



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

GB/T 11064.15-2013

Replacing GB/T 11064.15-1989

**Methods for chemical analysis of lithium carbonate, lithium
hydroxide monohydrate and lithium chloride - Part 15:
Determination of fluoride content - Ion selective method**

碳酸锂、单水氢氧化锂、氯化锂 化学分析方法 第15部分: 氟量的测定
离子选择电极法

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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:

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

The Part replaces GB/T 11064.15-1989 "Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part 15: Determination of fluoride content - Ion selective electrode method".

Compared with GB/T 11064.15-1989, the main changes of this Part are as follows:

- CHANGE "On Grignard drawing paper, take the volume of added fluorine standard solution as the abscissa, and the electrode potential as the ordinate to draw the curve." to "Create an Excel table, take the volume of fluorine standard solution B (V_x) as the abscissa, and $(V_s + V_x) \times 10^{-E/S}$ as the ordinate to draw the curve [$V_s = 50\text{mL}$, $S = 58$ (fluorine monovalent ions)]."
- 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 Research Institute of Nonferrous Metals, Beijing Research Institute of Mining & Metallurgy, and Jiangxi Ganfeng Lithium Co., Ltd.

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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 fluoride content in lithium carbonate.

This Part applies to the determination of fluoride content in lithium carbonate. The determination range is 0.0005% ~ 0.10%.

2 Method summary

The sample is decomposed with nitric acid; use sodium citrate nitrate solution as ionic strength adjustment solution; at the condition that the pH value is 7.0, use fluoride ion selective electrode to determine the fluorine content.

3 Reagents

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

3.1 Nitric acid (1 + 3), guarantee reagent.

3.2 Sulfuric acid (1 + 1), guarantee reagent.

3.3 Sulfuric acid (9 + 95), guarantee reagent.

3.4 Sodium hydroxide solution (40g/L).

3.5 Sodium citrate nitrate solution: Weigh 294g of sodium citrate dihydrate and 20g of potassium nitrate; place in a 500mL beaker; add water to dissolve; transfer to a 1000mL volumetric flask; dilute with water to the mark; mix well.

3.6 P-nitrophenol indicator (1g/L).

3.7 Fluorine standard stock solution: Weigh 2.210g of sodium fluoride that has been barked at 110°C for 2h and cooled in the dryer to room temperature; place in a 250mL beaker; dissolve with water and transfer into a 1000mL volumetric flask; dilute with water to the mark; mix well. Store in plastic bottles. 1mL of this solution contains 1mg of fluorine. 3.8 Fluorine standard Solution A: Pipette 25.00mL of fluorine standard stock solution (3.7); place in a 250mL volumetric flask;

4 Instruments

4.1 Ion meter: the accuracy is $\pm 0.2\text{mV}$.

4.2 pH meter.

4.3 Electromagnetic stirrer.

4.4 Fluorine electrode: before using, use fluorine-containing solution ($10\mu\text{g/mL} \sim 50\mu\text{g/mL}$) to soak for 30min.

4.5 Reference electrode: double salt bridge saturated calomel electrode, the outer salt bridge contains the reagent blank solution (3.10).

5 Samples

Samples shall be baked in advance at 250°C for 2h, and placed in the dryer to cool to room temperature.

6 Analysis steps