

SPECIFICATION



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TITLE OF THE INVENTION:

Method for Producing Lithium Hexafluorophosphate

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TECHNICAL FIELD

The present invention relates to a method for producing lithium hexafluorophosphate, and specifically relates to a method for producing lithium hexafluorophosphate useful as an electrolyte for lithium secondary batteries and a catalyst for organic synthesis.

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BACKGROUND OF THE INVENTION

Conventionally, lithium hexafluorophosphate is produced by the following steps:

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1) Lithium fluoride (LiF) is dissolved in hydrofluoric acid anhydride to give a solution.

2) PF_5 gas is blown into this solution. This operation causes LiF to react with PF_5 to produce lithium hexafluorophosphate.

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3) The solution is cooled to deposit lithium hexafluorophosphate.

4) The solution is filtered to separate the deposited lithium hexafluorophosphate.

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5) The separated lithium hexafluorophosphate is then dried to yield lithium hexafluorophosphate as a product.

6) Following this step, a product of lithium hexafluorophosphate is dissolved in an organic solvent, for example, for use as an electrolyte for a lithium battery.

However, according to the related method for producing

lithium hexafluorophosphate, many kinds of impurities such as lithium fluoride and oxyfluoride acid compound are contained in hexafluorophosphate.

5 When lithium hexafluorophosphate containing such impurities is dissolved in an organic solvent for use as an electrolyte for a lithium battery, lithium fluoride contaminate is not soluble in the organic solvent and the electrolyte therefore has to be filtered.

10 Examples of methods for removing these impurities are a method where lithium hexafluorophosphate is dissolved in organic solvent to remove impurities using an ion exchange resin (Japanese Published Unexamined Patent Application No. 1984-87774) and a method where neutralization treatment is carried out in an organic solvent (1984-81870.) However, these methods
15 are complex in operation and provide low productivity.

As described above, related methods for producing lithium hexafluorophosphate require complex after-treatment. Even if after-treatment is performed, lithium hexafluorophosphate of high purity cannot be produced, which is problematic.

20 The present invention provides a method for producing lithium hexafluorophosphate capable of yielding lithium hexafluorophosphate of high purity without requiring after-treatment.

25 DISCLOSURE OF THE INVENTION

The method for producing lithium hexafluorophosphate according to the present invention is characterized by filtering lithium hexafluorophosphate coexisting with the solvent and then drying after filtering in a gas atmosphere containing PF_5 .

Organic solvents that may be used as the solvent are solvents that do not react with hydrofluoric acid anhydride or lithium hexafluorophosphate.

5 A state where lithium hexafluorophosphate coexists with the solvent is a state in which the following steps are completed.

1) Lithium fluoride (LiF) is dissolved in hydrofluoric acid anhydride to give a solution.

2) PF_5 gas is blown into this solution. This operation causes LiF and PF_5 to react to produce lithium hexafluorophosphate.

10 3) The solution is cooled to deposit lithium hexafluorophosphate.

In the present invention, the step of filtering the solution is performed in a gas atmosphere containing PF_5 .

15 Gases that may be employed as the gas containing PF_5 (atmosphere gas, hereafter) are gases that are prepared by diluting PF_5 with a gas (for example, HF gas, HCl gas, nitrogen gas, argon gas, helium gas, xenon gas, dried air, a gas that does not react with PF_5 or lithium hexafluorophosphate.)

20 The concentration of PF_5 in the atmosphere gas is preferably 1 to 50 mol%, with 1 to 20 mol% being more preferable, and 1 to 10 mol% being still more preferable. A concentration less than 1 mol% may result in the effects being insufficient. A concentration of over 50 mol% causes an increase in PF_5 consumption and is therefore expensive.

25 On the other hand, 0.003 to 0.3 kg of PF_5 is preferable for 1 kg of lithium hexafluorophosphate, with 0.003 to 0.03 kg of PF_5 being more preferable. less than 0.003 kg of PF_5 may not cause effects that are sufficient. An amount over 0.3 kg causes the increase in PF_5 consumption which is expensive.

Meanwhile, the concentration of impurities, particularly moisture, contained in the atmosphere gas is preferably 100 ppb or smaller, more preferably 10 ppb or smaller, and still more preferably 1 ppb or smaller. Such control brings about a yield
5 of lithium hexafluorophosphate of high purity.

The pressure of the atmosphere gas may be normal pressure. The temperature may also be normal.

Fig. 1 shows a filtering apparatus used in the present invention.

10 Drying of lithium hexafluorophosphate separated as described above is carried out in the atmosphere gas as described above.

A transition time between filtering and drying is preferably short during which no exposure to an outside (air)
15 atmosphere is preferable.

That shown in Fig. 2 is preferably used as a drying apparatus,.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a conceptual diagram of the filtering apparatus
20 used in the present invention.

Fig. 2 is a conceptual diagram of the drying apparatus used in the present invention.

BEST MODE FOR CARRYING OUT THE INVENTION

25 An Embodiment will be described below with reference to a comparative example.

(Embodiment 1)

Hydrofluoric acid anhydride solution containing 22% lithium hexafluorophosphate is cooled at -20°C to deposit

crystals of lithium hexafluorophosphate.

Next, this slurry is transferred to a filtering apparatus (20 L (liter) volume) with inlet and outlet orifices for the atmosphere gas to be filtered for 10 minutes in a flow of the atmosphere gas consisting of 3 mol% of PF_5 and 97 mol% of nitrogen at a flow rate of 15 L/ min, dried in the same atmosphere gas at a room temperature for 25 min, and finally heated at 105 °C overnight.

The amount of the lithium hexafluorophosphate crystal yielded was 11.3 kg and impurities were 50 ppm in HF and 70 ppm in LiF.

The amount of PF_5 used was 0.008 kg a 1 kg of lithium hexafluorophosphate.

15 (Comparative example 1)

Hydrofluoric acid anhydride solution containing 22% lithium hexafluorophosphate was cooled at -20°C to deposit a crystal of lithium hexafluorophosphate.

Next, this slurry was transferred to the filtering apparatus (20 L (liter) volume) with inlet and outlet orifices for the atmosphere gas to be filtered for 10 minutes flowing the atmosphere gas exclusively consisting of nitrogen at a flow rate of 15 L/ min, dried in the atmosphere gas at a room temperature for 25 min, and finally heated at 105 °C overnight.

25 The amount of the lithium hexafluorophosphate crystal yielded was 11.1 kg and impurities were 58 ppm in HF and 930 ppm in LiF.

The slurry is decomposed during filtering and drying resulting in an increase in LiF as an impurity.

{Embodiment 2}

Hydrofluoric acid anhydride solution containing 22% of lithium hexafluorophosphate is cooled at -20°C to deposit a crystal of lithium hexafluorophosphate.

5 Next, this slurry is transferred to filtering apparatus (100 L (liter) volume) with inlet and outlet orifices for the atmosphere gas to be filtered for 60 minutes flowing the atmosphere gas consisting of 15 mol% of PF_5 and 85 mol% of nitrogen at a flow rate of 12 L/ min, dried in the same atmosphere gas
10 at the room temperature for 30 min, and further dried for 90 min in the flow of the atmosphere gas consisting of 5 mol% of PF_5 and 95 mol% of nitrogen at a 35 L/ min flow rate, and finally heated at 105°C overnight.

15 The amount of the lithium hexafluorophosphate crystal yielded was 57 kg and impurities was 52 ppm in HF and 90 ppm in LiF.

The amount of PF_5 used is 0.022 kg per 1 kg of lithium hexafluorophosphate.

(Comparative example 2)

20 Hydrofluoric acid anhydride solution containing 22% lithium hexafluorophosphate is cooled at -20°C to deposit a crystal of lithium hexafluorophosphate.

25 Next, this slurry is transferred to filtering apparatus (100 L (liter) volume) with inlet and outlet orifices for the atmosphere gas to be filtered for 60 minutes in the flow of the atmosphere gas exclusively consisting of nitrogen at a flow rate of 12 L/ min, dried in the atmosphere gas at a room temperature for 30 min, and finally heated at 105°C overnight.

The amount of the lithium hexafluorophosphate crystal

yielded was 54 kg and impurities were 54 ppm in HF and 1190 ppm in LiF.

The slurry was decomposed during filtering and drying resulting in an increase in LiF as an impurity.

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INDUSTRIAL APPLICABILITY

According to the present invention without after-treatment, lithium hexafluorophosphate of a higher purity than that of conventional can be yielded.

WHAT IS CLAIMED IS:

1. A method for producing lithium hexafluorophosphate characterized in that the method is carried out by filtering lithium hexafluorophosphate coexisting with a solvent and
5 carrying out post-filtering drying in a gas atmosphere containing PF_5 .
2. The method for producing lithium hexafluorophosphate according to claim 1, wherein concentration of PF_5 in the gas
10 ranges from 1 to 50 %.
3. The method for producing lithium hexafluorophosphate according to claim 1 or 2, wherein PF_5 ranges from 0.003 to 0.3 kg for 1 kg of lithium hexafluorophosphate.