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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 23367.1-2024

Replacing GB/T 23367.1-2009

Methods for the chemical analysis of lithium cobalt oxide - Part 1: Determination of the cobalt content - EDTA titration and potentiometric titration

钴酸锂化学分析方法 第1部分：钴含量的测定 EDTA滴定法和电位滴定法

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Foreword

Lithium-ion batteries are the core of 3C consumer electronic products, and lithium cobalt oxide positive electrode materials are important raw materials for 3C lithium-ion batteries. Therefore, vigorously developing lithium cobalt oxide materials is a prerequisite for achieving high-quality development of 3C consumer electronic products. GB/T 23367.1-2009 "Methods for chemical analysis of lithium cobalt oxide - Part 1: Determination of cobalt content - EDTA titration" was issued in March 2009 and implemented in January 2010. It has been more than ten years since then. The implementation and application of this Standard has unified the determination method of cobalt content in lithium cobalt oxide, significantly reducing the communication cost and trade cost of both supply and demand sides for the determination of cobalt content in lithium cobalt oxide products. In recent years, with the iteration of lithium cobalt oxide products and the upgrading of testing technology, the determination method of cobalt content in lithium cobalt oxide products has also gradually developed from the traditional EDTA manual titration to the EDTA automatic titration and redox potentiometric titration, and more stringent requirements have been put forward for the test conditions. GB/T 23367 "Methods for chemical analysis of lithium cobalt oxide" consists of 2 parts. - Part 1: Determination of cobalt content - EDTA titration and potentiometric titration; - Part 2: Determination of lithium, nickel, manganese, magnesium, aluminium, iron, sodium, calcium and copper content - Inductively coupled plasma atomic emission spectrometry. This document is formulated based on the needs of domestic and foreign lithium cobalt oxide trade. The revision of this Standard can make the determination method of cobalt content in lithium cobalt oxide products further adapt to the current level of technological development, promote the progress of related testing technologies, provide technical guidance for related domestic industries, and promote the continuous healthy and stable development of the lithium-ion battery positive electrode material industry. It is convenient for suppliers and customers to use unified analytical methods for testing, which is conducive to reducing commercial disputes caused by differences in testing methods, and has good economic and social benefits. Methods for chemical analysis of lithium cobalt oxide - Part 1: Determination of cobalt content - EDTA titration and potentiometric titration

1 Scope

China's national method for determining the cobalt content of lithium cobalt oxide, by EDTA titration and by potentiometric titration. It is Part 1 of GB/T 23367 and specifies the determination of cobalt in the cathode material. Cobalt is the expensive element in a lithium-ion battery and, in this material, the majority constituent: lithium cobalt oxide is roughly sixty per cent cobalt by mass, and cobalt has been among the most volatile commodities in the world, with supply concentrated in one country and a price that has moved by a factor of three within a year. So the cobalt assay is not a quality check but the basis on which the material is bought and sold, and the tonnage of a shipment is settled on it. That is why the method is titrimetric rather than instrumental. A titration is an absolute measurement: the analyte reacts stoichiometrically with a standardised reagent and the result depends on the purity of that reagent and on the accuracy of a burette, not on a calibration curve built from standards that might themselves be wrong. For a determination with money attached, at a concentration where precision of a fraction of a per cent matters, that traceability is worth more than the speed of an instrumental method. Two routes are given: complexometric titration with EDTA against a visual indicator, which needs no instrument at all, and potentiometric titration, which locates the endpoint electrically and is more precise and less dependent on the analyst. The standard fixes the dissolution of a material that is not easily dissolved, the removal or masking of the interferences, the standardisation of the titrant, both endpoint procedures and the precision. Issued on 28 November 2024 and in force since 1 June 2025, it replaces GB/T 23367.1-2009.

This document specifies the method for determining the cobalt content in lithium cobalt oxide. Method 1 is EDTA titration, and method 2 is potentiometric titration. This document applies to the determination of cobalt content in lithium cobalt oxide. Determination range: 56.00 % ~ 62.00 %.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

GB/T 17433 Foundation terms for chemical analysis of metallurgical products

3 Terms and definitions

For the purpose of this document, the terms and definitions defined in GB/T 17433 apply.

4 Method 1: EDTA titration

4.1 Principle After the sample is dissolved in hydrochloric acid (or a mixed solution of nitric acid and perchloric acid), in an ammonia-ammonium chloride buffer solution, with murexide as the indicator and EDTA standard titration solution as the titrant, the cobalt content is calculated based on the volume of EDTA standard titration solution consumed. It can use EDTA manual titration or EDTA automatic titration.

4.2 Reagents or materials

4.2.1 Water, GB/T 6682, grade 3 and above.

4.2.2 Hydrochloric acid (1 + 1).

4.2.3 Nitric acid (rho =

1.42 g/mL), analytical grade or above.

4.2.4 Perchloric acid (rho =

1.67 g/mL), analytical grade or above.

4.2.5 Cobalt standard solution: WEIGH 1.0000 g of metallic cobalt (wCo >= 99.98 %) in a 400 mL beaker, ADD 30 mL of hydrochloric acid (4.2.2), COVER with a watch glass, DISSOLVE completely at low temperature, COOL to room temperature, TRANSFER to a 1000 mL volumetric flask, DILUTE to the mark with water, and MIX well. 1 mL of this solution contains

1.0 mg of cobalt. Commercially available certified cobalt standard solution can also be used.

4.2.6 Ammonia-ammonium chloride buffer solution (pH ~ 10): WEIGH

54.5 g of ammonium chloride and dissolve in water, ADD 350 mL of ammonia, DILUTE to 1000 mL with water, and MIX well.

4.2.7 Murexide indicator: WEIGH

0.4 g of murexide, ADD 50 g of potassium sulfate, and GRIND thoroughly in a mortar. Store in a desiccator.

4.2.8 Preparation of disodium ethylenediaminetetraacetate ($C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O$, referred to as EDTA) standard titration solution (about

0.01 mol/L). WEIGH

3.72 g of EDTA into a 400 mL beaker, ADD water to dissolve with slight heat, COOL to room temperature, TRANSFER to a 1000 mL volumetric flask, DILUTE to the mark with water, and MIX well.

4.2.9 Standardization of disodium ethylenediaminetetraacetate ($C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O$, referred to as EDTA) standard titration solution (about

0.01 mol/L) (calibration before use): TRANSFER

15.00 mL of cobalt standard solution (4.2.5) into the titration cup, record as V1. ADD 50 mL of water and 20 mL ~ 22 mL of EDTA standard titration solution (preferably 3 mL ~ 5 mL before the end point of the reaction process), then ADD 10 mL of ammonia-ammonium chloride buffer solution (4.2.6), and finally ADD about

0.1 g of murexide indicator (4.2.7). If EDTA manual titration is selected, directly titrate with EDTA standard titration solution until the solution changes from orange-yellow to purple-red, which is the end point. If EDTA automatic titration is selected, insert the photometric electrode into the automatic potentiometric titrator, and titrate to the end point with EDTA standard titration solution under stirring according to the titration end point