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GB/T 11064.14-2013

Replacing GB/T 11064.14-1989

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 spectropotometric method

1 Scope

GB/T 11064.14-2013 is the Chinese national standard on methods for chemical analysis of lithium carbonate, lithium hydroxide monohydrate and lithium chloride - part 14: determination of arsenic content - molybdenum blue 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 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. 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

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

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