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Methods for Chemical Analysis of Lithium – Part 12: Determination of Impurity Element Contents – Inductively Coupled Plasma Atomic Emission Spectrometry

锂化学分析方法 第 12 部分：杂质元素含量的测定

电感耦合等离子体原子发射光谱法

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Foreword

This Document was drafted as per the rules specified in GB/T 1.1-2020 *Directives for Standardization – Part 1: Rules for the Structure and Drafting of Standardizing Documents*.

This Document is Part 12 of GB/T 20931 *Methods for Chemical Analysis of Lithium*. GB/T 20931 has published the following parts:

--- Determination of potassium content - Flame atomic absorption spectrometric method;

--- Determination of sodium content - Flame atomic absorption spectrometric method;

--- Determination of calcium content - Flame atomic absorption spectrometric method;

--- Determination of iron content - Orthopenanthroline spectrophotometric method;

--- Determination of silicon content - Molybdenum blue spectrophotometric method;

--- Determination of aluminum content – Chrome azurol S-cetylpyridinium bromide spectrophotometric method;

--- Determination of nickel content - α-Furil dioxime spectrophotometric method;

--- Determination of chlorine content - Thiocyanate spectrophotometric method;

--- Determination of nitrogen content - Mercuric potassium iodide spectrophotometric method;

--- Determination of copper content - Flame atomic absorption spectrometric method;

--- Determination of magnesium content - Flame atomic absorption spectrometric method;

--- Part 12: Determination of impurity element contents - Inductively coupled plasma atomic emission spectrometry.

Please note some contents of this Document may involve patents. The issuing agency of this Document shall not assume the responsibility to identify these patents.

This Document was proposed by China Nonferrous Metals Industry Association.

This Document shall be under the jurisdiction of National Technical Committee on Nonferrous Metals of Standardization Administration of China (SAC/TC 243).

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Methods for Chemical Analysis of Lithium – Part 12: Determination of Impurity Element Contents – Inductively Coupled Plasma Atomic Emission Spectrometry

1 Scope

This Document specifies the method for determining the content of impurity elements such as aluminum, calcium, copper, iron, potassium, magnesium, sodium, nickel, lead, and silicon in metallic lithium by inductively coupled plasma atomic emission spectrometry.

This Document is applicable to the determination of the content of impurity elements such as aluminum, calcium, copper, iron, potassium, magnesium, sodium, nickel, lead, and silicon in industrial-grade metallic lithium and battery-grade metallic lithium. The determination range is shown in Table 1.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) 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

JJG 768 Verification Regulation of Emission Spectrometer

3 Terms and Definitions

For the purposes of this Document, there are no terms and definitions that need to be defined.

4 Principle

The test material is dissolved in water. In the hydrochloric acid medium, the matrix matching standard curve method is used to determine the content of impurity elements such as aluminum, calcium, copper, iron, potassium, magnesium, sodium, nickel, lead and silicon in metal lithium using the inductively coupled plasma atomic emission spectrometer.

5 Reagents or Materials

Unless otherwise stated, only reagents that are confirmed to be guaranteed reagents are used in the analysis.

5.1 Water, GB/T 6682, Grade-1.

5.2 High-purity lithium carbonate (with mass fraction of no less than 99.999%).

5.3 Hydrochloric acid (1+1).

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

5.5 Standard storage solution of aluminum, calcium, copper, iron, potassium, magnesium, sodium, nickel, lead, silicon (1,000 μg/mL). Standard storage solutions can be purchased with certified standard substances or standard samples.

5.6 Mixed standard solution A: Respectively transfer 10.00 mL of each standard storage solution (5.5) into a 200 mL volumetric flask; dilute it with water to the scale; and mix well. 1 mL of this solution contains 50.00 μg of each measured element. If the silicon standard storage solution is sodium hydroxide or other alkaline matrix, the silicon standard solution must be prepared separately.

5.7 Mixed standard solution B: Respectively transfer 2.00 mL of each standard storage solution (5.5) into a 200 mL volumetric flask; dilute it with water to the scale; and mix well. 1 mL of this solution contains 10.00 μg of each measured element. If the silicon standard storage solution is sodium hydroxide or other alkaline matrix, the silicon standard solution must be prepared separately.

6 Instruments and Equipment

6.1 Inductively coupled plasma atomic emission spectrometer: The equipment performance

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