

HG

**CHEMICAL INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

HG/T 4701-2021

Replacing HG/T 4701-2014

Iron phosphate for batteries

电池用磷酸铁

(English Translation)

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1 Scope

This document specifies the requirements, test methods, inspection rules, marking, labeling, packaging, transportation, storage of iron phosphate for battery materials.

This document applies to iron phosphate for battery materials.

Note: This product is mainly used to manufacture lithium iron phosphate (LiFePO_4) lithium-ion battery positive electrode materials.

2 Normative references

The contents of the following documents constitute the essential terms of this document through normative references in the text. Among them, for referenced documents with dates, only the version corresponding to that date is applicable to this document; for referenced documents without dates, the latest version (including all amendments) is applicable to this document.

GB/T 191-2008 Packaging - Pictorial marking for handling of goods

GB/T 5162 Metallic powders - Determination of tap density

GB/T 6003.1-2012 Test sieves - Technical requirements and testing - Part 1: Test sieves of metal wire cloth

GB/T 6678 General principles for sampling chemical products

GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods

GB/T 6730.61-2005 Iron ores - Determination of carbon and sulfur content - High frequency combustion with infrared absorption method

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

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

HG/T 3696.3 Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis - Part 3: Preparations of reagent solutions solution turns a stable purple-red color, which is the end point. The iron content is calculated based on the consumption of potassium dichromate standard titration solution.

7 Test method

7.4.3.2

Electric constant temperature drying oven. Temperature can be controlled at $180\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

7.3.2 Reagents or materials

7.3.2.1

Hydrogen peroxide.

7.3.2.2

Hydrochloric acid.

7.3.2.3

Sulfuric acid-phosphoric acid mixed solution

Slowly add 15 mL of sulfuric acid to 70 mL water; cool it; add 15 mL of phosphoric acid; mix well.

7.3.2.4

Stannous chloride solution. 100 g/L

Weigh 10.0 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$); place in a dry beaker; dissolve in 40 mL of hydrochloric acid; use water to dilute to 100 mL; add 1 tin particle; store in a brown bottle.

7.3.2.5

Titanium trichloride solution. 2%

Pipette 10 mL of titanium trichloride solution (15% ~ 20%); use hydrochloric acid solution (1 + 9) to dilute to 100 mL; store in a brown bottle. It can be prepared before use.

Prepare and process the test data according to the following steps.

- a) Preparation. Weigh $2.45 \text{ g} \pm 0.2 \text{ g}$ of the standard potassium dichromate (accurate to 0.0001 g), which was dried at $120 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$ to a constant mass; dissolve it in water; transfer all to a 1000 mL volumetric flask; use water to dilute to the mark; shake well;
- b) Test data processing. The concentration of the standard potassium dichromate titration solution is expressed in moles per liter (mol/L), which is calculated according to formula (1).

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

M - The molar mass of potassium dichromate, in grams per mole (g/mol)

7.3.2.6

Potassium dichromate standard titration solution

Where. m - The mass of the standard potassium dichromate weighed, in grams (g);

7.3.2.7

Sodium tungstate indicator solution. 10%

Weigh 10.0 g of sodium tungstate ($\text{NaWO}_4 \cdot 2\text{H}_2\text{O}$); dissolve in 85 mL of water; add 5.0 mL of phosphoric acid; mix well; store in a brown bottle.

7.3.2.8

Sodium diphenylamine sulfonate indicator solution. 5 g/L.

7.3.3 Instruments and equipment

Test sieve. $\phi 200 \times 50$ - 0.1/0.071 GB/T 6003.1-2012.

7.3.4 Test steps

7.3.4.1

Preparation of test solution A Weigh 4 g of the sieved specimen (accurate to 0.0002 g); place it in a 250 mL beaker; moisten it with a small amount of water. Add 25 mL of hydrochloric acid; cover with a watch glass; heat at low temperature in a fume hood to dissolve. Rinse the watch glass with a small amount of water; cool it (filter if necessary); transfer it to a 250 mL volumetric flask; dilute to the mark; shake well. This is test solution A, which is used for the determination of iron and phosphorus content.

7.3.4.2

Test

Use a pipette to transfer 10 mL of test solution A into a 250 mL conical flask. Add 5 mL of hydrochloric acid; heat to near boiling. While hot, shake the conical flask and add stannous chloride solution drop by drop, until the solution changes from brown to light yellow (if the

solution becomes colorless after adding too much stannous chloride, add hydrogen peroxide until the solution turns light yellow). Add 4 ~ 5 drops of sodium tungstate indicator solution; add titanium trichloride solution drop by drop while shaking the conical flask, until the solution turns light blue. Immediately cool with running water; add 50 mL of water; titrate with potassium dichromate standard titration solution, until the blue color just fades (usually 1 ~ 2 drops, do not record the reading).

Add water to dilute it to 100 mL. Add 10 mL of sulfuric acid-phosphoric acid mixed solution, 3 ~ 4 drops of sodium diphenylamine sulfonate indicator solution; titrate with potassium dichromate standard titration solution until a stable purple-red color appears (does not disappear in 30 s), which is the end point.

At the same time, perform a blank test. Except for not adding the specimen (except the standard titration solution), the types and amounts of other reagents added to the blank

7.4.4 Test procedure

Use a pipette to transfer 25 mL of test solution A (see 7.3.4.1) into a 250 mL volumetric flask; dilute to the mark with water; shake well. Use a pipette to transfer 10 mL of this test solution into a 250 mL beaker; add 10 mL of nitric acid solution; add water to about 100 mL. Cover with a watch glass; slowly heat to boil; keep it at a slight boil for 5 minutes. Add 50 mL of quinoline molybdate solution; keep it warm for 30 seconds (do not use open flame when adding reagents or heating; do not stir when adding reagents or heating to avoid the formation of clots). Cool to room temperature and stir 3 ~ 4 times during the cooling process. Use a glass crucible that has been dried to constant mass at $180\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ to filter the upper clear liquid; wash the precipitate 5 ~ 6 times by decantation, using about 20 mL of water each time. Transfer the precipitate to a glass crucible and continue washing with water 3 ~ 4 times. Place the glass crucible and the precipitate in an electric constant temperature drying oven at $180\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$; dry for 45 minutes. Take it out; cool it slightly; place it in a dryer; cool it to room temperature. Weigh it to an accuracy of 0.0002 g.

At the same time, do a blank test. Except for not adding the sample to the blank test solution, the types and amounts of other reagents added are the same as those of the test solution.

7.4.5 Test data processing

The phosphorus content is calculated as the mass fraction w_2 of phosphorus (P), according to formula (3).

Wherein. m_1 - The mass of quinoline phosphomolybdic acid precipitate generated by the test solution, in grams (g); m_2 - The mass of quinoline phosphomolybdic acid precipitate generated by the blank test solution, in grams (g); m - The mass of the sample, in grams (g);