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Determination of contents of Fe (III) in nano lithium iron phosphate

纳米磷酸铁锂中三价铁含量的测定方法

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed by the Chinese Academy of Sciences.

This standard shall be under the jurisdiction of the National Nanotechnology Standardization Technical Committee of Nano Materials Subcommittee (SAC/TC 279 /SC 1).

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Determination of contents of Fe (III) in nano lithium iron phosphate

1 Scope

This standard specifies the principle, equipment, reagents, procedures and test report contents for the method of determination of trivalent iron content in nano lithium iron phosphate.

This standard is applicable to the determination of trivalent iron in nano lithium iron phosphate in the range of 0.2% ~ 5.0%.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this Standard.

GB/T 601-2002 Chemical reagent - Preparation of standard volumetric solutions

GB/T 603 Chemical reagent - Preparation of reagent solutions for use in test methods

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

3 Test principles

The difference between the mass fraction of total iron and the mass fraction of ferrous iron in the sample is the mass fraction of trivalent iron content in the sample.

First USE the hydrochloric acid to dissolve the sample to be tested to form a solution; respectively TAKE two sets of solution sample, 20.00 mL for each set. TAKE one set of sample solution; under the acidic condition (pH controlled at 1 ~ 2), USE the $SnCl_2$ solution to reduce most of the Fe (III); ADD the $CuSO_4$-isatin indicator; AND the solution changes from yellow to green; USE the $TiCl_3$ solution to reduce the Fe (III) of the remaining part; the slightly excessive $TiCl_3$

5 Reagent

WARNING: Some of the reagents used in this test method are toxic or corrosive AND shall be handled with care!

5.1 General requirements

Unless otherwise specified, it shall use the analytical reagents and level 3 water as specified in GB/T 6682-2008. AND all the reagents shall be prepared in accordance with the requirements of GB/T 603.

5.2 Hydrochloric acid

The mass fraction is more than 36%.

5.3 Sulfuric acid

The mass fraction is more than 98%.

5.4 Phosphoric acid

The mass fraction is more than 85%.

5.5 Hydrochloric acid solution (1 + 1)

MEASURE the equal volume of hydrochloric acid (5.2) and water; MIX it uniformly to prepare for use.

5.6 Sulfur phosphoric acid solution (15%)

Slowly ADD 150 mL of sulfuric acid (5.3) into 500 mL of water while stirring; after cooling it down, ADD 150 mL of phosphoric acid (5.4); USE water to dilute it to 1000 mL; MIX it uniformly.

5.7 Tin dichloride solution

WEIGH 5 g of tin chloride; DISSOLVE it in 10 mL of hydrochloric acid solution (5.5); USE water to dilute it to 100 mL; AND the solution shall be filtered if it is turbid.

5.8 Potassium dichromate standard titration solution [c(1/6 $K_2Cr_2O_7$) = 0.0500 mol/L]

PREPARE it in accordance with the method II in clause 4.5.2 of GB/T 601-2002.

5.9 Titanium trichloride solution

heating device to heat it slightly boiling for 30 min; TAKE it off and quickly ADD approximately 1.0 g of sodium bicarbonate (5.16) and 30 mL of water; USE water bath to cool it quickly to room temperature.

6.1.2 PLACE the medium-speed filter paper in a 100.00 mL volumetric flask; USE water to rinse the volumetric flask for 3 ~ 4 times; RINSE the precipitate for 3 ~ 4 times; MAKE its volume reach to the mark; SHAKE it uniformly to prepare for use.

6.2 Determination of total iron content in sample solution

6.2.1 USE the single-line pipette (4.3) to accurately pipette 20.00 mL of the newly prepared sample solution (6.1.2) into a 250 mL conical flask; ADD 30 mL of water and 2 mL of hydrochloric acid (5.5); SHAKE it uniformly and PLACE it on the heating device to heat it to boil for 30 s; TAKE it off; immediately ADD tin dichloride solution (5.7) until the solution is pale yellow (if the solution itself is yellow but not apparent, it can skip this step); then ADD 2 drops of copper sulphate-isatin indicator (5.13) and the solution turns green; ADD titanium trichloride solution (5.9) until the green disappears; ADD one more drop; LET it stand; COOL it down.

6.2.2 After the solution is cooled to room temperature, it changes into blue; ADD 20 mL of sulfur phosphoric acid solution (5.6) and 4 drops of diphenylamine sulfonate indicator (5.14); USE the potassium dichromate standard titration solution (5.8) for titration, until the solution changes from green to purple, which is the end point; RECORD the volume of the consumed potassium dichromate V.

6.2.3 USE the single-line pipette (4.4) to accurately pipette 5.00 mL of ferrous ammonium sulfate solution (5.15) to perform blank test together with the sample. USE the potassium dichromate standard titration solution (5.8) for titration, until the solution changes from green to purple, which is the end point; RECORD the volume of the consumed potassium dichromate standard titration solution as V_1; then ADD 5.00 mL of ammonium ferrous sulfate solution (5.15); USE the potassium dichromate standard titration solution for titration, until the solution changes from green to purple, which is the end point; RECORD the volume of the consumed potassium dichromate standard titration solution as V_2.

Calculate the mass fraction of total iron in the sample in accordance with the formula (4).

$$W_{Fe}=\frac{c(1/6K_2Cr_2O_7)\times[V-(V_1-V_2)]\times 55.84}{m\times\frac{20}{100}\times 1\,000}\times 100 \qquad \cdots\cdots(4)$$

V_3 - Volume of potassium dichromate standard titration solution consumed for the titration of the ferrous iron in the sample solution, in milliliters (mL);

V_1 - Volume of potassium dichromate standard titration solution consumed for the titration of blank solution 1 (ADD 5.00 mL of ferrous ammonium sulfate solution into the blank solution), in milliliters (mL);

V_2 - Volume of potassium dichromate standard titration solution consumed for the titration of blank solution 2 (TITRATE the blank solution 1 to the end point and ADD 5.00 mL of ammonium ferrous sulfate solution), in milliliters (mL);

55.84 - Molar mass of iron in grams per mole (g/mol);

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

6.4 Calculation of trivalent iron content in sample

CALCULATE the mass fraction of trivalent iron in the sample in accordance with the formula (6):

$$W_{Fe^{3+}} = W_{Fe} - W_{Fe^{2+}} \quad \cdots\cdots (6)$$

Where:

W_{Fe3+} - Mass fraction of trivalent iron in the sample, expressed in %

W_{Fe} - Mass fraction of total iron in the sample, expressed in %

W_{Fe2+} - Mass fraction of ferrous iron in the sample, expressed in %.

7 Repeatability

7.1 This method does not have a suitable standard sample for determining the deviation, so the repeatability is determined by repeated testing.

7.2 USE the same sample for the test in 4 laboratories, AND the inner-laboratory and inter-laboratory sample average value and standard deviation are respectively as shown in Table 1.

Table 1 -- Inner-laboratory and inter-laboratory sample average and standard deviation

Item	Requirements
Inter-laboratory average value	1.24%
Inter-laboratory standard deviation	0.052%