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Lithium carbonate made of brine

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Lithium carbonate made of brine

1 Scope

This document specifies the classification, requirements, test methods, inspection rules and signs, labels and accompanying documents, packaging, transportation and storage for lithium carbonate made of brine.

This document applies to lithium carbonate made of brine.

NOTE: This product is mainly used in lithium-ion battery material production, glass manufacturing, ceramics production and refrigeration, welding, lithium alloy and metallurgical continuous casting and other fields.

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 191-2008, *Packaging - Pictorial marking for handling of goods*

GB/T 3050-2000, *Inorganic chemical products for industrial use - General method for determination of chloride content - Potentiometric method*

GB/T 6678, *General principles for sampling chemical products*

GB/T 6682-2008, *Water for analytical laboratory use - Specification and test methods*

GB/T 8170, *Rules of rounding off for numerical values and expression and judgement of limiting values*

GB/T 19077, *Particle size analysis - Laser diffraction methods*

HG/T 3696.1, *Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis - Part 1: Preparations of standard volumetric solutions*

HG/T 3696.2, *Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis - Part 2: Preparations of standard solutions for impurity*

7.3.2 Reagents

7.3.2.1 Hydrochloric acid solution: 1+4.

7.3.2.2 Alkali metal iron periodate solution. Dissolve 2g of potassium periodate in 10mL of potassium hydroxide solution (80g/L). Use water to dilute to 50mL. Add 3mL of ferric chloride solution (100g/L). After well stirring, use potassium hydroxide solution (80g/L) to dilute to 100mL.

7.3.2.3 Sodium chloride saturated solution.

7.3.3 Test steps

7.3.3.1 Preparation of test solution

Weigh about 2g of sample, to the nearest of 0.1g. Place in a 100mL tall beaker. Add water to moisten. Add hydrochloric acid solution dropwise until the sample is just dissolved. Use water to dilute to about 50mL. Mix well.

7.3.3.2 Identification

Add 1 drop of test solution to the watch glass. Then, add 1 drop of saturated sodium chloride solution and 2 drops of alkali metal ferric periodate solution dropwise on it. Use alcohol lamp or other safe heat source to heat to 45℃~50℃. Hold for 15s~20s. Compared with the blank test, the solution shall be yellow-white turbid.

In the blank test, except that the test solution is not added, the amount of other reagents added is exactly the same as that of the test solution.

7.4 Determination of lithium carbonate content

7.4.1 Principle

After adding water to the sample, use bromocresol green-methyl red as the indicator solution. Use hydrochloric acid standard titration solution to titrate. Calculate the lithium carbonate content according to the consumption of hydrochloric acid standard titration solution.

7.4.2 Reagents or materials

7.4.2.1 Hydrochloric acid standard titration solution: $c(HCl)\approx0.5mol/L$.

7.4.2.2 Bromocresol green-methyl red indicator solution.

7.4.2.3 Carbon dioxide free water.

7.4.3 Test steps

Weigh about 0.6g of the sample that has been dried at 250℃~260℃ to constant mass, to the nearest of 0.0002g. Place it in a conical flask. Add 50mL of carbon dioxide-free water and 10 drops of bromocresol green-methyl red indicator solution. Use hydrochloric acid standard titration solution to titrate until the solution changes from green to dark red. Boil for 2min. After cooling, continue to use hydrochloric acid standard titration solution to titrate to dark red as the end point. Conduct the blank test at the same time.

7.4.4 Test data processing

Lithium carbonate is calculated as the mass fraction w_1 of lithium carbonate (Li_2CO_3) according to formula (1):

$$w_1 = \frac{c\,[(V_1 - V_0)/1\,000]\,M}{m} \times 100\% \quad \cdots\cdots\cdots\cdots\cdots\cdots (1)$$

Where,

c - The exact value of the concentration of hydrochloric acid standard titration solution, in moles per liter (mol/L);

V_1 - The value of the volume of hydrochloric acid standard titration solution consumed by titrating the sample solution, in milliliters (mL);

V_0 - The value of the volume of hydrochloric acid standard titration solution consumed by the blank test, in milliliters (mL);

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

M - The value of the molar mass of lithium carbonate, in grams per mole (g/mol) [M ($1/2Li_2CO_3$) = 36.94].

Take the arithmetic mean of the parallel determination results as the determination result. The absolute difference between the two parallel determination results shall not be greater than 0.2%.

7.5 Determination of sulfate content

7.5.1 Principle

In the acidic medium, sulfate ions and barium ions form insoluble barium sulfate. When the sulfate ion content is low, barium sulfate will be in suspension within a certain period of time, making the solution turbid. Visually compare the turbidity of the sample solution to the standard turbidity solution.

7.5.2 Reagents or materials

7.5.2.1 Hydrochloric acid solution: 1+1.

7.5.2.2 Sodium chloride-hydrochloric acid-glycerol mixed solution. Weigh 12g of sodium chloride and dissolve it in 40mL of water. Add 1mL of hydrochloric acid. Use glycerol to dilute to 100mL. Mix well. If glycerol has a yellow color, it shall use activated carbon to decolorize in advance.

7.5.2.3 Barium chloride solution: 250g/L.

7.5.2.4 Sulfate standard solution: 1mL of solution contains 0.10mg of sulfate (SO_4). Pipette 10mL of sulfate standard stock solution prepared according to HG/T 3696.2. Place in a 100mL volumetric flask. Use water to dilute to the scale. Shake well.

7.5.3 Test steps

7.5.3.1 Preparation of test solution

Weigh about 2.5g ± 0.01g of sample. Place in a 100mL tall beaker. Use a little water to moisten. After adding a watch glass, slowly add hydrochloric acid solution dropwise until the sample is completely dissolved. Add 10mL of water. Heat to boil. After cooling, transfer the entire solution to a 250mL volumetric flask. Use water to dilute to the scale. Shake well. After the solution is still, use medium-speed qualitative filter paper to dry filter. Discard the first 10mL of filtrate.

7.5.3.2 Test

Pipette 10mL of test solution. Place in a 50mL colorimetric tube. Add water to about 30mL. Add 1mL of hydrochloric acid solution, 10mL of sodium chloride-hydrochloric acid-glycerol mixed solution. Shake well. Add 3mL of barium chloride solution dropwise while shaking. Use water to dilute to the scale. Shake well. Place for 5min. The turbidity shall not be greater than the standard turbidity comparison solution.

The standard turbidity comparison solution is to take 0.10mL (type I), 0.50mL (type II), and 1.00mL (type III) of sulfate standard solution. Treat it in the same way as the sample solution.

7.6 Determination of chloride content

7.6.1 Potentiometric titration (arbitration method)

7.6.1.1 Principle

See the provisions in Chapter 3 of GB/T 3050-2000.

7.6.1.2 Reagents or materials

Prepare according to the provisions of Chapter 4 in GB/T 3050-2000.

7.6.1.3 Instruments and equipment

Prepare according to the provisions of Chapter 5 in GB/T 3050-2000.

7.6.2.2.1 Nitric acid solution: 1+4.

7.6.2.2.2 Silver nitrate solution: 17g/L.

7.6.2.2.3 Chloride standard solution: 1mL of the solution contains 0.10mg of chlorine (Cl). Pipette 10mL of chloride standard stock solution prepared according to HG/T 3696.2. Place in a 100mL volumetric flask. Use water to dilute to the scale. Shake well. This solution is prepared before use.

7.6.2.3 Test steps

7.6.2.3.1 Preparation of test solution

Weigh about 2.5g ± 0.01g of sample. Place in a 100mL tall beaker. Use a little water to moisten. After adding a watch glass, slowly add nitric acid solution dropwise until the sample is completely dissolved. Heat to boil. After cooling, transfer all to a 250mL volumetric flask. Use water to dilute to the scale. Shake well. After the solution is still, use medium-speed qualitative filter paper to dry filter. Discard the first 10mL of filtrate.

7.6.2.3.2 Test

Pipette 10mL of test solution. Place in a 50mL colorimetric tube. Add water to about 40mL. Add 1mL of nitric acid solution, 1mL of silver nitrate solution. Use water to dilute to the scale. Shake well. Place for 2min. The turbidity shall not be greater than the standard turbidity comparison solution.

The standard turbidity comparison solution is to take 0.20mL (type I), 0.50mL (type II) and 1.00mL (type III) of chloride standard solution. Treat it in the same way as the sample solution.

7.7 Determination of content of hydrochloric acid insoluble matter

7.7.1 Principle

Weigh a certain amount of sample and dissolve it in hydrochloric acid solution. The filtered residue is dried at a certain temperature to a constant mass. Determine the content of hydrochloric acid insoluble matter.

7.7.2 Reagents or materials

7.7.2.1 Hydrochloric acid solution: 1+1.

7.7.2.2 Nitric acid solution: 1+4.

7.7.2.3 Silver nitrate solution: 17g/L.

7.7.2.4 Methyl orange indicator solution: 1g/L.

7.7.3 Instruments and equipment

7.9.2.4 Standard mixed solution of metal ions and boron and silicon: 1mL of the solution contains 10μg of sodium (Na), potassium (K), calcium (Ca), magnesium (Mg), iron (Fe), manganese (Mn), copper (Cu), boron (B) and silicon (Si) each. Pipette 10mL of the standard solution of each ion in the standard solution of metal ions, boron and silicon. Place in the same 100mL volumetric flask. Use water to dilute to the scale. Shake well.

7.9.2.5 Water: Grade two water specified in GB/T 6682-2008.

7.9.3 Instruments and equipment

Inductively coupled plasma atomic emission spectrometer (ICP-OES): Determine 1mg/L or 10mg/L multi-element mixed standard solution. The RSD of 10 repeated determinations is ≤0.5%. See Annex A for the commonly used spectral lines and detection limits of ICP-OES for each measured ion.

7.9.4 Test steps

7.9.4.1 Preparation of test solution A

Weigh about 5g of sample, to the nearest of 0.01g. Put in a 400mL beaker. Add a little water to moisten. After adding a watch glass, slowly add hydrochloric acid solution dropwise until the sample is completely dissolved. Heat to boil. Cool to room temperature. Transfer all into a 250mL (V_1) volumetric flask. Use water to dilute to the scale. Shake well. This solution is test solution A, used for the determination of metal ions, boron and silicon contents.

7.9.4.2 Test

Pipette 20mL (V_2) of test solution A respectively (see 7.9.4.1). Place in six 100mL volumetric flasks. Add 5mL of nitric acid solution to each. Then add 0.0mL, 1.00mL, 2.00mL, 3.00mL, 4.00mL, 5.00mL of metal ions and standard mixed solutions of boron and silicon, respectively. Use water to dilute to the scale. Shake well.

Turn on the inductively coupled plasma atomic emission spectrometer (ICP-OES). After the operation is stable, under the selected optimized conditions, measure the emission spectrum intensities of different masses of each ion separately. Take the measured ion mass (A) as the abscissa, and the corresponding emission spectrum intensity (I) as the ordinate, to draw a working curve. Intersect the reverse extension of the curve with the abscissa. The distance between the intersection point and the origin is the measured ion mass. The schematic diagram is shown in Figure 1. Calculate to obtain the content of each ion.

inductively coupled plasma atomic emission spectrometer (ICP-OES) and standard working curve method to determine the content of magnetic substances.

7.10.2 Reagents or materials

7.10.2.1 Nitric acid solution: 1+19. Use premium pure nitric acid to prepare.

7.10.2.2 Aqua regia: Mix 1 volume of nitric acid and 3 volumes of hydrochloric acid. Prepare before use. Use premium pure nitric acid and premium pure hydrochloric acid to prepare.

7.10.2.3 Magnetic substance standard solution: 1mL of the solution contains 0.1mg of iron (Fe), zinc (Zn), chromium (Cr), and nickel (Ni) each. Respectively pipette 10mL of iron (Fe), zinc (Zn), chromium (Cr), nickel (Ni) standard stock solutions prepared according to HG/T 3696.2. Place in 100mL volumetric flasks separately. Use water to dilute to the scale. Shake well.

7.10.2.4 Magnetic substance standard mixed solution: 1mL of the solution contains 10μg of iron (Fe), zinc (Zn), chromium (Cr) and nickel (Ni) each. Pipette 10mL of standard solution of each ion in the magnetic substance standard solution, respectively. Place in the same 100mL volumetric flask. Use water to dilute to the scale. Shake well.

7.10.2.5 Water: Grade two water specified in GB/T 6682-2008.

7.10.3 Instruments and equipment

7.10.3.1 Inductively coupled plasma optical emission spectrometer (ICP-OES): Determine 1mg/L or 10mg/L multi-element mixed standard solution. The RSD of 10 repeated determinations is ≤0.5%. See Annex A for the commonly used spectral lines and detection limits of ICP-OES for each measured ion.

7.10.3.2 Magnetic rod: Appearance size is about ϕ17mm×58mm. Magnetic induction intensity is 0.6T~0.8T. The outer layer is covered with Teflon. Before use, place in a 250mL tall beaker. Add 12mL of aqua regia and an appropriate amount of water to cover the magnetic rod. In a constant temperature water bath at 90°C±0.5°C, heat for 30min and take it out. Use water to wash it clean.

7.10.3.3 Ultrasonic cleaning (oscillation) device: The frequency can be set to 100Hz.

7.10.3.4 Constant temperature water bath: The temperature can be controlled at 90°C±0.5°C.

7.10.3.5 Sample bottle: 500mL wide-mouth plastic bottle with a sealing cap. Before use, use nitric acid solution to soak for more than 2h. Use water to wash it clean.

7.10.3.6 Colorimetric tube: 50mL, equipped with a cap.

7.10.4 Test steps

7.10.4.1 Preparation of test solution B

Weigh 200g~300g of sample, to the nearest of 0.1g. Place in a sample bottle. Add a little water to moisten. Add the magnetic rod and 400mL of water. Cover with the sealing cap. Make sure the seal is tight. Shake the sample bottle by hand for 2min~3min until the sample is evenly mixed. Place the sample bottle on an ultrasonic cleaner (shaker). Under the condition of frequency of 100Hz, carry out the adsorption and enrichment for 30min (or use the method with the same enrichment effect for adsorption and enrichment).

Remove the sample bottle. Use another magnetic rod outside the bottle to hold the magnet rod that is inside the bottle. Open the sealing cover. Carefully discard the slurry in the bottle. Remove the magnet rod outside the bottle. Use a clean, non-metallic tweezer to take the magnet rod from the bottle. Transfer to a 250mL tall beaker.

Add an appropriate amount of water to the beaker along the wall of the beaker to cover the magnetic rod. Use another magnet rod at the bottom of the beaker to hold the magnet that is inside the beaker. After rolling back and forth 10 times, discard the wash water. Repeat the above steps at least 3 times. Drain as much water as possible each time. Use a clean non-metallic tweezer to move the rinsed magnetic rod to the colorimetric tube. Add 12mL of aqua regia and an appropriate amount of water to cover the magnetic rod. Cover the colorimetric tube cap (do not cover tightly). Place in a constant temperature water bath at a temperature of 90°C ± 0.5°C. Heat for 30min. Take out the colorimetric tube. Cool to room temperature.

Use another magnetic rod outside the colorimetric tube to hold the magnetic rod that is in the colorimetric tube. Open the cover of the colorimetric tube. Use water to rinse the cover. Carefully transfer all the solution in the colorimetric tube into a 100mL volumetric flask. Use a small amount of water to rinse the colorimetric tube and the magnetic rod. Repeat 3 times. Combine the wash water in the volumetric flask. Use water to dilute to the scale. Shake well. This solution is test solution B, used for the determination of magnetic foreign matters.

7.10.4.2 Preparation of working curve solution

in six 100mL volumetric flasks, add 0.0mL, 0.20mL, 0.50mL, 1.00mL, 2.00mL, 5.00mL of magnetic substance standard mixed solution and 12mL of aqua regia, respectively. Use water to dilute to the scale. Shake well.

7.10.4.3 Test

Turn on the inductively coupled plasma atomic emission spectrometer (ICP-OES). After the operation is stable, under the selected optimized conditions, determine the emission spectrum intensities of different masses of each ion, respectively. Take the measured ion mass (B) as the abscissa, and the corresponding emission spectrum intensity (I) as the ordinate, to draw a working curve.

material, basically the same production conditions, continuous production or the same specification produced by the same team shall be selected as a batch. Each batch of products shall not exceed 20t.

8.3 Determine the number of sampling units in accordance with the provisions of GB/T 6678. Each package is a package unit. When sampling, insert the sampler obliquely from the top of the packaging bag to 3/4 of the depth of the material layer for sampling. After mixing the collected samples well, reduce it to not less than 1000g according to the method of quartering. Divide the sample into two clean, dry containers. Seal. Label them, indicating manufacturer's name, product name, type, batch number, date of sampling and name of sampler. One is for inspection and the other for future reference. The storage time shall be determined according to the needs of the manufacturer.

8.4 When the inspection result does not meet the indicator requirements specified in Chapter 6, it shall be resampled from twice the amount of packaging for reinspection. If one of the indicators does not meet the re-inspection results, the whole batch of products shall be rejected.

8.5 Use the rounding value comparison method specified in GB/T 8170 to judge whether the test results meet the indicator requirements specified in Chapter 6.

9 Signs, labels and accompanying documents

9.1 There shall be strong and clear signs and labels on the packaging for lithium carbonate made of brine. The content shall include at least: manufacturer name, factory address, product name, type, net content, batch number (or production date), this document number, and the "fear of rain" mark specified in GB/T 191-2008.

9.2 Each batch of lithium carbonate made of brine shall be accompanied by a quality certificate. The content shall at least include manufacturer's name, address, product name, type, net content, batch number (or production date), certification that the product quality conforms to this document and this document number.

10 Packaging, transportation and storage

10.1 Lithium carbonate made of brine shall be double-packed. The inner packaging bag shall be polyethylene or polypropylene film bag. The outer packaging shall be a composite plastic woven bag. The net content of each bag is 25kg. It can also use FIBC packaging. The net content of each bag is 500kg.

10.2 When the brine carbonic acid is packed in the inner bag, the air in the bag shall be exhausted. The mouth of the bag is fastened with nylon rope or sealed by other equivalent methods. The outer packaging shall be firmly seamed with nylon thread or other thread of equivalent quality. Or align and fold the inner and outer packaging bag

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