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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

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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;
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- Part 8: Determination of silicon content - Molybdenum blue spectrophotometric method;
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- 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

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 (ρ=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-Na_2EDTA): weigh 3.60 g of zinc powder [$w(Zn)$≥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 [$w(Al)$≥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.

4.2 pH meter.

5 Samples

m_0 — The content of aluminum in blank solution found from working curve, in microgram (μg);

m — The mass of sample, in gram (g);

V_0 — The total volume of the test solution, in milliliter (mL);

V_1 — 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 3

$w(\mathrm{Al})$/%	0.00064	0.0054	0.010
r/%	0.00005	0.0009	0.002

8.2 Allowable difference

The difference of analysis results among laboratories shall not be more than the allowable difference in Table 4.

Table 4

Mass fraction of aluminum/%	Allowable difference/%
0.0002~0.0008	0.0002
>0.0008~0.0020	0.0004
>0.0020~0.0050	0.0008
>0.0050~0.0080	0.0010
>0.0080~0.010	0.0020
>0.010~0.025	0.003
>0.025~0.050	0.005

9 Test report

The test report shall contain the following content:

— Sample;

— The number of this Standard;

— Analysis result and its expression;

— Difference with the basic analysis step;

— Anomalies observed in the determination;

— Test date.

_____ End _____

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