CHE261A Patent Application

Nature of Invention: Chemical molecule and synthesis route

Applicant: Ultraviolet Chemicals

Inventors: 1. Akshat Tiwari

2. Pratibha Bajaj

3. Rutvik Nawade

4. Diptansu Poddar

Chemical Formula: $LiPF_6$

Chemical Name: Lithium Hexafluorophosphate

Chemical synthesis routes:

a. LiPF6 is predominantly produced from the reaction of phosphorus pentafluoride (PF5) and lithium fluoride (LiF) according to the following reaction in the presence of a solvent.

$$LiF + PF_5 \rightleftharpoons LiPF_6 \qquad (1)$$

Raw materials:

- The key raw materials are HF (l), LiCl (s), and PCl5 (s), where l and s represent the physical state of materials, i.e., liquid and solid, respectively.

Production process involves HF as the solvent. The purchased HF typically contains around 100−150 ppm of moisture. This moisture is reduced by bubbling fluorine gas (F2) through it at 10 °C and 1 bar.

$$H_2O \text{ in } HF(l) + 2F_2(g) = 2HF(l) + OF_2(g) \qquad (2)$$

OF2 has a very low boiling point (ca. −145 °C) and is easily volatilized and removed from the liquid HF. The dried HF obtained after the moisture removal step is then distributed across the process as anhydrous HF (AHF). AHF is added to Reactors 1, 2, and 3 to produce PF5, LiF, and eventually LiPF6, respectively. In Reactor 1, a slurry is made by

adding liquid AHF to the PCl5 powder. The reactor is maintained at −30 °C, 27 bar, with a residence time of 1 h. PCl5 readily reacts with HF in these conditions as follows.

$$PCl_5(s) + 5HF(l) = PF_5(g) + 5HCl(g) \quad \Delta H = -313\ \text{kJ mol}^{-1} \tag{3}$$

Complete conversion of the PCl5 is ensured by using 5% excess AHF. As the reaction 3 is exothermic and with the feeds at −10 and 25 °C, 924 kW of heat is removed from the reactor with a liquid cooled refrigeration system to maintain the desired temperature of −30 °C. The gaseous products, PF5 and HCl with molar ratio of 1:5, exit the top. It is decompressed to 10 bar and then heated to 0 °C with process water in a heat exchanger, before entering Reactor 3. The excess liquid AHF at the bottom is sent to the HF recycling unit. In Reactor 2, a slurry is formed by mixing excess liquid AHF with LiCl at 0 °C and 1 bar, with a residence time of 1 h, to facilitate the following reaction.

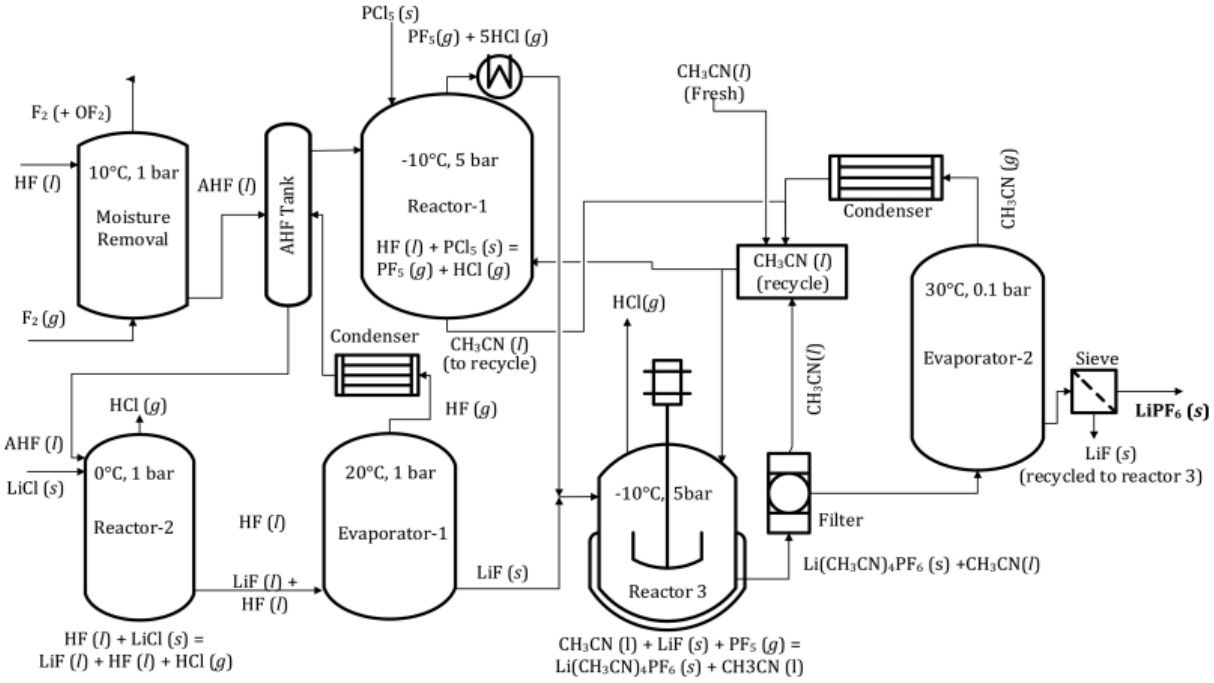
$$LiCl(s) + HF(l) \overset{\text{in liq AHF}}{\Longleftrightarrow} LiF(\text{in liq AHF}) + HCl(g)$$
$$\Delta H = -1.19\ \text{kJ mol}^{-1} \tag{4}$$

The excess AHF is to ensure that the product LiF remains soluble. The LiF in AHF solution is removed from the bottom of the reactor. The gaseous HCl product is collected at the top of the reactor and is packaged to be sold as a byproduct. Gaseous mixture PF5 and HCl from Reactor 1 is bubbled through the solution of LiF in AHF in Reactor 3 at 0 °C and 10 bar to produce LiPF6.

$$LiF(\text{in liq AHF}) + PF_5(g) \overset{\text{in liq AHF}}{\Longleftrightarrow} LiPF_6(s) \quad \Delta H = -85\ \text{kJ mol}^{-1} \tag{5}$$

Reaction 5 is carried out in Reactor 3 in the presence of liq. AHF (~96 wt %) as a solvent to produce LiPF6 crystals. The reaction reaches completion in about 5 h. Reactor 3 system is modelled as a series of CSTRs. The presence of HCl(g) in reaction 5 is not shown as it acts as inert, which is collected at the top of the reactor, packaged, and sold as a by-product. Solvent AHF is removed by vaporization in Evaporator 1, then condensed, and recycled. The dried product in Evaporator 1 consisting of LiPF6 and 7% by weight of HF is sent to the crystallizer. Additional HF is added to the crystallizer such that HF content in the solution is around 75% by weight. The solution is cooled to −10 °C, where LiPF6 crystals grow to form larger crystals of higher purity (~99.9%). HF separated in Evaporator 2 is then condensed and recycled. The product of Evaporator 2 is a solid mixture consisting of both 99.9%

pure LiPF6 crystals and LiF powder, which is separated through a vibrating sieve. LiF obtained is recycled. The recovery of LiPF6 from Evaporator 2 is around 85% with a purity of 99.9%, where the impurities consist of both HF and LiF.



References

- https://www.alfa.com/en/catalog/011529/#:~:text=Used%20as%20an%20electrolyte%20in,spectrometer%20and%20x%2Dray%20monochromator.
- https://pubs.acs.org/doi/pdf/10.1021/acs.iecr.8b03752#:~:text=LiPF6%20is%20predominantly%20produced%20from,the%20presence%20of%20a%20solvent.

List the contributions of each author:

- Author 1 and 2 worked on selection of the chemical.
- Author 2,3,4 worked on the selection of the manufacturing process.
- Author 1,2,3 worked on studying the process in detail, doing its feasibility analysis and other factors.

Name	Roll No	Signature
Mohd Hamza	210619	
Diptansu Poddar	210347	
Rutvik Nawade	210659	
Pratibha Bajaj	210757	
Akshat Tiwari	210095	