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

GB/T 11064.13-2013

Replacing GB 11064.13-1989

**Methods for chemical analysis of lithium carbonate, lithium hydroxide
monohydrate and lithium chloride - Part 13: Determination of aluminum content -
Chromazurol S-cetylpyridine bromide spectrophotometric method**

碳酸锂、单水氢氧化锂、氯化锂化学分析方法 第13部分：铝量的测定 铬天青S-
溴化十六烷基吡啶分光光度法

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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 4. Determination of potassium and sodium content - Flame atomic absorption spectrometric method; - Part 5. Determination of calcium content - Flame atomic absorption spectrometric method; - Part 6. Determination of magnesium content - Flame atomic absorption spectrometric method; - Part 7. Determination of iron content - 1,10-phenanthroline spectrophotometric method; - Part 8. Determination of silicon content - Molybdenum blue spectrophotometric 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 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. Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part 13. Determination of aluminum content - Chromazurol S-cetylpyridine bromide spectrophotometric method

1 Scope

GB/T 11064.13-2013 is the Chinese national standard on methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - part 13: determination of aluminum content - chromazurol s-cetylpyridine bromide spectrophotometric 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 for aluminum content in lithium carbonate, lithium hydroxide monohydrate and lithium chloride. This Part applies to the determination of aluminum content in lithium carbonate, lithium hydroxide monohydrate and lithium chloride. The determination range. 0.00020%~0.050%.

2 Method summary

Use hydrochloric acid to decompose lithium carbonate and lithium hydroxide monohydrate samples; use water to dissolve lithium chloride sample; use boric acid, Zinc - ethylene diamine tetraacetic acid disodium as masking agents; in hexamethylenetetramine buffer solution medium, aluminum and chromazurol S-cetylpyridine bromide form into green complex; measure its absorbency at the position of spectrophotometer of which the wavelength is 630 nm. Interference measurement of fluorine needs to use sulfuric acid with smoke to remove.

3 Reagents

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

3.1 Sulfuric acid (1+1), guaranteed reagent.

3.2 Hydrochloric acid (1+1), guaranteed reagent.

3.3 Hydrochloric acid (1+47), guaranteed reagent.

3.4 Ammonia water ($\rho =$

0.90 g/mL), guaranteed reagent.

3.5 Ammonia water (1+10).

3.6 Boric acid solution (40 g/L), guaranteed reagent.

3.7 Hexamethylenetetramine solution (250 g/L).

3.8 Zinc - ethylene diamine tetraacetic acid disodium (Zn-Na₂EDTA). weigh

3.60 g of zinc powder [(Zn) w $\geq 99.9\%$]; use 40 mL of hydrochloric acid (3.2) to heat and dissolve; weigh

18.6 g of ethylene diamine tetraacetic acid disodium (reference reagent); add water to dissolve. Mix the two solutions; on pH meter, adjust pH value to 4.0; use water to dilute to 1000 mL; shake to uniform.

3.9 Mixed solution of chromazurol S-cetylpyridine bromide. Mix the equivalent volume of solution (3.9.1) and solution (3.9.2).

3.9.1 Chromazurol S solution (1 g/L). dissolve

0.25 g of chromazurol S into 250 mL of ethanol (1+4) solution.

3.9.2 Cetylpyridine bromide solution (4 g/L). dissolve 1 g of cetylpyridine bromide into 250 mL of warm water.

3.10 P-Nitrophenol indicator (1 g/L). use ethanol to prepare.

3.11 Aluminum standard stock solution. weigh 0.1000 g of pure aluminum [Al](w $\geq 99.9\%$]; put them into a 250 mL beaker; slowly add 10 mL of hydrochloric acid (3.2) to heat and dissolve; cool them; move them into a 1000 mL flask; use water to dilute to scale; shake to uniform. 1 mL of this solution contains 100 μg of aluminum.

3.12 Aluminum standard solution A. take

10.00 mL of aluminum standard stock solution (3.11) into 100 mL flask; add 1 mL of hydrochloric acid (3.2); use water to dilute to scale; shake to be uniform. 1 mL of solution contain 10 μg of aluminum.

3.13 Aluminum standard solution B. Place

10.00 mL of aluminum standard solution A (3.12) into a 100 mL flask; use water to dilute to scale; shake to uniform. 1 mL of this solution contains 1 μg of aluminum. Prepare when it is

to be used.

4 Apparatus

4.1 Spectrophotometer.

5 Samples

0m - The content of aluminum in blank solution found from working curve, in microgram (μg); m - The mass of sample, in gram (g); 0V - The total volume of the test solution, in milliliter (mL); 1V - The volume of the test solution, in milliliter (mL); The result shall be expressed to three decimal places, and expressed to four decimal places when the result is less than 0.01%.

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 3, adopts linear interpolation or extrapolation to obtain. Table