

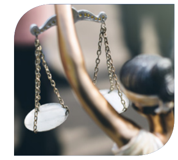

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Hydrofluoric Acid Titration: Quality Control Parameter in Battery Electrolytes

Hydrofluoric acid (HF) titration provides insights into electrolyte decomposition and impurity levels.

Hydrofluoric acid (HF) is a critical quality control parameter found in battery electrolytes that will significantly impact cell stability, performance, and safety. HF titration provides insights into electrolyte decomposition and impurity levels. This application note highlights the importance of HF measurements in maintaining electrolyte quality.

Introduction

A common electrolyte salt in lithium-ion batteries, lithium hexafluorophosphate ($LiPF_6$) dissolved in carbonate solvents, is highly sensitive to moisture and impurities. After $LiPF_6$ decomposes, trace water hydrolyzes PF_5 to form HF:[1]

$$LiPF_6 \rightarrow LiF + PF_5$$

$$PF_5 + H_2O \rightarrow POF_3 + 2HF$$

High levels of HF are detrimental as it can corrode battery components, degrade the solid electrolyte interphase (SEI) layer, and ultimately compromise overall battery lifespan. Quantifying HF provides insight into the extent of $LiPF_6$ degradation therefore routine HF monitoring is essential for quality assurance and safe battery manufacturing.

Experimental

A 1.0M lithium hexafluorophosphate ($LiPF_6$)-based electrolyte formulated with ethylene carbonate (EC), dimethyl carbonate (DMC), and diethyl carbonate (DEC) in a 1:1:1 ratio was evaluated. The manufacturer's certificate of analysis reported 17ppm HF from its release date listed as March 9, 2022.[2] Quantification of hydrofluoric acid (HF) was measured in triplicate through an acid-base titration using a sample weight of 3g per sample run with the expectation of degradation over time.

Results and Discussion

Hydrofluoric acid (HF) was quantified through the titration curve (pH vs. mL) and the identified equivalence point observed in figure 1.

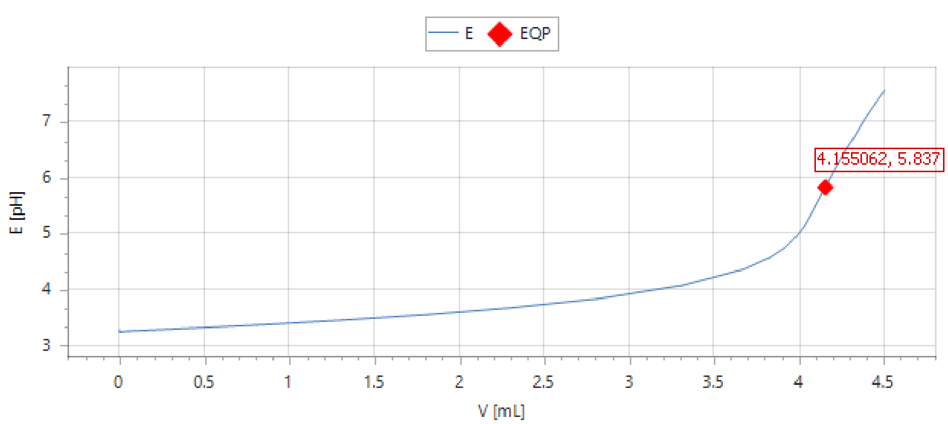

Figure 1: Acid-base titration curve (pH vs. mL) with identified equivalence point to quantify hydrofluoric acid

The sample size and HF content quantified from the $LiPF_6$ in EC/DMC/DEC (1:1:1) is reported in table 1. The average determined HF content was 263 ppm identified in table 2 along with additional statistics such as relative standard deviation that confirms the accuracy and repeatability of the data collected. Maintaining low HF levels is critical and confirms that the electrolyte has degraded over time and can no longer be used in battery applications.

Table 1: Hydrofluoric acid (HF) content (ppm) and sample size of $LiPF_6$ EC/DEC/DMC 1:1:1 measured in triplicate

Sample ID	Sample Size	HF Content
1	3.2222 g	264 ppm
2	3.1968 g	262 ppm
3	3.1058 g	264 ppm

Table 2: Statistics of hydrofluoric acid (HF) content of $LiPF_6$ EC/DEC/DMC 1:1:1 measured in triplicate

Mean	Minimum	Maximum	Standard Deviation	Relative Standard Deviation
263 ppm	262 ppm	264 ppm	1 ppm	0.438 %

Conclusion

This study demonstrates the effectiveness of acid-base titration for quantifying hydrofluoric acid (HF) in battery electrolytes. Maintaining low water levels is essential in minimizing HF formation to avoid degrading battery components and compromising safety. Conducting quality control and monitoring HF levels provides a reliable framework for stability and performance in battery electrolyte applications.

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References

1. Lux, S. F.; Lucas, I.T.; Pollak, E; Passerini, S.; Winter, M.; Kostecki, R. The mechanism of HF formation in $LiPF_6$ based organic carbonate electrolytes. *Electrochem Commun* 2012, 14 (1), 47-50.

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2. Sigma-Aldrich. *Certificate of Analysis*, Lithium Hexafluorophosphate, Product No. 901685, Batch MKCR2962; Sigma-Aldrich: St Louis, MO, Quality release date: March 2022.