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Determination of magnetic foreign metal particles in lithium carbonate, lithium hydroxide monohydrate and lithium chloride - Cleanliness meter test method

碳酸锂、单水氢氧化锂、氯化锂中磁性异物金属颗粒的测定 洁净度仪测试法

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Foreword

This document was drafted in accordance with the provisions of GB/T 1.1-2020 *Directives for standardization - Part 1: Rules for the structure and drafting of standardizing documents*.

Please note that some of the contents of this document may involve patents. The issuing organization of this document does not assume the responsibility for identifying patents.

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1 Scope

This document describes the method for testing the content of magnetic foreign metal particles in lithium carbonate, lithium hydroxide monohydrate and lithium chloride using a cleanliness meter.

This document is applicable to the determination of the content of magnetic foreign metal particles in lithium carbonate, lithium hydroxide monohydrate and lithium chloride. The measurement range is 10 particles/kg~500 particles/kg.

2 Normative references

The provisions of the following documents constitute the essential clauses of this document through normative references in this text. Among them, for referenced documents with dates, only the versions corresponding to the dates are applicable to this document; for referenced documents without dates, the latest versions (including all amendments) are 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 judgment of limiting values

GB/T 25915.1-2021 Cleanrooms and associated controlled environments - Part 1: Classification of air cleanliness by particle concentration

GB/T 41481-2022 Road vehicles - Cleanliness of components and systems

3 Terms and definitions

The terms and definitions defined in GB/T 41481-2022 and the following apply to this document.

3.1 particle standard

Standard particles of known shape and size used for testing and calibration of optical analysis systems.

4 Principle

The test specimen is dispersed in demagnetizing water in a clean room, and the magnetizable magnetic foreign metal particles in the test specimen are adsorbed and enriched by a magnetic rod covered with a heat-shrink tubing. After the magnetic rod is separated from the heat shrink tubing, the magnetizable magnetic foreign metal particles on the tubing are collected through a filter membrane, and the particles on the filter membrane are scanned and analyzed using a non-approximate algorithm on a cleanliness meter. After analyzing the two images obtained by scanning in parallel and cross arrangement states, metals, non-metals and fibers are automatically identified, thereby determining the content of magnetizable magnetic foreign metal particles.

5 Test conditions

The test shall be carried out in a clean room whose cleanliness meets the ISO 8 grade requirements in GB/T 25915.1-2021.

6 Reagents or materials

Unless otherwise stated, only reagents of guaranteed grade or higher are used in the analyses.

6.1 Demagnetizing water: GB/T 6682, grade 1, adsorb with a magnetic rod [the magnetic induction intensity is not less than 1.2 T (12000 Gs)] for 5 minutes.

6.2 Hydrochloric acid (1+1).

6.3 Ceramic scissors (or ceramic knife).

6.4 Plastic mixing barrel: volume=10 L; (diameter × height)=246 mm×325 mm.

6.5 Heat shrink tubing: with a diameter of 25 mm, a wall thickness of 0.31 mm, and a width of 41 mm.

6.6 Sealing film: PE breakpoint cling film, 20 cm×10 cm.

6.7 Filter membrane (water system): (pore size)=5 μm and diameter=47 mm.

6.8 Slide: size=70 mm×70 mm and visible area=55 mm×55 mm.

6.9 Magnetic rod [the magnetic induction intensity is not less than 0.6 T (6000 Gs)]:

9.3 Determination

9.3.1 Test preparation: Take out a rubber mat and spread it on the workbench. Place the magnetic rod (6.10) on the rubber mat and roll it to absorb the magnetic material.

9.3.2 Plastic-sealed magnetic rod: Take a heat shrink tubing (6.5), seal it on the heat-sealing machine (7.7), install demagnetizing water (6.1), make sure the seal is leak-proof, then put it on the magnetic rod (6.9), and then seal the other end. Place the sealed magnetic rod on a clean rubber mat.

9.3.3 Add sample: Take a clean plastic mixing barrel (6.4) and add (6.0±0.2) L of demagnetizing water (6.1) into the barrel. Slowly add the test specimen (9.1) into the barrel, then gently place the sealed plastic magnetic rod (9.3.2) into the barrel and seal the plastic barrel.

9.3.4 Extraction: Place the sealed plastic mixing barrel (9.3.3) into the roller machine (7.1) and extract for 15 min at a speed (measured by the speed of the barrel) of (60±5) r/min.

9.3.5 Rinsing: Take out the plastic-sealed magnetic rod after extraction (9.3.4), take another clean plastic mixing barrel (6.4), add (6.0±0.2) L of demagnetizing water (6.1) into the barrel, then gently place the plastic-sealed magnetic rod (9.3.4) into the barrel, seal the plastic barrel, place the sealed plastic barrel into the roller machine (7.1), and rinse for 15 minutes at a speed (measured by the speed of the barrel) of (60±5) r/min.

9.3.6 Collection: Take out the rinsed plastic-sealed magnetic rod (9.3.5) and place it in a clean 500 mL beaker; use ceramic scissors (6.3) to cut one end of the heat shrink tubing, cut both ends of the opening, bend the heat shrink tubing, pull out the magnetic rod, remove the heat shrink tubing, and use demagnetizing water (6.1) to rinse all the magnetic particles in the heat shrink tubing into the beaker. Use a magnetic block (6.11) on the outside of the bottom of the beaker to adsorb and aggregate the magnetic particles, pour out the aqueous solution, and repeat the washing 5 times. The magnetic particles remain in the beaker.

9.3.7 Acid cleaning: Pour (15±2) mL of hydrochloric acid (6.2) into the beaker containing the magnetic particles, seal the mouth of the beaker with a sealing film (6.6), wrap tape around the edge, and place it in an ultrasonic cleaner (7.5) for 2 min (power=200 W, frequency=53 kHz).

9.3.8 Water washing: After the ultrasonic treatment is completed, take out the beaker, add 50 mL of demagnetizing water (6.1) to the beaker, and use a magnetic block (6.11) on the outside of the bottom of the beaker to adsorb and aggregate the magnetic particles. Pour the solution into the waste liquid bucket, and inject (50±10) mL of demagnetizing water (6.1) into the beaker to wash the magnetic particles. Repeat the washing 5 times. Finally, add (10±2) mL of demagnetizing water (6.1) to the beaker and wait for suction

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