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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 11064.7-2013

Replacing GB 11064.7-1989

**Methods for chemical analysis of lithium carbonate, lithium hydroxide
monohydrate and lithium chloride - Part 7: Determination of iron
content-1,10-phenanthroline spectrophotometric method**

碳酸锂、单水氢氧化锂、氯化锂化学分析方法 第7部分：铁量的测定 邻二氮杂菲
分光光度法

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Foreword

GB/T 11064 "Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride" is divided into 16 parts. - Part

- 1.Determination of lithium carbonate content - Acid-alkali titrimetric method; - Part
- 2.Determination of lithium hydroxide content - Acid-alkali titrimetric method; - Part

3 Determination of lithium chloride content - Potentiometric method; - Part

- 4.Determination of potassium and sodium content - Flame atomic absorption spectrometric method; - Part
- 5.Determination of calcium content - Flame atomic absorption spectrometric method; - Part
- 6.Determination of magnesium content - Flame atomic absorption spectrometric method; - Part
- 7.Determination of iron content - 1,10-Phenanthroline spectrophotometric method; - Part
- 8.Determination of silicon content - Molybdenum blue spectrophotometric method; - Part

5 Samples

5.1 The samples of lithium carbonate and lithium chloride shall be preheated at 250°C~260°C for 2h; then cooled down to room temperature in a desiccator.

6 Analysis Steps

6.1 Samples Weigh 5.00g of the sample, accurate to 0.0001g.

6.2 Number of Determination Independently carry out two measurements; take the mean value.

6.3 Blank Test Carry out a blank test for the accompanying samples. Add the same amount of acid as that for sample dissolving. Evaporate it to be almost dry at a low temperature.

6.5 Working Curve Plotting

6.5.1 Take 0mL, 0.25mL, 0.50mL, 1.00mL, 2.00mL, and 3mL of iron standard solutions (3.10) to a group of 25ml volumetric flasks, respectively; dilute them with water to 10mL; add 2 drops of hydrochloric acid (3.1), as stated in 6.4.3.

7 Calculation of Analysis Results The iron content is calculated in mass fraction $w(\text{Fe})$, expressed in %, according to equation (1).

7 Determination of iron content - 1,10-Phenanthroline spectrophotometric method

1 Scope This part of GB/T 11064 specifies the determination method of iron content in lithium carbonate, in lithium hydroxide monohydrate, and in lithium chloride. This part applies to the determination of iron content in lithium carbonate, in lithium hydroxide monohydrate, and in lithium chloride. The determination range. 0.00030%~0.030%.

2 Method Summary Dissolve the sample in hydrochloric acid. Reduce the iron (III) to iron (II) by ascorbic acid. In the pH

3.5 acetate buffer medium, iron (II) reacts with phenanthroline to produce a salmon complex compound, whose absorbance shall be measured at the 510nm wavelength of a spectrophotometer.

3 Reagents Unless otherwise specified, the reagent used in this Part is an analytical pure reagent, and the water used is the secondary deionized water.

3.1 Hydrochloric acid (1+1), GR.

3.2 Hydrochloric acid (1+7), GR.

3.3 Hydrochloric acid (2+1), GR.

3.8 P-Nitrophenol indicator (2g/L). use ethanol to prepare.

3.9 Iron standard stock solution. weigh 0.1430g of ferric oxide [$w(\text{Fe}_2\text{O}_3) \geq 99.9\%$] which has been dried at $105^\circ\text{C} \sim 110^\circ\text{C}$ in advance for 2h, and then cooled down to room temperature. Place it into a 200mL beaker. Add 20mL of hydrochloric acid (3.1) in the beaker; heat the beaker at a low temperature until the ferric oxide completely dissolves. Cool the mixture down; transfer it to a 1,000mL volumetric flask. Dilute it with water to the scale; shake it up. 1mL of this solution contains $100\mu\text{g}$ of iron.

4 Instrumentation Spectrophotometer.

8 Precision

8.1 Repeatability For the two independent test results obtained under repeatability conditions, within the average-value range as given below, the absolute difference of these two test results does not exceed the repeatability limit (r), and the situation exceeding repeatability limit (r) is not more than 5%. Repeatability limit (r), according to Table 2, adopts linear interpolation to obtain.

11 Determination of Acid-insolubles Content - Gravimetric method; - Part

12.Determination of carbonate content - Determination of lithium chloride content - Potentiometric method; - Part

13.Determination of aluminum content - Chromazurol S-cetylpyridine bromide spectrophotometric method; - Part

14.Determination of arsenic content - Molybdenum blue spectrophotometric method; - Part

15 Determination of fluoride content - Ion selective method; - Part

16.Determination of calcium, magnesium, copper, lead, zinc, nickel, manganese, cadmium and aluminum content - Inductively coupled plasma atomic emission spectrometry. This Part is part 7 of GB/T 11064. This Part is drafted in accordance with rules given in GB/T 1.1-2009. This Part replaces GB/T 11064.7-1989 "Lithium carbonate, lithium hydroxide monohydrate and lithium chloride-Determination of iron content - 1.10-phenanthroline spectrophotometric method". Compared with GB/T 11064.7-1989, the main changes of this Part are as follows: - ADD the repeatability terms; - RE-EDIT the text format; ADD the test report. This Part shall be under the jurisdiction of National Standardization Technical Committee of Nonferrous Metals (SAC/TC 243). Drafting organizations of this Part. Xinjiang Wuxin Lithium Salt Development Co., Ltd., Jiangxi Dongpeng New Material Co., Ltd., Lithium-Calcium Company of CNNC Jianzhong Nuclear Fuel Co., Ltd., Beijing General Research Institute of Mining & Metallurgy, and Xinjiang Research Institute of Non ferrous

Metals. The main drafters of this Part. Ji Juanjuan, Wu Xiufen, Meng Cuiping, Zhong Haihua, Tong Zhongkun, Luo Aibao, Jiang Qiutao, Chen Yan, and Guan Yuzhen. The historical version replaced by this Part is as follows: - GB/T 11064.7-1989. GB/T 11064.7-2013 Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part

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