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Cell liquor of lithium hexafluorophosphate

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Cell liquor of lithium hexafluorophosphate

Warning: The samples and some of the reagents used in this test method are toxic or corrosive, and shall be handled carefully in a fume hood! If it splashes on the eyes or skin, rinse it with water for more than 15 minutes and treat immediately. Where flammable products are used, it is strictly forbidden to use an open flame for heating.

1 Scope

This Standard specifies the classification, requirements, test methods, inspection rules, marking, labeling, packaging, transportation and storage of cell liquor of lithium hexafluorophosphate.

This Standard applies to cell liquors containing lithium hexafluorophosphate in different solvent systems. This product is used in the production of lithium-ion batteries and lithium-ion ultra-capacitors.

2 Normative references

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

GB 150.1, Pressure vessels - Part 1: General requirements

GB 150.2, Pressure vessels - Part 2: Materials

GB 150.3, Pressure Vessels - Part 3: Design

GB 150.4, Pressure vessels - Part 4: Fabrication, inspection and testing, and acceptance

GB 190-2009, Packing symbol of dangerous goods

GB/T 191-2008, Packaging - Pictorial marking for handling of goods

GB/T 605-2006, Chemical reagent - General method for the measurement of colour

GB/T 6678, General principles for sampling chemical products

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

Use a pipette to transfer 1 mL of sulfate standard stock solution prepared according to HG/T 3696.2; put it in a 100 mL volumetric flask; use water to dilute to the mark; shake well. Formulate the solution when needed.

5.3.3 Analysis steps

5.3.3.1 Preparation of standard comparison solution: Into a series of 50 mL colorimetric tubes, transfer 0.50 mL, 1.00 mL, 1.50 mL, 2.00 mL, 2.50 mL and 3.00 mL of sulfate standard solution respectively; add 0.3 mL of hydrochloric acid solution and 3 mL of absolute ethanol in turn; use water to dilute to 25 mL; shake well; then, respectively transfer into 2 mL of barium chloride solution; shake well; let stand for 1 h. Compare with the sample solution.

5.3.3.2 Preparation of sample solution: In a fume hood, weigh about 3 g of the sample, accurate to 0.01 g. Then, operate in accordance with 5.3.3.1 from "add 0.3 mL ... in turn" to "...shake well; let stand for 1 h". Perform visual turbidimetry with the standard comparison solution prepared at the same time.

5.3.3.3 Judgment: The turbidity of the sample solution shall not be greater than the standard comparison solution containing 1.50 mL of sulfate standard solution.

5.4 Determination of chloride content

5.4.1 Method summary

After the sample is dissolved, add silver nitrate to the nitric acid medium to form a white silver chloride suspension with chloride ions, and compare the turbidity with the standard.

5.4.2 Reagents

5.4.2.1 Nitric acid solution: 1+2.

5.4.2.2 Silver nitrate solution: 17 g/L.

5.4.2.3 Chloride standard solution: 1 mL of solution contains 10 μg of chloride (Cl).

Use a pipette to pipette 10 mL of chloride standard stock solution prepared according to HG/T 3696.2; put it in a 100 mL volumetric flask; use water to dilute to the mark; shake well. Use a pipette to pipette 10 mL; place in a 100 mL volumetric flask; use water to dilute to the mark; shake well. Formulate the solution when needed.

5.4.3 Analysis steps

5.4.3.1 Preparation of standard turbidimetric solution: Into a series of 50 mL colorimetric tubes, transfer 0.05 mL, 0.10 mL, 0.20 mL, 0.30 mL, 0.40 mL and 0.50 mL of chloride standard solution, respectively. Use water to dilute to about 15 mL; add

5 mL of nitric acid solution and 1 mL of silver nitrate solution; use water to dilute to 25 mL; shake well; let stand for 15 min. Compare with the sample solution.

5.4.3.2 Preparation of sample solution: Weigh about 5 g of the sample in the glove box, accurate to 0.01 g. Take it out; put it into a 50 mL colorimetric tube filled with 15 mL of water; add 5 mL of nitric acid solution and 1 mL of silver nitrate solution; use water to dilute to 25 mL; shake well; let stand for 15 min. Determine by visual turbidimetry; compare the turbidity presented with the standard turbidimetric solution.

5.4.3.3 Judgment: The turbidity of the sample solution shall not be greater than the standard turbidimetric solution containing 0.50 mL of chloride standard solution.

5.5 Determination of cation content

5.5.1 Method summary

Measure each cation content in the sample by the working curve method on an inductively coupled plasma optical emission spectrometer (ICP-OES) with a fluoride-resistant sampling system.

5.5.2 Reagents

5.5.2.1 Ethyl methyl carbonate-absolute ethanol solution: Dissolve the electronic grade ethyl methyl carbonate, absolute ethanol and water evenly in each other according to the volume ratio of 1:4:5.

5.5.2.2 Mixed standard solution: 1 mL of solution contains 0.01 mg of cations Al, As, Cd, Cr, Cu, Hg, K, Mg, Ni, Pb and Zn each, and 0.10 mg of cations Ca, Fe and Na each.

Pipette 1 mL of standard stock solutions of cations Al, As, Cd, Cr, Cu, Hg, K, Mg, Ni, Pb, and Zn prepared according to HG/T 3696.2, and 10 mL of standard stock solutions of cations Ca, Fe, and Na; place in the same 100 mL volumetric flask; use ethyl methyl carbonate-absolute ethanol solution to dilute to the mark; shake well. Alternatively, use the standard stock solution (1 000 μg/mL) of the national standard sample to accurately dilute. Formulate this solution when needed.

5.5.3 Instruments and apparatuses

5.5.3.1 Inductively coupled plasma-optical emission spectrometer (ICP-OES): equipped with a fluoride-resistant sampling system.

5.5.3.2 Volumetric flask: polyethylene or other corrosion-resistant materials, with a volume of 100 mL.

Before each use, use nitric acid solution (10%) to soak and wash.

5.5.3.3 Pipette and tip: 100 μL ~ 1 000 μL.

5.6.1 Colorimeter method (arbitration method)

5.6.1.1 Equipment

Automatic colorimeter: measuring wavelength range 420 nm ~ 720 nm, step 10 nm.

5.6.1.2 Analysis steps

Import the sample into a cuvette with an optical path of 50 mm; pour it into about 2/3 of the volume of the cuvette; use lens tissue to wipe the outside of the cuvette; put the cuvette into the colorimeter; close the cover and perform measurement; read the measured platinum-cobalt data.

5.6.2 Platinum-cobalt standard solution colorimetric method

5.6.2.1 Method summary

Prepare potassium chloroplatinate, cobalt chloride and hydrochloric acid in a certain proportion into an aqueous solution (platinum-cobalt standard solution), and the hue of the obtained solution is similar to that of the sample to be tested in most cases. Obtain the chromaticity of the sample by visually comparing the sample with the platinum-cobalt standard solution.

5.6.2.2 Reagents and solutions

Platinum-cobalt standard solution: 50 Hazen.

Pipette 10 mL of 500 Hazen platinum-cobalt standard solution which is prepared according to GB/T 605-2006; put it in a 100 mL volumetric flask; use water to dilute to the mark; shake well. Formulate the solution when needed.

5.6.2.3 Instruments and apparatuses

5.6.2.3.1 Colorimetric tube: 50 mL.

The glass color and the scaled height the selected colorimetric tubes shall be basically the same.

5.6.2.3.2 Standard light box.

5.6.2.4 Analysis steps

Inject the sample into the colorimetric tube; compare it with the platinum-cobalt standard solution of 50 Hazen visually along the axis of the colorimetric tube in the standard light box. The color of the sample shall not be darker than the platinum-cobalt standard solution.

5.7 Determination of density

5.7.1 Density instrument method (arbitration method)

5.7.1.1 Method summary

Use a density instrument to measure the density of the cell liquor at 20°C.

5.7.1.2 Instrument

Density instrument: measuring range 0 g/mL ~ 3 g/mL; precision 0.001 g/mL.

5.7.1.3 Analysis steps

Set the measurement temperature of the instrument to 20°C; inject the sample into the measurement cell of the instrument; perform the measurement and read the data.

5.7.2 Density meter method

5.7.2.1 Method summary

Use a density meter to measure the density of the cell liquor at 20°C.

5.7.2.2 Instrument

5.7.2.2.1 Density meter.

5.7.2.2.2 Constant temperature water bath: the temperature can be controlled at 20°C ± 0.5°C.

5.7.2.3 Analysis steps

Put about 100 mL of the sample into a dry and clean stoppered measuring cylinder; seal it tightly; place it in a constant temperature water bath at 20°C ± 0.5°C; after 30 minutes of constant temperature, open the measuring cylinder, and slowly insert a clean and dry density meter; make it sink naturally, not contacting with the wall or bottom of the cylinder; keep the density meter suspended and still, without bubbles; read the scale at the point where the density meter is tangent to the meniscus, which is the density (ρ) of the tested sample at 20°C.

5.8 Determination of electrical conductivity

5.8.1 Method summary

The cell liquor of lithium hexafluorophosphate is of a certain conductivity, which is related to the ratio of solvent and solute. Under constant temperature conditions, use a conductivity meter to measure the conductivity of the electrolyte.

5.8.2 Equipment

5.8.2.1 Conductivity meter.

(d_{max} <2 cm) and 30 mL of ice water (temperature ≤4°C)]; add 10 drops of bromothymol blue indicator solution; use sodium hydroxide standard titration solution for rapid titration until the solution turns blue as the end point; there shall still be ice cubes in the solution at the end point. The analysis process shall be controlled within 20 s.

At the same time, do a blank test. Except that no sample is added to the blank test solution, the types and volume of other reagents added are the same as those of the test solution.

5.10.5 Result calculation

According to the provisions of 3.9.5 in GB/T 19282-2014.

6 Inspection rules

6.1 This Standard adopts type inspection and exit-factory inspection. Type inspection and exit-factory inspection shall meet the following stipulations:

a) All index items specified in the requirements are type inspection items, which, under normal circumstances, shall be carried out every month.

   In any of the following cases, type inspection shall be carried out:

   -- when key equipment and production processes are updated;

   -- when there are changes in the main raw materials;

   -- when the suspended production is resumed;

   -- when there is a significant difference from the previous type inspection;

   -- when it is stipulated in the contract.

b) Chromaticity, density, conductivity, moisture and free acid specified in the requirements are exit-factory inspection items, which shall be inspected batch by batch.

6.2 The cell liquor of lithium hexafluorophosphate, which is produced continuously – with the same materials and basically under the same production conditions – by the production enterprise, shall be the same batch. Each batch of products shall not exceed 50 t.

6.3 The number of sampling units shall be determined in accordance with the provisions of GB/T 6678. Use clean and dry corrosion-resistant bottles for airtight sampling. The sampling volume of each batch of products shall not be less than 200 g. Seal immediately after sampling. Mark the product name, batch number, sampling date and the name of the sampler on the bottle. Use one bottle for inspection; seal the other bottle

with aluminum foil and place it in a cool, dry place. The storage time is determined by the manufacturer according to actual needs.

6.4 If any of the inspection results does not meet the requirements of this Standard, re-inspection shall be taken from twice the number of products. Even if only one index of the re-inspection results does not meet the requirements of this Standard, judge the entire batch of products as unqualified.

6.5 Use the comparison method of rounding off for numerical values specified in GB/T 8170 to judge whether the inspection results meet this Standard.

7 Marking, labeling

7.1 There shall be firm and clear markings on the outer packaging of the cell liquor product of lithium hexafluorophosphate, including: manufacturer name, factory address, product name, category, net content, batch number or production date, shelf life, number of this Standard, the marking of "toxicity" specified in GB/T 190-2009, and the markings of "afraid of rain" and "afraid of sun" stipulated in GB/T 191-2008.

7.2 Each batch of cell liquor products of lithium hexafluorophosphate leaving the factory shall be accompanied by a quality certificate, including: manufacturer name, factory address, product name, category, net content, batch number or production date, shelf life, evidence of product quality conforming to this Standard and number of this Standard.

8 Packaging, transportation and storage

8.1 Cell liquor of lithium hexafluorophosphate shall be packaged under airtight and dry conditions. The products shall be packaged in stainless-steel barrels that meet the requirements of GB 150.1~150.4. The stainless-steel barrels shall have a pressure resistance greater than 0.6 MPa. The stainless-steel barrels shall be filled with nitrogen or argon, and the pressure at 25°C shall be ≥20 kPa and ≤80 kPa. The quick-connect joints of stainless-steel barrels shall have no corrosion marks and be covered with protective covers. The net weight of each barrel is 20 kg or 200 kg, or packed according to the weight required by the user.

8.2 During transportation, there shall be coverings to avoid the sun and rain, so as to prevent the packaging from being damaged. For special products, select the appropriate transportation conditions according to the instructions attached to the product.

8.3 Keep the original packing barrel intact and airtight; place it in a cool, dry and well-ventilated warehouse; prohibit direct sunlight. When the temperature in the warehouse is higher than 30°C, cooling measures shall be taken. For special products, select the appropriate storage conditions according to the instructions attached to the product.

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