



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

GB/T 11064.12-2013

Replacing GB/T 11064.12-1989

**Methods for chemical analysis of lithium carbonate, lithium
hydroxide monohydrate and lithium chloride - Part 12:
Determination of carbonate content - Acid-alkali titrimetric
method**

碳酸锂, 单水氢氧化锂, 氯化锂 化学分析方 法第 12 部分: 碳酸根量的测定
酸碱滴定法

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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 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;

manganese, cadmium and aluminum content - Inductively coupled plasma atomic emission spectrometry.

This Part is drafted in accordance with rules given in GB/T 1.1-2009.

This Part replaces GB/T 11064.12-1989 "Lithium hydroxide monohydrate - Determination of carbon dioxide content - Absorption titrimetric method".

Compared with GB/T 11064.12-1989, the main changes of this Part are as follows:

- 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., Sichuan Tianqi Lithium Industries Inc., Haimen Ronghui General Lithium Co., Ltd., Jiangxi Ganfeng Lithium Co., Ltd., and Xinjiang Research Institute of Non ferrous Metals.

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The historical version replaced by this Part is as follows:

1 Scope

This Part of GB/T 11064 specifies the determination method of carbonate content in lithium carbonate, in lithium hydroxide monohydrate and in lithium chloride.

This Part applies to the determination of carbonate content in lithium carbonate, in lithium hydroxide monohydrate, and in lithium chloride. The determination range: 0.15%~2.00%.

2 Method Summary

Dissolve the sample with water; take phenolphthalein as the indicator; use hydrochloric acid standard solution to titrate until the red in the solution is disappeared; then take methyl orange as the indicator. Continue to titrate it with hydrochloric acid standard solution until orange-red is stable, that is the end; calculate the amount of carbonate.

3 Reagents

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

3.1 Phenolphthalein indicator (10 g/L): weigh 1 g of phenolphthalein and dissolve it in 100 mL of ethanol (95%).

3.2 Methyl orange indicator (1 g/L): weigh 0.1 g of methyl orange, dissolve it in water at 70°C, cool it down, dilute it to 100 mL.

3.3 Hydrochloric acid solution (2 mol/L): pipette 167 mL of hydrochloric acid ($\rho = 1.19$ g/mL), place it in a 1000 mL volumetric flask, dilute it to the mark, shake it well.

3.4 Methyl red bromocresol green indicator: pipette 30 mL of bromocresol green solution (3.4.1) and 10 mL of methyl red solution (3.4.2), mix them well.

3.4.1 Bromocresol green solution: weigh 0.1 g of bromocresol green and dissolve it in 100 mL of ethanol.

3.4.2 Methyl red solution: weigh 0.2 g of methyl red and dissolve it in 100 mL of ethanol.

3.5 Hydrochloric acid standard titration solution [$c(\text{HCl}) = 0.10$ mol/L].

3.5.1 Preparation: pipette 8.3 mL of hydrochloric acid ($\rho = 1$).

4 Instrument

The sample shall be filled in a plastic container, sealed and stored. The storage period shall not exceed one month.

5 Analysis Steps

5.1 Samples

Weigh 5.0g of the sample, accurate to 0.0001g.

5.2 Number of Determination

Independently carry out two measurements; take the mean value.

5.3 Blank Test

6 Calculation of Analysis Results

The carbonate content is calculated in mass fraction $w(\text{CO}_3^{2-})$, expressed in %, according to equation (2).

c — The actual concentration of the hydrochloric acid standard titration solution (3.5), in moles per liter (mol/L);