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**Hard carbon**

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## Table of Contents

|                                                                                                                     |    |
|---------------------------------------------------------------------------------------------------------------------|----|
| Foreword .....                                                                                                      | 3  |
| 1 Scope .....                                                                                                       | 4  |
| 2 Normative references .....                                                                                        | 4  |
| 3 Terms and definitions .....                                                                                       | 4  |
| 4 Classification and code .....                                                                                     | 5  |
| 5 Technical requirements .....                                                                                      | 6  |
| 6 Test methods .....                                                                                                | 8  |
| 7 Inspection rules .....                                                                                            | 9  |
| 8 Packaging, marks, storage and transportation .....                                                                | 10 |
| Annex A (normative) Determination method for first discharge specific capacity and first coulombic efficiency ..... | 11 |

# Hard carbon

## 1 Scope

This document specifies the classification and code, technical requirements, test methods, inspection rules, packaging, marks, storage and transportation of hard carbon materials.

This document applies to hard carbon materials for lithium-ion batteries, lithium-ion capacitors and sodium-ion batteries.

## 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 1427, *Specimen method of carbon material*

GB/T 4369-2015, *Lithium*

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

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

GB/T 8719, *General rule for packing, marking, storage, transport and quality certificates of carbonaceous material and products*

GB/T 19077, *Particle size analysis -- Laser diffraction methods*

GB/T 19587, *Determination of the specific surface area of solids by gas adsorption using the BET method*

GB/T 21354, *Powders -- Determination of tap density*

GB/T 24533-2019, *Graphite negative electrode materials for lithium-ion battery*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 3.1 hard carbon

Amorphous carbon that is difficult to graphitize at temperatures above 2500°C

**NOTE:** It is generally believed that amorphous carbon with a (002) interplanar spacing ( $d_{002}$ ) greater than or equal to 0.368 nm is hard carbon.

## 4 Classification and code

### 4.1 Product classification

Hard carbon products are classified into resin hard carbon, asphalt hard carbon and bio-based hard carbon according to the raw material.

- a) Resin hard carbon: hard carbon made from natural or synthetic resin polymer materials, represented by RHC.
- b) Asphalt hard carbon: hard carbon made from coal tar or petroleum asphalt, represented by PHC.
- c) Bio-based hard carbon: hard carbon made from animals and plants, represented by BHC.

### 4.2 Product grades

Product grades are classified according to Table 1.

### 4.3 Product code

The product code is composed of the classification code, first discharge specific capacity, and median particle size ( $D_{50}$ ) in sequence. See Table 2 for specific examples.

## **5 Technical requirements**

### **5.1 Appearance**

Black powder material, no metallic luster, no impurities visible.

### **5.2 Physical and chemical indicators**

The physical and chemical indicators of hard carbon materials shall comply with the requirements of Table 3, Table 4 and Table 5. If there are special requirements, they shall be determined through negotiation between the supplier and the buyer.

## **6 Test methods**

### **6.1 Appearance**

Visual observation under sufficient lighting conditions.

### **6.2 Particle size distribution**

Measure in accordance with the provisions of GB/T 19077.

### **6.3 Moisture content**

The moisture content shall be determined in accordance with the provisions of Annex B of GB/T 24533-2019.

### **6.4 pH value**

The pH value shall be determined in accordance with the provisions of Annex C of GB/T 24533-2019.

### **6.5 Specific surface area**

Measure in accordance with the provisions of GB/T 19587.

#### **6.6 (002) Interplanar spacing ( $d_{002}$ )**

Measure in accordance with the provisions of Annex E of GB/T 24533-2019.

#### **6.7 First discharge specific capacity and first coulombic efficiency**

Measure in accordance with the provisions of Annex A.

#### **6.8 Trace metal elements**

Measure in accordance with the provisions of Annex H of GB/T 24533-2019.

#### **6.9 Magnetic materials**

Measure in accordance with the provisions of Annex K of GB/T 24533-2019.

#### **6.10 Tap density**

Measure in accordance with the provisions of GB/T 21354.

### **7 Inspection rules**

#### **7.1 Sampling method**

Sampling shall be carried out in accordance with GB/T 1427. Samples shall be kept in a sealed container.

#### **7.2 Inspection categories**

##### **7.2.1 Exit-factory inspection**

Exit-factory inspection items: particle size, moisture, pH value, tap density, specific surface area, inter-planer spacing, first discharge specific capacity, first coulomb efficiency, trace elements and magnetic substances.

##### **7.2.2 Type inspection**

Type inspection items include all technical requirements specified in Chapter 5. Type inspection shall be carried out in any of the following situations:

- a) When there are changes in the model or supplier of raw materials;
- b) When there are changes in the production process;
- c) When the production equipment has been shut down for more than half a year and is resuming production.

### **7.3 Acceptance rules**

**7.3.1** Products that meet all technical indicators in Table 3, Table 4 or Table 5 are qualified products. If any indicator does not meet the requirements, double sampling shall be taken from the sampling bag of the same batch of products to re-test the unqualified items. If all the re-test results meet the requirements, the batch of products is judged to be qualified products, otherwise, the batch of products is judged to be unqualified products.

**7.3.2** Products shipped by the manufacturer shall comply with the requirements of this document. Each batch of products shall be accompanied by an inspection report when shipped.

**7.3.3** The re-inspection period is 1 month. If there is any objection, double sampling shall be carried out for re-inspection. If there is still a dispute, the inspection shall be carried out by a qualified third-party testing agency.

## **8 Packaging, marks, storage and transportation**

**8.1** The packaging and labeling of the product shall comply with the provisions of GB/T 8719. The net weight shall be negotiated by the supplier and the buyer.

**8.2** Packaging shall be carried out in a dry environment. Put the product into waterproof packaging bags (polyethylene sealed bags and aluminum-plastic sealed bags are recommended). Special packaging requirements shall be agreed upon by the supplier and the buyer.

**8.3** Products shall be stored in a ventilated and dry warehouse.

**8.4** Products shall be stacked neatly and cleanly. Labels such as production batch numbers shall be clearly identifiable.

**8.5** Avoid mixing storage or transportation with items that may cause product deterioration or damage packaging bags.

**8.6** The product packaging shall not be damaged during storage and transportation.

## Annex A

(normative)

### Determination method for first discharge specific capacity and first coulombic efficiency

**WARNING -- Personnel using this appendix shall have practical experience in formal laboratory work. This appendix does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring that the conditions specified by relevant national laws and regulations are met. Metallic lithium shall not come into contact with water and shall be protected from moisture.**

#### A.1 Overview

This appendix specifies the test method for the first discharge specific capacity and first coulombic efficiency of hard carbon materials.

#### A.2 Reagents and materials

**A.2.1** Water: It shall meet the requirements of Grade III water in GB/T 6682.

**A.2.2** Conductive agent: Acetylene carbon black; specific surface area is  $50\text{ m}^2/\text{g}$ ~ $80\text{ m}^2/\text{g}$ ; median particle size is  $D_{50}\ 30\text{ nm}$ ~ $60\text{ nm}$ .

**A.2.3** Binder: polyacrylic acid (PAA); viscosity is greater than  $7300\text{ MPa}\cdot\text{s}$ ,  $D_{50}\leq 1\text{ }\mu\text{m}$ ; or binder with equivalent performance.

**A.2.4** Metal lithium sheet or lithium ribbon: Metal lithium sheet or lithium ribbon that meets the requirements of Li-3 designation in GB/T 4369-2015; the diameter shall be  $12\text{ mm}$ ~ $18\text{ mm}$ ; the thickness shall be  $0.6\text{ mm}$ ~ $1.2\text{ mm}$ .

**A.2.5** Hard carbon current collector: Copper foil.

**A.2.6** Lithium current collector: Nickel mesh; thickness shall be  $0.15\text{ mm}$ ~ $0.25\text{ mm}$ ; diameter shall be  $14\text{ mm}$ ~ $18\text{ mm}$ .

**A.2.7** Diaphragm: Polyethylene-polypropylene (PE-PP) composite film; thickness shall be  $20\text{ }\mu\text{m}$ ~ $150\text{ }\mu\text{m}$ ; diameter shall be  $18\text{ mm}$ ~ $24\text{ mm}$ .

**A.2.8** Electrolyte: A solution with a concentration of  $1\text{ mol/L}$  formed by dissolving lithium hexafluorophosphate ( $\text{LiPF}_6$ ) in an organic solvent [ethylene carbonate (EC), dimethyl carbonate (DMC), and ethyl methyl carbonate (EMC) in a volume ratio of 1:1:1].

## A.4 Preparation of button cells

**A.4.1** Pole preparation environment: temperature is  $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$ , relative humidity is  $\leq 50\%$ ; button cell assembly environment: temperature is  $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$ , moisture content is not more than  $1 \text{ mg/m}^3$ , and oxygen content is not more than  $10 \text{ mg/m}^3$ .

**A.4.2** Place the specimen and conductive agent (A.2.2) in a forced air-drying oven (A.3.4). Dry at  $80^{\circ}\text{C} \sim 120^{\circ}\text{C}$  for 4 h, take out and place in a desiccator to cool to room temperature for use. Take a certain amount of binder (A.2.3) and place it in a beaker. Add water (A.2.1) and mix and dilute thoroughly to obtain a binder aqueous solution with a solid content of 5% for use.

**A.4.3** Calculate the solid content of the dried specimen and binder and the dried conductive agent in a mass fraction ratio of 91:6:3. Use an electronic balance (A.3.5) to accurately weigh and mix the dried specimen, binder aqueous solution and dried conductive agent in A.4.2. Use a high-speed disperser (A.3.10) to stir at 800 r/min for 20 s. Then stir at 2 000 r/min for 5 min to form a paste.

**NOTE:** The reference weighing steps are as follows: Take 4.55 g of dry specimen and 0.15 g of dry conductive agent in A.4.2 into a 50 mL beaker. Add 6.0 g of binder aqueous solution with a solid content of 5% (see A.4.2).

**A.4.4** Use a film coating applicator (A.3.8) to evenly apply the paste on the hard carbon current collector (A.2.5) (or use other coating methods) until the surface is smooth. Place it horizontally on a glass plate. Move the whole into a forced air-drying oven (A.3.4) and dry it at  $95^{\circ}\text{C} \sim 105^{\circ}\text{C}$  for at least 2 h to obtain a pole piece to be processed with a single-sided surface density of  $60 \text{ g/m}^2 \sim 100 \text{ g/m}^2$ .

**A.4.5** Take the electrode to be processed. Prepare the electrode with a diameter of 8 mm~14 mm by cutting and pressing. After accurate weighing with an electronic balance (A.3.6), its mass is recorded as  $m_{\text{electrode}}$ . Wrap it with weighing paper and aluminum foil and move it into a vacuum drying oven (A.3.3). Vacuum dry it at  $110^{\circ}\text{C} \sim 130^{\circ}\text{C}$  for 6 h~12 h. Then put it into a parts box. Transfer it to an inert atmosphere (argon) glove box (A.3.2) for storage to obtain the processed electrode.

**A.4.6** Cut a piece of hard carbon current collector (A.2.5). Punch out 10 discs with the same diameter as the electrode piece processed in A.4.5. Use an electronic balance (A.3.6) to accurately weigh them and calculate their average mass, which is recorded as  $m_{\text{copper}}$ .

**A.4.7** The assembly of button cells shall be carried out in an inert atmosphere (argon) glove box (A.3.2). The assembly sequence from bottom to top is: positive electrode shell, electrode sheet, diaphragm, metal lithium sheet, lithium current collector, negative electrode shell. The specific operation is carried out according to the following steps:

- a) Place the positive electrode shell. The battery shell shall not be contaminated by

dust;

- b) Add a drop of electrolyte (A.2.8) and put in the electrode (see A.4.5). Use tweezers to gently press down so that the electrolyte (A.2.8) absorbs the electrode. Add a drop of electrolyte (A.2.8) on the surface of the electrode;
- c) Hold the diaphragm (A.2.7) with tweezers. The edge of the diaphragm (A.2.7) shall be against the edge of the battery shell. Slowly lower the diaphragm (A.2.7). If there are bubbles between the electrode and the diaphragm (A.2.7), slowly lift the diaphragm (A.2.7) and then slowly lower it. Remove the bubbles. Finally, release the tweezers. At this time, the diaphragm (A.2.7) shall be in the center of the positive electrode shell;
- d) Add a drop of electrolyte (A.2.8) in the middle of the diaphragm (A.2.7). Use tweezers to pick up the metal lithium sheet (A.2.4). Use a sponge polishing stick (A.3.9) to polish it. Put in the polished metal lithium sheet (A.2.4). Press lightly so that the metal lithium sheet (A.2.4) is absorbed by the electrolyte (A.2.8). Adjust the position of the metal lithium sheet (A.2.4) to the middle;
- e) Place the lithium current collector (A.2.6) in the middle. Cover the negative electrode case. The lithium current collector (A.2.6) shall not be displaced. The total amount of electrolyte (A.2.8) added shall be 30  $\mu\text{L}$ ~80  $\mu\text{L}$ .

## A.5 Test steps

**A.5.1** Ambient temperature: 25°C $\pm$ 2°C; relative humidity is  $\leq 40\%$ .

**A.5.2** Perform a charge-discharge cycle on the button cell on the battery tester (A.3.1). The charge-discharge rate is 0.1 C. The voltage range is 0.005 V~1.5 V. The charge-discharge requirements are as follows. The constant voltage method is preferred.

a) Constant voltage method:

- Stand still for 2 h;
- Discharge: discharge at a constant rate of 0.2 C until the voltage is less than 0.005 V; discharge at a constant voltage of 0.005 V until the current is less than  $3.5 \times 10^{-6} \text{ A}$ ;
- Stand still for 15 min;
- Charge: charge at a constant rate of 0.2 C until the voltage is greater than 1.5 V;
- Stand still for 15 min.

b) Constant current method:

- Stand still for 1 h;

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