred test solution.

The calculation result shall be expressed to two significant figures after the decimal point.

Annex B

(Normative)

Crude nickel cobalt hydroxide – Determination of hydrochloric acid insoluble matter content – Gravimetric method

B.1 Principle

Dissolve the test sample with hydrochloric acid, filter with a glass sand core crucible. Wash with water. Place in an electric thermostatic drying oven at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. Dry to constant weight, and calculate the content of hydrochloric acid insoluble matter.

B.2 Reagents

Unless otherwise specifies, use only analytical pure reagents and above and only grade 3 water and above as specified in GB/T 6682.

B.2.1 Hydrochloric acid ($\rho=1.19\text{ g/mL}$).

B.2.2 Hydrochloric acid, diluted 1+1.

B.2.3 Silver nitrate solution: 10 g/L.

B.3 Apparatus

B.3.1 Electrothermal constant temperature drying oven: capable of controlling temperature at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

B.3.2 Glass sand core crucible: the aperture of filter plate is $5\text{ }\mu\text{m}\sim 15\text{ }\mu\text{m}$.

B.4 Test procedures

B.4.1 Test portion

Weigh 10.0 g of test sample (7.4.5) to the nearest 0.0001 g.

B.4.2 Parallel test

Carry out two tests in parallel and take the average value.

B.4.3 Determination

Place the test portion (B.4.1) in a 500 mL beaker. Moisten with a small amount of water, add 60 mL of hydrochloric acid (B.2.2), and boil at low temperature for $5\text{ min}\sim 10\text{ min}$. Use a glass sand core crucible (B.3.2) with constant weight for suction filtration immediately while the solution is hot, and wash with hot water until there is no chloride ion (inspect with silver nitrate solution). Place the glass sand core crucible containing hydrochloric acid insoluble matter in an electric thermostatic drying oven (B.3.1) at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ to dry for 1 h, take

out and put into a dryer to cool to room temperature, and weigh. Dry repeatedly to constant weight.

B.5 Test-data processing

The content of hydrochloric acid insoluble matter is expressed as the mass fraction acid insoluble matter (ω), given by the formula (B.1).

$$\omega = \frac{m_8 - m_7}{m_6} \times 100\% \quad (\text{B.1})$$

Where:

m_8 is the total mass, in g, of hydrochloric acid insoluble matter and glass sand core crucible after drying;

m_7 is the mass, in g, of the crucible;

m_6 is the mass, in g, of the weighed test portion.

The calculation result shall be expressed to two significant figures after the decimal point.

Annex C

(Normative)

Crude nickel cobalt hydroxide – Determination of fluorine content – Ion selective electrode method

C.1 Principle

Melt the test sample with sodium hydroxide. Extract with water. Use sodium citrate as total ionic strength regulator. Adopt working curve method. Determine the content of fluorine by fluoride ion selective electrode.

C.2 Reagents

Unless otherwise specifies, use only analytical pure reagents and above and only grade 3 water and above as specified in GB/T 6682.

C.2.1 Sodium hydroxide: guaranteed reagent.

C.2.2 Sodium fluoride: guaranteed reagent.

C.2.3 Nitric acid ($\rho=1.42$ g/mL).

C.2.4 Nitric acid, diluted 1+4.

C.2.5 Sodium citrate solution: weigh 294 g of sodium citrate. Put in a 500 mL beaker. Add water to dissolve. Transfer into a 1000 mL volumetric flask. Dilute to the mark with water, and mix well.

C.2.6 Fluorine standard storage solution: weigh 2.211 0 g of sodium fluoride (C.2.2) dried at 120°C for 2 h, dissolve with water, transfer to a 1000 mL volumetric flask, dilute to the mark with water, mix well, and store in a plastic bottle. 1 mL of this standard solution contains 1 mg of fluorine.

C.2.7 Fluorine standard solution A: transfer 10.00 mL of fluorine standard storage solution (C.2.6) to 100 mL volumetric flask, dilute to the mark with water, mix well, and store in a plastic bottle. 1 mL of this standard solution contains 100 μ g of fluorine.

C.2.8 Fluorine standard solution B: transfer 20.00 mL of fluorine standard solution A (C.2.7) to a 100 mL volumetric flask, dilute to the mark with water, mix well and store in a plastic bottle. 1 mL of this standard solution contains 20 μ g of fluorine.

C.2.9 Phenol red indicator (2 g/L): weigh 0.2 g of phenol red to a 100 mL volumetric flask, add 12 mL of sodium hydroxide solution (2 g/L), dilute to the mark with water and mix well.

C.3 Apparatus

C.3.1 Nickel crucible (30 mL).

C.3.2 High temperature furnace: the temperature can be controlled at 600 °C \pm 2 °C.

C.3.3 Fluorine ion selective electrode.

C. 3. 4 Saturated calomel electrode.

C. 3. 5 Magnetic stirrer.

C. 3. 6 Potentiometer: the accuracy is 0.1 mV.

C. 4 Test procedures

C. 4. 1 Test portion

Weigh 0.50 g of test sample (7.4.5) to the nearest 0.0001 g.

C. 4. 2 Parallel test

Carry out two tests in parallel and take the average value.

C. 4. 3 Blank test

Carry out a blank test with the test sample.

C. 4. 4 Determination

C. 4. 4. 1 Place the sample (C. 4. 1) in a nickel crucible (C. 3. 1). Add 4 g of sodium hydroxide (C. 2. 1). Heat to melt in an electric furnace and mix well. Place in a high-temperature furnace (C. 3. 2) which has been raised to 600 °C for 20 min. Take out. Shake the melt evenly on the inner wall of the crucible, and cool slightly.

C. 4. 4. 2 Put the crucible and the melt in a 250 mL beaker containing 100 mL hot water. Cover with a watch glass. Heat and leach the melt. Wash out the crucible with water. Cool to room temperature. Transfer into a 250 mL volumetric flask. Dilute to the mark with water. Mix well. Filter with quick qualitative filter paper.

C. 4. 4. 3 Transfer 10.00 mL of test solution (C. 4. 4. 2) to a 100 mL volumetric flask. Add 20 mL of sodium citrate solution (C. 2. 5) and 2 drops of phenol red indicator (C. 2. 9). Adjust the solution to just turn yellow with nitric acid (C. 2. 4). Dilute to the mark with water and mix well.

C. 4. 4. 4 Pour all the test solution into a dry 250 mL beaker. Put a stirrer to it. Insert the fluorine ion selective electrode (C. 3. 3) and the saturated calomel electrode (C. 3. 4). Stir on an electromagnetic stirrer (C. 3. 5). Determine the equilibrium potential value of the test solution on a potentiometer (C. 3. 6).

C. 4. 5 Working curve drawing

Transfer 0.50 mL, 1.00 mL, 2.50 mL, 5.00 mL, and 10.00 mL of fluorine standard solution B (C. 2. 8) to a series of 100 mL volumetric flasks. Add 20 mL of sodium citrate (C. 2. 5) and 2 drops of Phenol red indicator (C. 2. 9). Adjust the solution to just turn yellow with nitric acid (C. 2. 4). Dilute to the mark with water and mix well. Pour all the test solution into a dry 250 mL beaker. Put a stirrer. Insert the fluorine ion selective electrode (C. 3. 3) and the saturated calomel electrode (C. 3. 4). Stir on an electromagnetic stirrer (C. 3. 5). According to the mass concentration of fluorine, from low to high, simultaneously determine the equilibrium

potential value of the test solution and the test portion by the potentiometer (C.3.6). Draw the working curve of E_x (mV) - $\lg C_F$ ($\mu\text{g/mL}$) with the logarithm of fluorine mass concentration as abscissa and the corresponding potential value as ordinate.

C.5 Test-data processing

The content of fluorine is expressed as the mass fraction (ω_F), given by the formula (C.1) :

$$\omega_F = \frac{(\rho_5 - \rho_4) \cdot V_5 \cdot V_7 \times 10^{-6}}{m_9 \cdot V_6} \times 100\% \quad (\text{C.1})$$

Where:

ρ_5 is the mass concentration, in $\mu\text{g/mL}$, of fluorine found out from sample working curve of the test solution;

ρ_4 is the mass concentration, in $\mu\text{g/mL}$, of fluorine found out from working curve of the blank solution;

V_5 is the volume, in mL, of constant volume of test solution;

V_7 is the volume, in mL, of constant volume of transferred test solution;

m_9 is the mass, in g, of the weighed test portion;

V_6 is the volume, in mL, of transferred test solution.

The calculation result shall be expressed to two significant figures after the decimal point.

Annex D

(Normative)

Crude nickel cobalt hydroxide – Determination of moisture content – Oven drying method

D.1 Abstract

Place the sample in an electric thermostatic drying oven at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. Dry to constant weight. Determine the mass loss and calculate the moisture content.

D.2 Apparatus

D.2.1 Electric thermostatic drying oven: the temperature can be controlled at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

D.2.2 Electronic balance: the accuracy is 0.1 g.

D.2.3 Sample tray: stainless steel metal tray or enamel tray with the surface smooth and clean.

D.3 Test procedures

D.3.1 Parallel test

Carry out three tests in parallel and take the average value.

D.3.2 Determination

Weigh about 1300 g of sample (7.4.3) with a sample tray (m_3) which has been dried to constant weight at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ to the nearest 0.1 g. Spread the sample on the bottom of the tray and weigh (m_4). Put in the electric thermostatic drying oven (D.2.1) at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ to dry for not less than 24 h, take out and weigh while it is hot. Then put in the electric thermostatic drying oven (D.2.1) again. Dry for 2 hours, take out, and weigh while hot. Repeat the operation until the weight is constant (the difference between the two weighing values is not more than 0.05% of the initial mass). Record the last weighing mass (m_5).

D.4 Test-data processing

The moisture content is expressed as its mass fraction ($\omega_{\text{H}_2\text{O}}$), given by the formula (D.1).

$$\omega_{\text{H}_2\text{O}} = \frac{m_4 - m_5}{m_4 - m_3} \times 100\% \quad (\text{D.1})$$

Where:

m_4 is the total mass, in g, of sample and sample tray before drying;

m_5 is the total mass, in g, of sample and sample tray after drying;

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m_3 is the mass, in g, of the sample tray;

The calculation result shall be expressed to two significant figures after the decimal point.

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