

ICS 77.120.99

CCS H64



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

GB/T 11064.5-2013

Replacing GB/T 11064.5-1989

Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part 5: Determination of calcium 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5部分：钙量的测定 火焰原子吸收光谱法

Issued on: September 27, 2013

Implemented on: August 1, 2014

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

Table of Contents

1 Scope
2 Method summary
3 Reagents
4 Apparatus
5 Samples
6 Analysis steps
6.4 Determination
7 Calculation of analysis results
8 Precision
9 Determination of sulfate content - Barium sulfate nephelometry method; - Part
10 Determination of chloride content - Silver chloride nephelometry method; - Part
11 Determination of Acid-insolubles Content - Gravimetric method; - Part
15 Determination of fluoride content - Ion selective method; - Part

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

1 Scope

GB/T 11064.5-2013 is the Chinese national standard on methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - part 5: determination of calcium 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 27 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 August 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 calcium in lithium carbonate, in lithium hydroxide monohydrate, and in lithium chloride. This Part applies to the determination of calcium in lithium carbonate, in lithium hydroxide monohydrate, and in lithium chloride. The determination range. 0.0020%~0.35%.

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 of 422.7nm on atomic absorption spectrometer, use lanthanum salt and citric acid as release agent, 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 Lanthanum salt solution. weigh 5.864g of lanthanum oxide [%9.99)(32 OLaw]; place them in a 100mL beaker; add hydrochloric acid to dissolve and make it clear (heat it if necessary); transfer them in 500mL flask; use water to dilute to scale; shake well. 1mL of this solution contains 10mg of lanthanum.

3.5 Calcium standard solution. pipette 50.00mL of calcium standard stock solution (3.4); place in a 500mL flask; use water to dilute to scale; shake well. 1mL of this solution contains 100 μ m of calcium.

4 Apparatus

Atomic absorption spectrometer. with calcium 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 the dryer to cool to room temperature.

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

6.4.1 Place sample (6.1) in a 100mL polytetrafluoroethylene (PTFE) beaker; add about 5mL of water; cover the surface dish. Add hydrochloric acid (3.1) slowly according to Table 1; heat with low temperature and boil to remove carbon dioxide; cool down; transfer the test solution to a 100mL plastic flask; use water to dilute to scale; shake well.

7 Calculation of analysis results

Use mass fraction of calcium)(Caw to calculate the content of calcium; 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%. 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 5 of GB/T 11064. GB/T 11064.5-2013 This Part is drafted in accordance with rules given in GB/T 1.1-2009. The Part replaces GB/T 11064.5-1989 "Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part

5.Determination of calcium content - Flame atomic absorption spectrometric method".

Compared with GB/T 11064.5-1989, the main changes of this Part are as follows: - REVISE

the determination range "0.0060%~0.350%" to be "0.0020%~0.35%"; - REVISE "add 5mL of citric acid solution" to be "add 1mL of citric acid solution"; - 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., Ganzhou Nonferrous Metallurgy Research Institute, and Haimen Ronghui General Lithium Industry Co., Ltd. The main drafters of this Part. Guan Yuzhen, Zhao Fanglan, Lai Jian, Liu Hong, Deng Hongyun, Chen Xian'an, and Liu Yan. The historical version replaced by this Part is as follows: - GB/T 11064.5-1989. GB/T 11064.5-2013 Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part