

ICS 77.120.99

YS

**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 1028.1-2015

**Methods for chemical analysis of lithium iron phosphate. Part 1:
Determination of total iron content. Titanium trichloride
reduction potassium dichromate titration methods**

磷酸铁锂化学分析方法 第1部分：总铁量的测定 三氯化钛还原重铬酸钾滴定法
(English Translation)

Issued on: April 30, 2015

Implemented on: October 1, 2015

Issued by: Ministry of Industry and Information Technology of PRC

Table of Contents

Foreword	3
1 Scope	4
2 Summary of method	4
3 Reagents and materials	4
4 Specimens	5
5 Analytical procedures	5
6 Calculation of analysis results	6
7 Precision	6
8 Test report	7

1 Scope

This Part of YS/T 1028 specifies the method for the determination of total iron in lithium iron phosphate.

This section applies to the determination of total iron in lithium iron phosphate. The measuring range is: 28% ~ 36%.

2 Summary of method

After the sample is dissolved in hydrochloric acid, sodium tungstate is used as an indicator; a small amount of ferric iron is reduced to ferrous iron with titanium trichloride, to form "tungsten blue"; the excess trivalent titanium is naturally oxidized by the oxygen in the air. In the sulfuric acid-phosphoric acid medium, sodium diphenylamine sulfonate is used as an indicator, to titrate the divalent iron by the use of potassium dichromate standard solution.

3 Reagents and materials

3.1

Hydrochloric acid (1 + 1).

3.2 Sulfuric acid-phosphoric acid mixed solution: Slowly add 15 mL of sulfuric acid (rho

~ 1.84 g/mL) to 70 mL of water. After cooling, add 15 mL of phosphoric acid (rho~ 1.69 g/mL). Mix well.

3.3 Titanium trichloride solution (1 + 14): Take 2 mL of titanium trichloride solution

(about 15% titanium trichloride solution). Use hydrochloric acid (1 + 5), to dilute it to 30 mL.

3.4 Potassium dichromate standard titration solution [c() = 0.0500 mol/L]:

Weigh 2.4516 g of standard potassium dichromate (after predried at 140 °C ~ 150 °C for 2 h, place it in a desiccator and cool to room temperature). After dissolving in water, transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

3.5 Sodium tungstate indicator solution (250 g/L): Weigh 25 g of sodium tungstate and dissolve it in an appropriate amount of water. Add 5 mL of phosphoric acid. Use water to dilute it to 100 mL. Mix well.

3.6 Sodium diphenylamine sulfonate indicator solution (5 g/L): Weigh 0.50 g of sodium

diphenylamine sulfonate. Dissolve it in water. Use water to dilute it to 100 mL. Mix well. Store in a brown bottle.

4 Specimens

The particle size of the specimen shall not be greater than 0.10 mm.

5.1 Sample

Weigh 4.00 g of specimen, accurate to 0.0001 g.

5.2 Number of measurements

Carry out two independent measurements. Take the average value.

5.3 Blank test

Do a blank test with the sample.

5.4 Determination

5.4.1

Put the sample (5.1) in a 250 mL beaker. Add 40 mL of hydrochloric acid (3.1).

Cover a watch glass. Heat in a fume hood at low temperature to a slight boil, for 3 min ~ 5 min. Use a small amount of water, to rinse the watch glass. After cooling, use filter paper to filter it. Use water to wash the filter residue 5 ~ 7 times. Receive the filtrate in a 250 mL volumetric flask. Use water to dilute it to the mark. Shake well.

5.4.2 Pipette 25.00 mL of test solution (5.4.1) into a 250 mL conical flask. Add 1 mL

of sodium tungstate indicator solution (3.5). Dropwise add titanium trichloride solution (3.3) to the test solution, under constant shaking, until a steady blue color appears. Wait until the blue color disappears.

5.4.3 Immediately add 20 mL of sulfuric acid-phosphoric acid mixed solution (3.2).

Add 4 drops of sodium diphenylamine sulfonate indicator solution (3.6). Use potassium dichromate standard titration solution (3.4), to titrate it, until the solution turns purple and does not disappear for 30 seconds, which is the end.