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Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride -

Part 14: Determination of arsenic content - Molybdenum blue spectrophotometric method

碳酸锂、单水氢氧化锂、氯化锂 化学分析方法

第 14 部分：砷量的测定 钼蓝分光光度法

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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 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

This Part is Part 14 of GB/T 11064.

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

Methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Part 14: Determination of arsenic content - Molybdenum blue spectrophotometric method

1 Scope

This Part of GB/T 11064 specifies the determination method of arsenic content in lithium carbonate.

This Part applies to the determination of arsenic content in lithium carbonate. The determination range is 0.00001% ~ 0.00020%.

2 Method summary

The sample is decomposed with hydrochloric acid; in the presence of potassium iodide and stannous chloride, in hydrochloric acid medium, use zinc particles to reduce; generated arsine is absorbed by iodine solution. In 0.2mol/L sulfuric acid medium, arsenic and molybdate form arsenic molybdenum yellow; use ascorbic acid to reduce it to arsenic molybdenum blue complex; measure its absorbance at where the wavelength of the spectrophotometer is 840nm.

3 Reagents

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

3.1 Arsenic-free zinc particles: granularity of $\phi 1.5\text{mm} \sim 3\text{mm}$.

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

3.3 Sulfuric acid (5 + 13), guarantee reagent.

3.4 Sulfuric acid (1 + 99), guarantee reagent.

3.5 Hydrochloric acid ($\rho = 1.19\text{g/ml}$), guarantee reagent.

3.6 Hydrochloric acid (1 + 1), guarantee reagent.

$$w(As) = \frac{(m_1 - m_0)V \times 10^{-6}}{mV_1} \times 100 \quad \dots\dots\dots(1)$$

Where:

m_1 - The arsenic content in the measurement sample solution refereed from the working curve, in micrograms (μg);

m_0 - The arsenic content in the blank solution refereed from the working curve, in micrograms (μg);

m - The mass of the sample, in grams (g);

V - The total volume of the sample solution, in milliliters (mL);

V_1 - The volume of the pipette sample solution, in milliliters (mL).

The result shall be expressed to five decimal places.

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 1, adopts linear interpolation or epitaxy method to obtain.

Table 1

$w(As)/\%$	0.000010	0.000074	0.00015
$r/\%$	0.000003	0.000004	0.000005

8.2 Allowable difference

The difference of analysis results among laboratories shall not be more than the allowable difference in Table 2.

Table 2

Mass fraction of arsenic/%	Allowable difference/%
0.00001 ~ 0.00005	0.00001
>0.00005 ~ 0.00020	0.00003

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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