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Battery grade lithium carbonate

电池级碳酸锂

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Battery grade lithium carbonate

1 Scope

This document specifies the classification, technical requirements, test methods, inspection rules, marking, packaging, transportation, storage, accompanying documents, order forms of battery-grade lithium carbonate.

This document is applicable to battery-grade lithium carbonate, which is produced by various methods.

2 Normative references

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

GB/T 191 Packaging - Pictorial marking for handling of goods

GB/T 6284 Chemical products for industrial use - General method for determination of water content - The loss of mass on drying method

GB/T 6678-2003 General principles for sampling chemical products

GB/T 11064 (all parts) Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride

GB/T 19077 Particle size analysis - Laser diffraction methods

3 Terms and definitions

The following terms and definitions apply to this document.

3.1

Magnetic impurity

Material that can be directly or indirectly adsorbed by ferromagnetic bodies.

Note: Mainly refers to magnetic substances in transition elements such as iron, chromium, nickel, zinc and their alloys.

The moisture content in the product shall not exceed 0.20%.

5.4 Loss on ignition

The loss on ignition in the product shall not exceed 0.50%.

5.5 Particle size

The particle size of the product shall meet the following requirements: $D_{10} \geq 1$ μm, 4 μm $\leq D_{50} \leq 8$ μm, 9 μm $\leq D_{90} \leq 15$ μm, $D_{99} \leq 30$ μm; or determined by negotiation between the supplier and the buyer.

5.6 Appearance quality

The product is in the form of white powder with no visible inclusions.

6 Test methods

6.1 The determination of the chemical composition of the product shall be carried out in accordance with the provisions of GB/T 11064 (all parts). The determination of the boron content shall be carried out in accordance with the provisions of Appendix A.

6.2 The determination of magnetic impurity in the product shall be carried out in accordance with the provisions of Appendix B.

6.3 The determination of moisture in the product shall be carried out in accordance with the provisions of GB/T 6284.

6.4 The determination of the loss on ignition in the product shall be carried out in accordance with the provisions of Appendix C.

6.5 The determination of the particle size of the product shall be carried out in accordance with the provisions of GB/T 19077.

6.6 The appearance quality of the product shall be visually inspected.

7 Inspection rules

7.1 Inspection and acceptance

7.1.1 The product shall be inspected by the supplier or a third party, to ensure that the product quality complies with the provisions of this document and the order form.

7.1.2 The purchaser may inspect the received products in accordance with the provisions of this document. If the inspection results are inconsistent with the provisions of this document and the order form, it shall be proposed to the supplier

b) Product name;

c) Batch number;

d) Net weight;

e) Main content;

f) This document number;

g) Origin;

h) "Avoid-rain" mark in GB/T 191.

8.2 Packaging, transportation, storage

8.2.1 The product is packed in polyethylene (PE) lined bags, polypropylene (PP) outer bags or two to three layers of paper bags. The inner bag is tied or heat-sealed; the outer bag is firmly tied. The net weight of each bag is implemented according to the requirements of the buyer.

8.2.2 The product shall avoid contact with acid during transportation; the packaging bag shall be prevented from being damaged during handling; attention shall be paid to moisture-proof.

8.2.3 The product shall be stored in a dry, acid-free environment.

8.3 Accompanying documents

Each batch of products shall be accompanied by accompanying documents, which shall include supplier information, product information, this document number, exit-factory date or packaging date; it should also include:

a) Product quality guarantee, which shall include the following:

- Main performance and technical parameters of the product;
- Product characteristics (including manufacturing process and raw material characteristics);
- Responsibility for product quality;
- Quality certification obtained by the product and various analytical test results.

b) Product certificate, which shall include the following:

- Inspection items and their results or test conclusions;
- Batch size or batch number;

Appendix A

(Normative)

Determination of boron content in lithium carbonate -- Inductively coupled plasma atomic emission spectrometry

A.1 Overview

This Appendix is applicable to the determination of boron content in lithium carbonate, which has a determination limit of 0.000050% ~ 0.010% boron content.

A.2 Principle

The sample is dissolved in hydrochloric acid. The boron content is determined by the standard working curve method on an inductively coupled plasma atomic emission spectrometer.

A.3 Reagents or materials

Unless otherwise specified, only reagents confirmed to be of superior purity are used in the analysis. Boron-containing vessels shall be avoided during the experiment.

A.3.1 Water, GB/T 6682, grade 2.

A.3.2 Hydrochloric acid (ρ = 1.19 g/mL).

A.3.3 Hydrochloric acid (1 + 1).

A.3.4 Boric acid (99.99%), spectrally pure. Dry under vacuum at 60 °C for 2 h; place in a desiccator to cool to room temperature.

A.3.5 Boron standard stock solution: Weigh 5.7179 g of boric acid (A.3.4) in a 200 mL polytetrafluoroethylene beaker. Add 50 mL of water to dissolve completely. Transfer to a 1000 mL volumetric flask. Add water to 900 mL. Add 20 mL of hydrochloric acid (A.3.2). Use water to dilute it to the mark. Shake well. Transfer to a plastic bottle for storage. 1 mL of this solution contains 1 mg of boron.

A.3.6 Boron standard solution A: Pipette 20.00 mL of boron standard stock solution (A.3.5) into a 200 mL beaker. Add 4 mL of hydrochloric acid (A.3.2). Use water to dilute to the mark. Shake well. Transfer to a plastic bottle for storage. 1 mL of this solution contains 100 μg of boron.

A.3.7 Boron standard solution B: Pipette 10.00 mL of boron standard solution A (A.3.6) into a 100 mL volumetric flask. Add 2 mL of hydrochloric acid (A.3.2). Use water to dilute to the mark. Shake well. Transfer to a plastic bottle for storage. 1 mL of this solution contains 10 μg of boron.

Appendix B

(Normative)

Determination of magnetic impurity content in lithium carbonate - Inductively coupled plasma atomic emission spectrometry

B.1 Summary

This Appendix is used to determine the content of magnetic impurity in lithium carbonate, which has a determination limit of 5.00 μg/kg ~ 1000.00 μg/kg.

B.2 Principle

Magnetic impurity in the sample is adsorbed and enriched by magnetic rods, decomposed with aqua regia; the content of magnetic impurity is determined by the standard working curve method on an inductively coupled plasma atomic emission spectrometer

B.3 Reagents or materials

Unless otherwise specified, only reagents confirmed to be of superior purity are used in the analysis. All the instruments used in the experiment are soaked in nitric acid (1 + 4) and then thoroughly cleaned with ultrapure water.

B.3.1 Water, GB/T 6682, Grade II.

B.3.2 Nitric acid (ρ = 1.42 g/mL).

B.3.3 Hydrochloric acid (ρ = 1.19 g/mL).

B.3.4 Nitric acid (1 + 1)

B.3.5 Aqua regia: Mix 1 volume of nitric acid (B.3.2) and 3 volumes of hydrochloric acid (B.3.3); prepare it when needed.

B.3.6 Iron standard storage solution: Weigh 1.0000 g of pure metal iron wire (≥ 99.9%) into a 200 mL beaker. Add 20 mL of nitric acid (B.3.4). Dissolve in a water bath until clear. Cool it. Transfer to a 1000 mL volumetric flask. Use water to dilute to the mark. Shake well. 1 mL of this solution contains 1 mg of iron.

B.3.7 Zinc standard stock solution: Weigh 1.0000 g of pure metallic zinc (≥ 99.9%) in a 200 mL beaker. Add 20 mL of nitric acid (B.3.4). Dissolve at low temperature until clear. Cool it. Transfer to a 1000 mL volumetric flask. Use water to dilute to the mark. Shake well. 1 mL of this solution contains 1 mg of zinc.

B.3.8 Nickel standard stock solution: Weigh 1.0000 g of pure metallic nickel (≥ 99.9%) in a 200 mL beaker. Add 20 mL of nitric acid (B.3.4). Dissolve at low temperature until

clear. Cool it. Transfer to a 1000 mL volumetric flask. Use water to dilute to the mark. Shake well. 1 mL of this solution contains 1 mg of nickel.

B.3.9 Chromium standard storage solution: Weigh 1.0000 g of pure metallic chromium (purity 99.99%) into a 200 mL beaker. Add 20 mL of nitric acid (B.3.4). Dissolve at low temperature until clear. Cool it. Transfer to a 1000 mL volumetric flask. Use water to dilute to the mark. Shake well. 1 mL of this solution contains 1 mg of chromium.

B.3.10 Mixed standard solution A: Transfer 20.00 mL of each standard storage solution (B.3.6 to B.3.9) into a 200 mL volumetric flask. Add 20 mL of nitric acid (B.3.4). Use water to dilute to the mark. Shake well. 1 mL of this solution contains 100 μg of iron, zinc, nickel, chromium.

B.3.11 Mixed standard solution B: Pipette 10.00 mL of mixed standard solution A (B.3.10) into a 100 mL volumetric flask. Add 10 mL of nitric acid (B.3.4). Use water to dilute to the mark. Shake well. 1 mL of this solution contains 10 μg of iron, zinc, nickel, chromium.

B.3.12 Argon ($w_{Ar} \geq 99.995\%$).

B.4 Instruments and equipment

B.4.1 Inductively coupled plasma atomic emission spectrometer.

B.4.2 Magnetic bar: 6000 Gs ~ 8000 Gs, Φ17 mm × 53 mm, wrapped with polytetrafluoroethylene. Before adsorption, place it in a 200 mL beaker; add 130 mL of ultrapure water and 8 mL of aqua regia (B.3.5); boil slightly for 30 min; then wash with ultrapure water.

B.4.3 Rod mill, which has speed regulation and timing functions.

B.5 Sample

Take a dried, non-agglomerated battery-grade lithium carbonate sample; place it in a desiccator for testing.

B.6 Test steps

B.6.1 Parallel test

Perform two tests in parallel.

B.6.2 Blank test

Perform a blank test with the sample.

B.6.3 Preparation of test solution

Appendix C

(Normative)

Determination of loss on ignition

C.1 Principle

Weigh a certain amount of specimen. Dry it at 500 °C. Calculate the thermal drying loss. Calculate the loss on ignition in the specimen.

C.2 Equipment

C.2.1 Porcelain crucible: 30 mL.

C.2.2 Muffle furnace.

C.2.3 Dryer.

C.3 Test steps

Weigh 5 g ~ 10 g of specimen into a porcelain crucible that has been pre-dried at 500 °C to a constant weight (accurate to 0.0001 g). Place in a muffle furnace heated to 500 °C ±5 °C and heat for 30 min. Take out the sample and place it in a dryer. Cool to room temperature and weigh it.

Repeat the operation until the difference between the two weighing is no more than 0.0003 g, which is regarded as constant weight.

C.4 Test data processing

Calculate the loss on ignition of the sample according to formula (C.1); the value is expressed in %.

$$W_{\text{loss on ignition}} = \frac{m_2 - m_3}{m_2 - m_1} \times 100\% \quad \text{(C.1)}$$

Where:

m_2 - The mass of the porcelain crucible, in grams (g);

m_3- The mass of the porcelain crucible and the specimen before drying, in grams (g);

m_1 - The mass of the porcelain crucible and the specimen after drying, in grams (g).

The calculation result is expressed to two decimal places.

C.5 Allowable difference of results

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