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

GB/T 11064.8-2013

Replacing GB 11064.8-1989

**Methods for chemical analysis of lithium carbonate, lithium hydroxide
monohydrate and lithium chloride - Part 8: Determination of silicon
content - Molybdenum blue spectrophotometric method**

碳酸锂、单水氢氧化锂、氯化锂化学分析方法 第8部分：硅量的测定 钼蓝分光光度法

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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

3.1 Hydrochloric acid (1+1), GR

3.5 Ammonium molybdate solution (50g/L). filtered if necessary, stored in plastic bottles.

3.6 Oxalic acid solution (50g/L). stored in plastic bottles.

3.7 Ascorbic acid solution (20g/L). prepared when it is used.

3.8 P-Nitrophenol indicator (1g/L). GB/T 11064.8-2013

3.9 Silicon standard stock solution. weigh 0.2140g of silicon dioxide (spectroscopically pure) which has been burnt at 1,000°C for 1h in advance, and then cooled down to room temperature. Place it into a platinum crucible in which there is 1g of anhydrous sodium carbonate (GR). Add 3g of anhydrous sodium carbonate to the silicon dioxide; put them in a high temperature furnace at 950°C~1,000°C to melt to be bright red and clear. Take them out; cool down; put them into a polytetrafluoroethylene (PTFE) beaker. Extract them with hot water; heat them until the solution is clear; then cool it down. Transfer it to a 1,000 volumetric flask; dilute it with water to the scale; shake up. Transfer it to a plastic bottle immediately. 1mL of this solution contains 100µg of silicon.

4 Instrumentation Spectrophotometer.

5 Samples

5.1 The samples of lithium carbonate and lithium chloride shall be preheated at 250°C~260°C for 2h; then cool 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.

6.4.4 Take some of the solution (6.4.3) to a cuvette according to Table

1. With water as reference, determine the absorbance at the 800nm wavelength of a

spectrophotometer.

6.4.5 Subtract the absorbance of the blank solution of the accompanying samples; find out the corresponding silicon content on the working curve.

6.5.1 Take the silicon standard solution (

3.11 or 3.10) according to Table 2 into a group of 50mL volumetric flasks, respectively. Add water to about 11mL; add 1 drop of p-Nitrophenol indicator (3.8). Neutralize it with sulfuric acid (3.2) to be colorless, and to be 3mL over the volume. The following shall be conducted according to 6.4.3.

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

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 8 of GB/T 11064. This Part is drafted in accordance with rules given in GB/T 1.1-2009. This Part replaces GB/T 11064.8-1989 "Lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Determination of silicon content - Molybdenum blue spectrophotometric method". Compared with GB/T 11064.8-1989, 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. Sichuan

Tianqi Lithium Industries Inc., Xinjiang Research Institute of Non ferrous Metals, and Xinjiang Wuxin Lithium Salt Development Co., Ltd. The main drafters of this Part. Gou Haixia, Luo Yuping, Zhu Xiangyu, Guan Yuzhen, Mi Yongqiang, Zhang Xiuli, and Sun Tao. The historical version replaced by this Part is as follows: - GB/T 11064.8-1989. GB/T 11064.8-2013 Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part

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