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Cobalt sulfate for battery materials

电池用硫酸钴

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Table of Contents

Cobalt sulfate for battery materials

1 Scope

This document specifies the classification, requirements, test methods, inspection rules, signs, labels, packaging, transportation, storage of cobalt sulfate for batteries.

This document applies to cobalt sulfate for batteries.

2 Normative references

The contents of the following documents constitute essential provisions of this document through normative references in the text. Among them, for dated reference documents, only the version corresponding to the date applies to this document; for undated reference documents, the latest version (including all amendments) applies 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 6682-2008 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

3 Terms and definitions

There are no terms or definitions to be defined in this document.

7 Test methods

Warning: Some of the reagents used in this test method are corrosive, so be careful when operating! If necessary, perform it in a fume hood. If it splashes on your skin or eyes, rinse with water immediately. In severe cases, seek medical attention immediately.

7.1 General provisions

The reagents and water used in this document, unless otherwise specified, refer to analytically pure reagents and grade-three water specified in Table 1 of GB/T 6682-2008. The standard titration solutions, impurity standard solutions, preparations, products used in the test shall be prepared, in accordance with the provisions of HG/T 3696.1, HG/T 3696.2, HG/T 3696.3, unless otherwise specified.

7.2 Appearance inspection

Under natural light, the appearance of the solid cobalt sulfate for batteries is visually determined, on a watch glass or white porcelain plate with a white substrate. The cobalt sulfate solution for batteries is placed in a colorimetric tube and visually determined on a white porcelain plate.

7.3 Determination of cobalt content

7.3.1 Potentiometric titration method (arbitration method)

7.3.1.1 Principle

In an ammoniacal solution, potassium ferricyanide is used to oxidize Co^{2+} to Co^{3+}; the excess potassium ferricyanide is titrated with cobalt standard titration solution.

The reaction equation is as follows:

$$Co^{2+} + [Fe(CN)_6]^{3-} \longrightarrow Co^{3+} + [Fe(CN)_6]^{4-}$$

7.3.1.2 Reagents or materials

7.3.1.2.1 Ammonium chloride.

7.3.1.2.2 Ammonia-ammonium citrate mixed solution.

Weigh 50 g of ammonium citrate. Dissolve it in water. A dd 350 mL of ammonia water. Use water to dilute it to 1000 mL. Mix well.

7.3.1.2.3 Cobalt standard titration solution: ρ (Co) = 3 g/L.

Weigh 3.000 g of metallic cobalt (cobalt mass fraction is not less than 99.98%) (accurate to 0.0002 g). Place it in a 400 mL beaker. Add a small amount of water to moisten it. Slowly add 30 mL of nitric acid solution (1 + 1). Heat until it is completely dissolved. After cooling to room temperature, transfer the entire volume into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

7.3.1.2.4 Potassium ferricyanide standard titration solution: $c(K_3[Fe(CN)_6]) \approx 0.05$ mol/L.

Follow the following steps for preparation, calibration, test data processing:

a) Preparation: Weigh about 17 g of potassium ferricyanide. Dissolve it in water. Filter it. Use water to dilute it to 1000 mL. Shake well.

b) Calibration: Use a pipette to transfer 20 mL of potassium ferricyanide solution. Place it in a 250 mL beaker. Add 5 g of ammonium chloride and 80 mL of ammonia-ammonium citrate mixed solution. Stir on a magnetic stirrer. On the adjusted automatic potentiometric titrator, use the platinum electrode as the indicator electrode and the tungsten electrode as the reference electrode; use cobalt standard titration solution to make titration, to the sudden jump end point.

c) Test data processing: The concentration of potassium ferricyanide standard titration solution is measured in c; the value is expressed in moles per liter (mol/L). Make calculation in accordance with formula (1):

$$c = \frac{V_1 \rho}{VM} \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots \quad (1)$$

Where:

V_1 - The value of the volume of the cobalt standard titration solution, which is consumed by the titration test solution, in milliliters (mL);

ρ - The exact value of the mass concentration of the cobalt standard titration solution, in grams per liter (g/L);

V - The value of the volume of the potassium ferricyanide standard titration solution pipetted, in milliliters (mL);

M - The numerical value of the molar mass of cobalt (Co), in grams per mole (g/mol) ($M = 58.93$).

If two people do three parallel measurements at the same time, the ratio -- of the range of each person's three parallel measurements TO the average -- shall not be greater than 0.2%; the ratio -- of the difference between the average of two people's measurements TO the average result of two people -- shall not be greater than 0.2%. The result takes the average value. The concentration value is taken to 4 significant figures.

greater than 0.1%.

7.3.2 EDTA titration method

7.3.2.1 Principle

At around pH 5.5, using hexamethylenetetramine as buffer, xylenol orange and malachite green as indicators, use disodium ethylenediaminetetraacetate (EDTA) standard titration solution to make titration, until the solution changes from purple blue to bright green, which is taken as the end point.

7.3.2.2 Reagents or materials

7.3.2.2.1 p-Nitrophenol.

7.3.2.2.2 Hexamethylenetetramine.

7.3.2.2.3 Hydrochloric acid solution: 1 + 1.

7.3.2.2.4 Ammonia solution: 1 + 1.

7.3.2.2.5 Disodium ethylenediaminetetraacetate (EDTA) standard titration solution: c (EDTA) ≈ 0.05 mol/L.

7.3.2.2.6 Xylenol orange indicator liquid: 2 g/L.

7.3.2.2.7 Malachite green solution: 2 g/L.

7.3.2.3 Test procedures

Weigh an appropriate amount of specimen (about 3.0 g of solid product, about 7.5 g of solution product) (accurate to 0.0002 g). Place it in a 150 mL beaker. Add a small amount of water. Boil the solid sample to dissolve it. After cooling, transfer everything to a 250 mL volumetric flask. Use water to dilute it to the mark. Shake well.

Use a pipette to transfer 25 mL of the test solution. Place it in a 500 mL conical flask. Add water to approximately 200 mL. Add 1 ~ 2 drops of p-nitrophenol. Use ammonia solution to make adjustment, until the solution turns yellow green. Then use hydrochloric acid solution to make adjustment, until the yellow green color disappears. Add another 6 drops. Then add 1.0 g ~ 2.0 g of hexamethylenetetramine. Heat until it is slightly boiling. Add 4 ~ 6 drops of xylenol orange indicator solution and 1 ~ 2 drops of malachite green solution. Immediately use disodium ethylenediaminetetraacetate (EDTA) standard titration solution to make titration, until the solution turns from purple blue to bright green, which is taken as the end point.

At the same time, do the same blank test. Except that no specimen is added to the blank test solution, the types and amounts of other reagents added (except the standard titration solution) are the same as the test solution.

7.3.2.4 Test data processing

The cobalt content is calculated as the mass fraction w_1 of cobalt (Co), which is calculated according to formula (3):

$$w_1 = \frac{(V - V_0)cM \times 10^{-3}}{m \times 25/250} \times 100\% \quad \cdots\cdots\cdots\cdots (3)$$

Where:

V - The volume of disodium ethylenediaminetetraacetate (EDTA) standard titration solution, which is consumed by the titration test solution, in milliliters (mL);

V_0 - The volume of disodium ethylenediaminetetraacetate (EDTA) standard titration solution, which is consumed by titrating the blank test solution, in milliliters (mL);

c - The exact value of the concentration of disodium ethylenediaminetetraacetate (EDTA) standard titration solution, in moles per liter (mol/L);

M - The numerical value of the molar mass of cobalt (Co), in grams per mole (g/mol) (M = 58.93);

m - The numerical value of the mass of the sample, in grams (g).

Take the arithmetic mean of the parallel measurement results as the measurement result. The absolute difference between the two parallel measurement results shall not be greater than 0.1%.

7.4 Determination of water-insoluble matter content

7.4.1 Principle

The specimen is dissolved in water. After filtering and rinsing, it is dried until the mass is constant. The water-insoluble matter content is determined, based on the amount of residue after drying.

7.4.2 Instruments and equipment

7.4.2.1 Glass sand crucible: Filter plate pore size is 5 μm ~ 15 μm.

7.4.2.2 Electric constant temperature drying oven: The temperature can be controlled at 105 °C ± 2 °C.

7.4.3 Analytical procedures

Weigh about 50 g of specimen (accurate to 0.01 g). Place it in a 400 mL beaker. Add 200 mL of water. Heat and stir. Boil to dissolve it. After cooling to room temperature, use a glass sand crucible that has been previously dried at 105 °C ± 2 °C to a constant

7.6.2.3 Water: Grade-II water in compliance with Table 1 of GB/T 6682-2008.

7.6.3 Instruments and equipment

Atomic absorption spectrophotometer: It is equipped with lead hollow cathode lamp.

7.6.4 Test procedures

7.6.4.1 Drawing of standard curve

Use a pipette to transfer 0.00 mL, 0.25 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL of lead standard solutions, respectively. Place it in six 100 mL volumetric flasks. Add 25 mL of cobalt matrix solution (see 7.5.2.2) and 2 mL of nitric acid solution to each. Use water to dilute it to the mark. Shake well.

Adjust the atomic absorption spectrophotometer to the best working conditions. Use the standard blank solution to zero it. Measure the absorbance of the standard solution, at a wavelength of 283.3 nm. Draw a standard curve, using the mass of lead (mg) as the abscissa and the corresponding absorbance as the ordinate.

7.6.4.2 Test

Weigh about 2.5 g of the specimen (accurate to 0.0002 g). Place it in a 100 mL beaker. Add an appropriate amount of water. Boil the solid sample to dissolve it. After cooling, add 2 mL of nitric acid solution. Transfer the entire volume to a 100 mL volumetric flask. Add water to the mark. Shake well.

Measure the absorbance of the lead element in this test solution, on an atomic absorption spectrophotometer. Find out the corresponding mass of lead from the standard curve, based on the measured absorbance.

7.6.5 Test data processing

The lead content is measured as the mass fraction w_3 of lead (Pb), which is calculated according to formula (6):

$$w_3=\frac{m_1\times 10^{-3}}{m}\times 100\% \qquad \cdots\cdots\cdots\cdots\cdots\cdots (6)$$

Where:

m_1 - The value of the mass of lead in the test solution as found from the standard curve, in milligrams (mg);

m - The numerical value of the mass of the sample, in grams (g).

Take the arithmetic mean of the parallel measurement results as the measurement result. The absolute difference between the two parallel measurement results shall not be greater than 10% of the arithmetic mean.

7.7 Determination of sodium content

7.7.1 Principle

In nitric acid medium, the sodium content is determined by an inductively coupled plasma optical emission spectrometer, using the standard curve method.

7.7.2 Reagents or materials

7.7.2.1 Nitric acid solution: 1 + 1.

It is prepared with superior grade pure reagents.

7.7.2.2 Sodium standard solution: 1 mL of solution contains 0.01 mg of sodium (Na).

Use a pipette to transfer 1 mL of the sodium standard solution, which is prepared according to the requirements of HG/T 3696.2. Place it in a 100 mL volumetric flask. Use water to dilute it to the mark. Shake well. Prepare this solution before use. The solution shall be stored in polyethylene plastic bottles.

7.7.2.3 Water: Grade-II water compliant with Table 1 of GB/T 6682-2008.

7.7.3 Instruments and equipment

Inductively coupled plasma optical emission spectrometer.

7.7.4 Test procedures

7.7.4.1 Drawing of standard curve

Use a pipette to transfer 0.00 mL, 0.25 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL of sodium standard solutions, respectively. Place them into six 100 mL volumetric flasks. Add an appropriate amount of cobalt matrix solution to each (solid product 2.00 mL, solution product 0.20 mL) (see 7.5.2.2) and 2 mL of nitric acid solution. Use water to dilute it to the mark. Shake well.

Adjust the inductively coupled plasma emission spectrometer to the optimal working conditions. Use a standard blank solution to zero it. Measure the spectral intensity of the standard solution, at a wavelength of 589.592 nm. Draw a standard curve, using the mass of sodium (mg) as the abscissa and the corresponding spectral intensity as the ordinate.

7.7.4.2 Test

Use a pipette to transfer an appropriate amount of test solution (see 7.5.4.2) (20.00 mL for solid product, 2.00 mL for solution product). Place it in a 100 mL volumetric flask.

standard curve, in milligrams (mg)

m - The numerical value of the mass of the sample, in grams (g).

Take the arithmetic mean of the parallel measurement results as the measurement result. The absolute difference between the two parallel measurement results shall not be greater than 10% of the arithmetic mean.

7.9 Determination of oil content

7.9.1 Reagents or materials

7.9.1.1 Tetrachlorethylene.

7.9.1.2 Anhydrous sodium sulfate.

7.9.2 Instruments and equipment

Infrared spectrophotometer.

7.9.3 Test procedures

Weigh about 10 g of specimen (accurate to 0.01 g). Place it in a 200 mL beaker. Add 20 mL of water. Boil the solid sample to dissolve it. After cooling, move everything into a separatory funnel. Use a pipette to transfer 20 mL of tetrachlorethylene. Extract for 1 minute. Let it stand for stratification. Pour it all into a dehydration column, which has a thickness of 1 cm ~ 2 cm anhydrous sodium sulfate. Dehydrate it into a 25 mL colorimetric tube. Cover with lid and set aside.

Adjust the infrared spectroscopic oil meter to the best working conditions. Make measurement according to the instrument operating procedure. Input the mass of the specimen and the accurate volume of tetrachlorethylene. Read the oil content in the specimen directly from the instrument.

Take the arithmetic mean of the parallel measurement results as the measurement result. The absolute difference between the two parallel measurement results shall not be greater than 10% of the arithmetic mean.

8 Inspection rules

8.1 All indicator items specified in this document are exit-factory inspection items and shall be inspected batch by batch.

8.2 Cobalt sulfate for batteries of the same category and model, which is produced by the manufacturer using the same materials and basically the same production conditions, in continuous production or by the same shift, forms one batch. Each batch of products shall not exceed 40 t.

8.3 Determine the number of sampling units, according to the provisions of GB/T 6678. When sampling solid products, insert the sampler vertically from the center of the bag to 3/4 of the depth of the material layer to take sample. Mix the sample taken out uniformly. Use the quartering method to reduce it to not less than 1500 g. Divide it into two clean dry containers. Seal it. When sampling solution products, insert the sampling glass tube to 2/3 of the depth of the container to take a sample. Mix the sample collected uniformly. The total volume is not less than 500 mL. Divide it into two clean dry plastic bottle. Seal it. Attach a label, to indicate the manufacturer's name, product name, category, model, batch number, sampling date, sampler's name. One set is for inspection; the other set is kept for future reference. The storage time is determined according to the needs of the production enterprise.

8.4 If any index in the inspection results does not meet the requirements of this document, samples shall be taken from twice the quantity of packaging for re-inspection. Even if only one index does not meet the requirements of this document in the reinspection, the entire batch of products will be deemed unqualified.

8.5 Use the rounded value comparison method specified in GB/T 8170, to determine whether the inspection results comply with this document.

9 Signs, labels

9.1 Cobalt sulfate packaging bags for batteries shall have firm and clear signs, including: manufacturer name, factory address, product name, category, model, net content, batch number or production date, this document number, signs of "avoid rain" and "avoid sun exposure" in Table 1 of GB/T 191-2008.

9.2 Each batch of cobalt sulfate products for batteries leaving the factory shall be accompanied by a quality certificate, which indicates: manufacturer name, factory address, product name, category, model, net content, batch number or production date, this document number.

10 Packaging, transportation, storage

10.1 Cobalt sulfate solid products for batteries are double-packed, wherein the inner packaging uses polyethylene plastic film bags and the outer packaging uses plastic woven bags. The inner bag of the package shall be tied with vinyl rope or other ropes of equivalent quality, OR sealed with other equivalent methods. The outer bag is sewn using a sewing machine; the stitching is firm without leakage or skipping. The net content of each bag is 25 kg; it can also be packaged according to the specifications required by the user. Cobalt sulfate solution products for batteries are packaged in special tankers or polyethylene barrels. The net content of each barrel is 1000 kg; it can also be packaged according to the specifications required by the user.

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