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

GB/T 11064.9-2023

Replacing GB/T 11064.9-2013

**Methods for chemical analysis of lithium carbonate, lithium hydroxide
monohydrate and lithium chloride - Part 9: Determination of sulfate
content - Barium sulfate turbidity method**

碳酸锂、单水氢氧化锂、氯化锂化学分析方法 第9部分：硫酸根含量的测定 硫酸
钡浊度法

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Foreword

This document was drafted in accordance with the rules of GB/T 1.1-2020 Directives for Standardization - Part

1.Rules for the Structure and Drafting of Standardizing Documents. This is Part 9 of GB/T 11064 Methods for Chemical Analysis of Lithium Carbonate, Lithium Hydroxide Monohydrate and Lithium Chloride. GB/T 11064 has issued the following parts.

- 1.Determination of Lithium Carbonate Content - Acid-alkali Titrimetric Method;
- 2.Determination of Lithium Hydroxide Content - Acid-alkali Titrimetric Method;
- 3.Determination of Lithium Chloride Content - Potentiometric Method;
- 4.Determination of Potassium and Sodium Content - Flame Atomic Absorption Spectrometric Method;
- 5.Determination of Calcium Content - Flame Atomic Absorption Spectrometric Method;
- 6.Determination of Magnesium Content - Flame Atomic Absorption Spectrometric Method;
- 7.Determination of Iron Content - 1,10-phenanthroline Spectrophotometric Method;
- 8.Determination of Silicon Content - Molybdenum Blue Spectrophotometric Method;

- 9.Determination of Sulfate Content - Barium Sulfate Nephelometry Method;
- 10.Determination of Chloride Content - Silver Chloride Nephelometry Method;
- 11.Determination of Acid-insolubles Content - Gravimetric Method;
- 12.Determination of Carbonate Content - Acid-alkali Titrimetric Method;

1 Scope

GB/T 11064.9-2023 is the Chinese national standard on methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - part 9: determination of sulfate content - barium sulfate turbidity 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 August 2023 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 March 2024. Classification: ICS 77.120.99, CCS H14. 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 document describes the method for the determination of sulfate content in lithium carbonate, lithium hydroxide monohydrate and lithium chloride. This document is applicable to the determination of sulfate content in lithium carbonate, lithium hydroxide monohydrate and lithium chloride. The range of determination is 0.050% ~ 0.800% (mass fraction).

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in this text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

GB/T 17433 Foundation Terms for Chemical Analysis of Metallurgical Products

3 Terms and Definitions

The terms and definitions defined in GB/T 17433 are applicable to this document.

4 Principle

In the presence of hydrochloric acid medium and a stabilizer, barium ions and sulfate radicals in the sample generate insoluble barium sulfate, which is suspended in the solution for a certain period of time.

5 Reagents and Materials

Unless it is otherwise specified, only analytically pure reagents are used.

5.1 Water, GB/T 6682, Grade-2.

5.3 Hydrochloric acid (1 + 1).

5.4 Hydrochloric acid (1 + 3).

5.5 Ammonia water (1 + 3).

5.6 Glycerol (1 + 4).

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

5.9 Lithium salt matrix solution. weigh-take

20.00 g of lithium carbonate [w (Li₂CO₃) 99.99%] in a 300 mL beaker, use water to moisten it, then, dropwise add 2 drops of p-nitrophenol indicator (5.7) and slowly add hydrochloric acid ($\rho =$

1.19 g/mL). When lithium carbonate is completely decomposed (the yellow color disappears), heat and boil it to drive out carbon dioxide, then, cool to room temperature, and transfer the test solution into a 100 mL volumetric flask, use water to dilute to the scale and shake it well.

6 Instruments and Equipment

Spectrophotometer, 3 cm cuvette.

7 Samples

7.1 The lithium carbonate and lithium chloride samples are baked at 250 C ~ 260 C for 2 h in advance, then, placed in a desiccator to cool to room temperature.

8 Test Procedures

8.1 Test Portions In accordance with Table 1, weigh-take the sample, accurate to 0.0001 g.

8.2 Parallel Test Carry out two tests in parallel and take the average value.

8.5 Drawing of Working Curve

8.5.1 Transfer-take 0 mL,

4.00 mL of sulfate standard solution (5.8) and

5.0 mL of lithium salt matrix solution (5.9), respectively place them into a set of 100 mL volumetric flasks, then, in accordance with 8.4.3, conduct the determination.

8.5.2 Transfer part of the solution (8.5.1) into a 3 cm cuvette and use the reagent blank as a reference; at a wavelength of 420 nm of the spectrophotometer, measure its absorbance. With the mass of sulfate as the x-coordinate and the absorbance as the y-coordinate, draw the working curve.

9 Processing of Test Data

The sulfate content, which is counted by the mass fraction of sulfate, shall be calculated in accordance with Formula (1).

10 Precision

10.1 Repeatability In terms of the determined values of two independent test results obtained under repeatability conditions, within the range of average values provided in Table 2, the absolute difference between the two test results does not exceed the repeatability limit (r), and the cases of exceeding the repeatability limit (r) does not exceed 5%.

10.2 Reproducibility In terms of the determined values of two independent test results obtained under reproducibility conditions, within the range of average values provided in Table 3, the absolute difference between the two test results does not exceed the reproducibility limit (R), and the cases of exceeding the reproducibility limit (R) does not exceed 5%.