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CHEMICAL INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HG/T 4067-2015

Replacing HG/T 4067-2008

Lithium hexafluorophosphate electrolyte solution

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(English Translation)

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Foreword

This Standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This Standard replaces HG/T 4067-2008 Lithium hexafluorophosphate and cell liquor of lithium Hexafluorophosphate - Part 2: Cell liquor of lithium hexafluorophosphate; compared with HG/T 4067-2008, the main technical changes, except editorial changes, are as follows.

- Modify the scope of application of cell liquor of lithium hexafluorophosphate (see 1; 1 of the 2008 edition);
- Add the classification of cell liquor of lithium hexafluorophosphate (see 3);
- Adjust the index requirements for sulfate content, iron content and sodium content (see 4.2; 3.2 of the 2008 edition);
- Add the index requirements for aluminum (Al), calcium (Ca), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), magnesium (Mg), nickel (Ni), lead (Pb), zinc (Zn) and arsenic (As) (see 4.2);
- Modify the determination method of chloride to turbidimetric method (see 5.4; 4.9 of the 2008 edition);
- Add the colorimeter method (arbitration method) for the measurement of chromaticity (see 5.6; 4.3 of the 2008 edition);
- Add the density instrument method (arbitration method) for the measurement of density (see 5.7; 4.4 of the 2008 edition).

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

GB/T 8170, Rules of rounding off for numerical values & expression and judgment of limiting values

GB/T 19282-2014, Analytic method for lithium hexafluorophosphate

3 Classification

Cell liquor of lithium hexafluorophosphate is divided into two categories according to its different uses.

4 Requirements

4.2

Cell liquor of lithium hexafluorophosphate, when tested according to the test method

specified in this Standard, shall meet the technical requirements in Table 1.

5.1 General provisions

The reagents and water used in this Standard refer to analytical reagents and grade-3 water specified in GB/T 6682-2008, unless otherwise specified. The standard titration solutions, impurity standard solutions, preparations and products that are required in the test shall be prepared in accordance with the provisions of HG/T 3696.1, HG/T 3696.2, HG/T 3696.3 when no other requirements are specified.

5.2 Appearance inspection

In a fume hood, judge the appearance visually.

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.

5.4.1 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.

5.4.3 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.

5.5.1 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.

5.5.4.1 Preparation of sample solution

Weight 5 g of the sample in the glove box to the nearest 0.002 g. Take it out from the glove box; put it in a 50 mL (V) volumetric flask; add ethyl methyl carbonate-absolute ethanol solution to dilute to the mark; shake well.

5.5.5 Result calculation

Calculate the content of cations in mass fraction w_1 ; express the value in mg/kg; calculate according to Formula (1).

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.

5.6.2.4 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.

5.7.1.1 Method summary

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