

ICS 77.120.99

CCS H64



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

GB/T 11064.4-2013

Replacing GB/T 11064.16-1989

Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part 4: Determination of potassium and sodium content Flame atomic absorption spectrometric method

碳酸锂、单水氢氧化锂、氯化锂化学分析方法 第4部分：钾量和钠量的测定 火焰原子吸收光谱法

Issued on: September 6, 2013

Implemented on: May 1, 2014

**Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;
Standardization Administration of China**

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Foreword

GB/T 11064.2-2013 "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

1 Scope

GB/T 11064.4-2013 is the Chinese national standard on methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - part 4: determination of potassium and sodium content flame atomic absorption spectrometric method, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 6 September 2013 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 May 2014. Classification: ICS 77.120.99, CCS H64. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken

from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Part of GB/T 11064 specifies the determination method of lithium carbonate, lithium hydroxide monohydrate, and potassium and sodium in lithium chloride. This Part applies to the determination of lithium carbonate, lithium hydroxide monohydrate, and potassium and sodium in lithium chloride. The determination range. 0.0010%~0.40%.

2 Method summary

The lithium carbonate and lithium hydroxide samples are dissolved in hydrochloric acid; lithium chloride sample is dissolved in water; in the dilute hydrochloric acid medium, at wavelength 589.0nm and 766.5nm of atomic absorption spectrometer, use air - acetylene flame and standard addition method to determine it.

3 Reagents

Unless otherwise indicated, all reagents used in this Part are guaranteed reagent, the water used is deionized water.

3.1 Hydrochloric acid (1+1).

3.2 Sodium standard stock solution. weigh 2.5420g of sodium chloride (reference reagent) which has been burned in advance at 500°C~600°C for 1.5h, and placed in dryer to cool to room temperature.

3.3 Sodium standard solution. Pipette 50.00mL of sodium standard stock solution (3.2) to a 500mL volumetric flask; use water to dilute to scale; mix well; store in the plastic GB/T 11064.4-2013

3.4 Potassium standard stock solution. weigh 1.9070g of potassium chloride (reference reagent) which has been burned in advance at 500°C~600°C for 1.5h, and placed in dryer to cool to room temperature.

3.5 Potassium standard solution. Pipette 50.00mL of potassium standard stock solution (3.4) to a 500mL flask; use water to dilute to scale; mix well; store in the plastic bottle. 1mL of this solution contains 100μm of potassium.

3.6 Potassium, sodium mixed standard solution. Pipette 50.00mL of sodium standard solution (3.3) and 50.00mL of potassium standard solution (3.5) to 500mL flasks respectively; use water to dilute to scale; shake well; store in plastic bottles. 1

4 Apparatus

Atomic absorption spectrometer. With sodium, potassium hollow cathode lamp. Under the optimal working condition of apparatus, any apparatuses that can reach following indicators

can be used.

5 Samples

5.1 Lithium carbonate sample and lithium chloride sample are baked in advance at 250°C~260°C for 2 h; place them in dryer to cool to room temperature.

5.2 Lithium hydroxide monohydrate shall be full-contained in plastic containers, GB/T 11064.4-2013

6 Analysis steps

6.1 Samples Weigh the sample according to Table 1, accurate to 0.0001g.

6.2 Times of determination Determine twice independently, then take the average value.

6.4 Determination

6.4.3 On the atomic absorption spectrometer, at wavelength of 589.0nm and 766.5nm, use air - acetylene flame, use water to adjust zero, determine the absorbance of test solution (6.4.2) according to concentration in ascending order.

6.4.4 Use sodium (potassium) as abscissa and absorbance as ordinate to draw graph; extend the drawn straight line downward to intersect with X-axis; the distance between the point of intersection and coordinate origin is the amount of sodium (potassium) in the test solution.

7 Calculation of analysis results

Use mass fraction of sodium (potassium))(KNa_w to calculate the content of sodium (potassium); use % to express the value; calculate it according to formula (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%.

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 4 of GB/T 11064. GB/T 11064.4-2013 This Part is drafted in accordance with rules given in GB/T 1.1-2009. The Part replaces GB/T 11064.4-1989 "Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part

4.Determination of potassium and sodium content - Flame atomic absorption spectrometric method" and GB/T 11064.16-1989 "Determination of calcium, magnesium, copper, lead, zinc, nickel, manganese, cadmium and aluminum content - Inductively coupled plasma atomic emission spectrometry". Compared with GB/T 11064.4-1989 and GB/T 11064.16-1989, the main changes of this Part are as follows: - REVISE the determination range to be "0.0010%~0.40%"; - CHANGE the weigh amount of sample; - REVISE "dissolve the sample with nitric acid" to be "dissolve with hydrochloric acid"; - DELETE the addition of cesium chloride; - REVISE "working curve method" to be "standard addition method"; - 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., Xinjiang Research Institute Of Non ferrous Metals, and Haimen Ronghui General Lithium Industry Co., Ltd. The main drafters of this Part. Ji Juanjuan, Zhao Lanfang, Deng Hongyun, Xu Dahuang, and Liu Qicheng. The historical versions replaced by this Part are as follows: - GB/T 11064.4-1989; - GB/T 11064.16-1989. GB/T 11064.4-2013 Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part

4.Determination of potassium and sodium content - Flame atomic absorption spectrometric method