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Refined Nickel Sulfates

精制硫酸镍

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Standardization Administration of the People's Republic of China.

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Refined Nickel Sulfates

Warning: This product is listed in the *Catalogue of Hazardous Chemicals* (2015 Version) and is a hazardous chemical. Be careful when handling it. People who use this Document must have practical experience working in formal laboratory. This Document does not point out all possible safety issues. Users are responsible for taking appropriate safety and health measures, ensuring that the conditions specified in relevant national laws and regulations are met. The perchloroethylene used in the test method has irritating and anesthetic effects. When using it, it must be strictly followed in accordance with its safety technical instructions. Some reagents are corrosive. If splashed on the skin or eyes, rinse immediately with water. In severe cases, seek medical attention immediately.

1 Scope

This Document specifies the classification, technical requirements, test methods, inspection rules, marking and accompanying documents, packaging, transportation and storage of refined nickel sulfates.

This Document applies to the production and inspection of refined nickel sulfates.

NOTE: The product is mainly used in the battery industry, electroplating industry, oil hydrogenation catalyst, pharmaceutical industry catalyst, mordant for reducing dyes, metal colorant and the production of other industrial nickel salts.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 191-2008 Packaging - Pictorial Marking for Handling of Goods

GB/T 6678 General principles for sampling chemical products

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

HG/T 3696.1 Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis - Part 1: Preparations of standard volumetric solutions

HG/T 3696.2 Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis - Part 2: Preparations of standard solutions for impurity

HG/T 3696.3 Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis - Part 3: Preparations of reagent solutions

JJG 768 Verification Regulation of Emission Spectrometer

3 Terms and Definitions

For the purposes of this Document, there are no terms and definitions need to be given.

4 Molecular Formula and Relative Molecular Mass

Molecular formula: $NiSO_4 \cdot 6H_2O$.

Relative molecular mass: 262.86 (according to the international relative atomic mass in 2022).

5 Classification

Refined nickel sulfate is divided into the following categories according to its use:

--- Class I: for battery industry;

--- Class II: for electroplating and other industrial uses.

6 Technical Requirements

6.1 The appearance is emerald green granular crystals.

6.2 Refined nickel sulfates shall comply with the technical requirements in Table 1 when tested according to the test methods specified in this Document.

m_0 - mass of precipitate and sintered glass crucible, in g;

m_1 - mass of sintered glass crucible, in g;

m - mass of specimen, in g;

0.203 1 - coefficient of converting nickel dimethylglyoxime to nickel.

The arithmetic mean of the results of two parallel determinations is taken as the determination result, and the absolute difference of the results of two parallel determinations is no greater than 0.05%.

7.3.2 Complexometric titration

7.3.2.1 Principle

The test material is decomposed with acid, and impurities such as copper, iron, calcium, and magnesium are masked with potassium sodium tartrate, ammonium fluoride, and sodium thiosulfate. In an ammonia solution with a pH of 8 to 9, murexide is used as an indicator and EDTA standard titration solution is used to titrate until the solution turns purple-red.

7.3.2.2 Reagents or materials

7.3.2.2.1 Ammonium fluoride.

7.3.2.2.2 Hydrochloric acid solution: 1 + 1.

7.3.2.2.3 Ammonia solution: 1 + 1.

7.3.2.2.4 Nitric acid solution: 1 + 1.

7.3.2.2.5 Potassium sodium tartrate solution: 150 g/L.

7.3.2.2.6 Sodium thiosulfate solution: 500 g/L.

7.3.2.2.7 Disodium ethylenediaminetetraacetic acid (EDTA) standard titration solution: c (EDTA) ≈ 0.02 mol/L.

7.3.2.2.8 Murexide indicator: 1 g of murexide and 100 g of sodium chloride dried at 105 °C ~ 110 °C for 2 h are mixed and ground in a mortar.

7.3.2.3 Test steps

7.3.2.3.1 Preparation of test solution

Weigh about 1.0 g of the specimen, accurate to 0.000 1 g; and place it in a 250 mL beaker. Add 50 mL of water and 2 mL of hydrochloric acid solution; heat to dissolve. Cool to room temperature and then transfer completely to a 250 mL volumetric flask; dilute to the mark with

In nitric acid medium, the standard curve method is used to determine the content of the elements to be measured using an inductively coupled plasma emission spectrometer.

7.4.2 Reagents or materials

7.4.2.1 Nitric acid solution: 1 +1, prepared with guaranteed reagent of nitric acid.

7.4.2.2 Nickel matrix solution: ρ (Ni) = 20 g/L (the mass fraction of cobalt, copper, iron, sodium, lead, zinc, calcium, magnesium, manganese, cadmium and chromium is no more than 0.000 2%). Weigh about (20.0±0.1)g of metallic nickel (with the mass fraction of nickel no less than 99.99%) and place it in a 400mL beaker. Add a small amount of water to moisten it; slowly add 150mL of nitric acid solution; and heat until it is completely dissolved. After cooling to room temperature, transfer it to a 1000mL volumetric flask; dilute it to the mark with water, and shake it well.

7.4.2.3 Mixed standard solution: 1mL of solution contains 0.01mg of cobalt (Co), copper (Cu), iron (Fe), sodium (Na), lead (Pb), zinc (Zn), calcium (Ca), magnesium (Mg), manganese (Mn), cadmium (Cd), and chromium (Cr). Use a pipette to respectively take 1 mL of the standard stock solution of cobalt, copper, iron, sodium, lead, zinc, calcium, magnesium, manganese, cadmium and chromium prepared according to HG/T 3696.2; place it in a 100 mL volumetric flask; dilute it to the mark with water; and shake it well. The solution shall be immediately prepared before use.

7.4.2.4 Water: Grade-2 water in accordance with GB/T 6682.

7.4.3 Instruments and equipment

Inductively coupled plasma emission spectrometer: It shall meet the requirements of JJG 768.

7.4.4 Test steps

7.4.4.1 Drawing of standard curve

Pipette 0.00 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL, and 10.00 mL of the mixed standard solution, respectively and place them in seven 100 mL volumetric flasks. Then add 10 mL of nickel matrix solution and 2 mL of nitric acid solution, respectively; dilute to the scale with water; and shake well.

Adjust the inductively coupled plasma emission spectrometer to the optimal working conditions; and measure the spectral intensity of the standard solution at the recommended measurement wavelength of each element to be measured in Table 2. With the mass concentration (mg/L) of the element to be measured in each standard solution as the horizontal axis and the corresponding spectral intensity as the vertical axis, draw the standard curve of each element to be measured.

m - mass of the test material, in g.

The arithmetic mean of the results of two parallel determinations is taken as the determination result; and the absolute difference between the results of two parallel determinations is no greater than 10% of the arithmetic mean.

7.5 Determination of mercury content

7.5.1 Principle

Dissolve the specimen in nitric acid; reduce the divalent mercury ions to mercury atoms with potassium borohydride (or sodium borohydride solution) in an acidic solution; introduce high-purity argon gas into the atomic fluorescence absorption cell, and determine by atomic fluorescence method.

7.5.2 Reagents or materials

7.5.2.1 Nitric acid solution: 1 + 9.

7.5.2.2 Nitric acid solution: 5 + 95.

7.5.2.3 Potassium borohydride solution: 5 g/L; weigh 5.0 g of potassium borohydride; dissolve it with potassium hydroxide solution (5 g/L) and dilute it to 1 000 mL; mix well. Prepare it before use.

7.5.2.4 Sodium borohydride solution: 3.5 g/L; weigh 3.5 g of sodium borohydride; dissolve it with sodium hydroxide solution (3.5 g/L) and dilute it to 1 000 mL; mix well. Prepare it before use.

7.5.2.5 Potassium dichromate-nitric acid solution: 0.5 g/L; weigh 0.5 g of potassium dichromate; dissolve it with nitric acid solution (5 +95) and dilute it to 1 000 mL; mix well.

7.5.2.6 Mercury standard stock solution: 1 mL of solution contains 0.01 mg of mercury (Hg). Pipette 1.00 mL of the mercury standard stock solution prepared according to HG/T 3696.2 into a 100 mL volumetric flask; dilute it to the mark with potassium dichromate-nitric acid solution; and shake well.

7.5.2.7 Mercury standard solution: 1 mL of solution contains 0.05 μg of mercury (Hg). Pipette 0.50 mL of the stock solution of mercury standard solution into a 100 mL volumetric flask; dilute to the mark with potassium dichromate-nitric acid solution; and shake well. This solution is prepared before use.

7.5.3 Instruments

Atomic fluorescence spectrometer: equipped with a mercury hollow cathode lamp. Detection limit: ≤0.01 μg/L; Temperature: 10 °C ~30 °C.

result; and the absolute difference between the results of two parallel determinations is no greater than 0.001%.

7.7 Determination of oil content

7.7.1 Reagents or materials

7.7.1.1 Tetrachloroethylene ($\rho = 1.62$ g/cm^3).

7.7.1.2 Anhydrous sodium sulfate.

7.7.2 Instruments and equipment

Infrared spectrophotometer for oil content: Range is 0 mg/L~80 mg/L (with 4 cm cuvette); detection limit is 0.02 mg/L.

7.7.3 Test steps

Weigh about 40 g of the specimen, accurate to 0.01 g; and place it in a 200 mL beaker. Add 20 mL of water; boil it to dissolve it; and transfer it all to a separatory funnel after cooling. Use a pipette to transfer 20 mL of tetrachloroethylene; extract it for 2 min; let it stand for stratification; and pour it all into a dehydration column added 1 cm~2 cm thick anhydrous sodium sulfate; dehydrate it to a 25 mL colorimetric tube; cover it with a lid and set aside.

Adjust the infrared spectrophotometer for oil to the optimal working conditions; measure according to the instrument operation process; input the specimen mass and the accurate volume of tetrachloroethylene. Transfer the test solution to the colorimetric dish; put it into the instrument for measurement; and read the oil content in the specimen directly from the instrument.

Take the arithmetic mean of the results of two parallel measurements as the measurement result; and the absolute difference between the results of two parallel measurements shall be no greater than 10% of the arithmetic mean.

7.8 Determination of magnetic foreign matter content

7.8.1 Principle

After the sample is slurried, use a magnetic bar to adsorb iron, zinc and chromium; add hydrochloric acid and nitric acid to dissolve. Use the standard curve method, and take an inductively coupled plasma emission spectrometer to determine the iron, zinc and chromium content, and calculate the magnetic foreign matter content.

7.8.2 Reagents or materials

7.8.2.1 Nitric acid: guaranteed reagent.

8 Inspection Rules

8.1 All index items specified in Clause 6 are exit-factory inspection items and shall be inspected batch by batch.

8.2 The same type of refined nickel sulfate produced continuously or by the same team by the manufacturer using the same materials, basically the same production conditions is considered one batch. Each batch of products shall not exceed 100 t.

8.3 Determine the number of sampling units in accordance with the provisions of GB/T 6678. When sampling, insert the sampler vertically from the center of the packaging bag to 3/4 of the depth of the material layer. Mix the taken samples and reduce them to no less than 1,000 g using the quartering method. Divide the samples into two clean, dry containers; seal them; and label them with the manufacturer name, product name, category, batch number, sampling date, and name of the sampler. One copy is for inspection and the other is kept for future reference. The storage time is determined according to the needs of the manufacturer.

8.4 Determine whether the inspection results comply with this Document by the rounded value comparison method specified in GB/T 8170.

8.5 If any index of the inspection result does not meet the requirements of this Document, re-sample from double amount of packaging for re-inspection. If any index of the re-inspection result does not meet the requirements of this Document, the entire batch of products shall be unqualified.

9 Marking and Accompanying Documents

9.1 The packaging bags of refined nickel sulfate shall have firm and clear markings, including: manufacturer name, factory address, product name, category, net content, batch number or production date, this Document number; and "rainproof" and "sunproof" markings as specified in GB/T 191-2008.

9.2 Each batch of refined nickel sulfate products shipped out of the factory shall be accompanied by a quality certificate, including: manufacturer name, factory address, product name, category, net content, batch number or production date and this Document number.

10 Packaging, Transportation and Storage

10.1 Refined nickel sulfate products are double-packed, with polyethylene plastic film bags as the inner packaging and plastic woven bags as the outer packaging. The inner packaging bag is tied with vinyl rope or other ropes of equivalent quality; or sealed with other equivalent methods. The outer bag is sewn with a sewing machine; and the sewing is firm without leaking or

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