NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 42161-2022

Electrochemical performance test of lithium iron phosphate - Test method for the initial discharge specific capacity and the initial efficiency

磷酸铁锂电化学性能测试 首次放电比容量及首次充放电效率测试方法

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Standardization Administration of the PRC

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Foreword

This document was issued on 30 December 2022 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 April 2023.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 42161-2022 covers the two numbers that decide what a batch of lithium iron phosphate cathode material is worth - the initial discharge specific capacity and the initial charge-discharge efficiency - through the test conditions, the reagents and materials, the instruments and equipment, the test steps, the processing of the test data, the allowable difference between results, and the report. Both numbers depend on how the powder is handled as much as on the powder itself. Lithium iron phosphate is fine material, the grade described here having a particle size D50 of 0.5 to 8.0 micrometres and a specific surface area of 6 to 30 square metres per gram, and water taken up from the air during preparation is spent on side reactions in the first cycle, where it reads as a loss the batch never caused. The conditions clause therefore holds the work at a relative humidity of not more than 40.0 percent and an ambient temperature of 20 to 30 degrees C, with rolling below 30.0 percent, and the equipment clause fixes the vacuum oven, the ovens and a balance reading to 0.0001 g. The allowable difference clause says how far two results may sit apart before the gap belongs to the material. Written for cathode material producers, cell makers qualifying

what they buy, and third-party laboratories.

This document describes the test method for the initial discharge specific capacity and the initial charge-discharge efficiency of lithium iron phosphate, i.e., the cathode material for lithium-ion batteries.

This document applies to the test for the initial discharge specific capacity and the initial charge-discharge efficiency of lithium iron phosphate, i.e., the cathode material for lithium-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 20252-2014 Lithium cobalt oxide

3 Terms and definitions

For the purpose of this document, the terms and definitions defined in GB/T 20252-2014 apply.

4 Test conditions

Unless otherwise specified, each test step should be carried out at a relative humidity not greater than 40.0 % and an ambient temperature of 20 degrees C ~ 30 degrees C. The rolling process should be carried out at a relative humidity not greater than 30.0 % and an ambient temperature not greater than 30 °C.

5 Reagents and materials

5.1 Lithium iron phosphate: the particle size characteristic value D50 is 0.5 um ~ 8.0 um, and the specific surface area is 6 m2/g ~ 30 m2/g.

5.2 Conductive agent: conductive carbon material.

5.3 Polyvinylidene fluoride (PVDF): battery grade, the weight average molecular weight is not less than 5 x 105, and the moisture (mass fraction) is not greater than

0.10 %.

5.4 N-methylpyrrolidone (NMP): battery grade, the purity is not less than 99.9 %, and the moisture (mass fraction) is not greater than 0.02 %.

5.5 Aluminum foil: the thickness is 8 um ~ 20 um.

5.6 Ethanol: analytical reagent.

5.7 Lithium-ion battery separator: polyolefin porous membrane, the porosity is 35.0 % ~ 60.0 %, the air permeability is 100 s/100 mL ~ 500 s/100 mL, the average pore size is not greater than 1.0 um, the diameter is 16.0 mm ~ 18.0 m, and the thickness is 9.0 um ~ 32.0 um.

5.8 Metal lithium sheet: the diameter is 12.0 mm ~ 16.0 mm, and the thickness is 0.40 mm ~ 0.80 mm.

5.9 Battery standard structural parts: model CR2016, CR2025 or CR2032, including positive electrode case, negative electrode case, gasket, and spring support piece (or nickel foam sheet).

5.10 Lithium-ion battery electrolyte: lithium-ion battery electrolyte composed of lithium hexafluorophosphate (LiPF6) and mixed carbonate-based organic solvents [ethylene carbonate (EC), dimethyl carbonate (DMC), ethyl methyl carbonate (EMC), etc.], the moisture is not greater than 0.002 %, the free acid (HF) is not greater than 0.005 %, and the conductivity (25 degrees C) is not less than 7.0 mS/cm.

5.11 Dust-free paper.

5.12 Nitrogen (or argon): the purity (volume fraction) is not less than 99.99 %.

6 Instruments and equipment

6.1 Vacuum oven: 0 degrees C ~ 200 degrees C, and the temperature deviation is +/- 2 degrees C.

6.2 Oven.

6.3 Electronic balance: the accuracy is 0.0001 g.

6.4 Electronic balance: the accuracy is 0.00001 g.

6.5 Dispersing mixer or pulp mixer.

6.6 Small coating machine for lithium-ion battery electrode sheets.

7.2.1 Weighing

Calculate the lithium iron phosphate, conductive agent, and PVDF pretreated in 7.1 according to the mass fractions of 80 % ~ 97 %, 1 % ~ 10 %, and 2 % ~ 10 % respectively, and weigh them with an electronic balance (6.3). Calculate the amount of NMP (5.4) according to the design requirements of solid content (mass fraction) of 25 % ~ 65 %, and weigh with an electronic balance (6.3).

7.2.2 Pulping

The pulping process is as follows:

a) Add the weighed NMP into the mixing tank of a dispersing mixer or pulp mixer (6.5); gradually add the weighed PVDF; disperse and stir until completely dissolved; to prepare a transparent glue, of which the mass fraction of PVDF is 2 % ~ 10 %;

b) add the weighed conductive agent to the above transparent glue; vacuum, disperse and stir evenly;

c) gradually add the weighed lithium iron phosphate in portions; vacuum, disperse and stir evenly;

d) add another NMP according to the designed solid content, to control the slurry viscosity at 3000 mPa - s ~ 20000 mPa - s; vacuum, disperse and stir evenly; complete the pulping process.

NOTE: The solid content in this document is the ratio of the mass of the cathode active material

lithium iron phosphate, conductive agent and PVDF to the mass of the cathode slurry.

7.2.3 Coating

Use a small coating machine for lithium-ion battery electrode sheets (6.6) to evenly coat the cathode slurry mixed in 7.2.2 on one side (matte side) of the aluminum foil (5.5) or directly on the carbon-coated aluminum foil, and the thickness of wet slurry coating is 100 um ~ 300 um. After the coating is completed, transfer the positive electrode sheet to a vacuum oven (6.1) for drying. During drying, vacuum or circulate