Application Note

Energy and Materials

Improving Battery Production Yield, Performance, and Stability Using FTIR

Degradation of $LiPF_6$ and its effect on battery performance and safety

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Introduction

As the world moves rapidly towards electrification in transportation, electric vehicle (EV) battery manufacturers are under immense pressure to boost productivity and enhance product quality, performance, and safety.

These challenges require strict quality control (QC) of various raw materials before use, processed materials during manufacturing, and the final products. Some materials that are used to make lithium-ion batteries (LIBs) are known for their high reactivity. For instance, lithium hexafluorophosphate ($LiPF_6$), which is the most widely used salt in the electrolytes of commercial rechargeable LIBs, is highly reactive and can decompose into LiF and PF_5. When PF_5 is exposed to moisture, it reacts with water to form POF_3 and hydrogen fluoride (HF), a highly toxic and corrosive gas.[1–4] These properties pose a significant safety hazard and can accelerate battery degradation, potentially resulting in failure.

FTIR enables rapid, non-destructive chemical fingerprinting of a wide variety of materials and substances. To ensure the quality of battery-grade $LiPF_6$ before use, a simple QA/QC workflow can be conducted using a robust FTIR designed for manufacturing environments.

The Agilent Cary 630 FTIR spectrometer equipped with a diamond attenuated total reflection (ATR) (Figure 1) module can ensure the integrity (quality) of $LiPF_6$ based on the hit quality index (HQI) obtained from the material identification workflow.

The HQI value indicates how well the measured spectrum and the library spectrum match. The HQI is often used as pass/fail criteria in material identification and confirmation workflows (Figure 2). Spectral libraries can be easily created, maintained, and managed in the Agilent MicroLab FTIR software.

Figure 1. The Agilent Cary 630 FTIR spectrometer with its ultracompact, lightweight design (20 × 20 cm and 3.6 kg) can easily be used in a glovebox to produce high-quality results for moisture-sensitive or hazardous chemicals.

Experimental

To illustrate the workflow, three $LiPF_6$ samples that were kept and handled under different conditions were measured by FTIR using the parameters listed in Table 1.

Table 1. Agilent Cary 630 FTIR–ATR operating parameters.

Parameter	Setting
Method	Library search
Library Used	User-generated LIB salts library
Search Algorithm	Similarity
Spectral Range	4,000 to 650 cm^{-1}
Background/Sample Scans	32
Spectral Resolution	4 cm^{-1}
Background Collection	Spectral reference library: argon Samples 1 and 2: argon Sample 3: air
Zero Fill Factor	None
Apodization	HappGenzel
Phase Correct	Mertz
Color-Coded Confidence Level Thresholds	Green (high confidence): > 0.95 Orange (medium confidence): 0.90 to 0.95 Red (low confidence): < 0.90

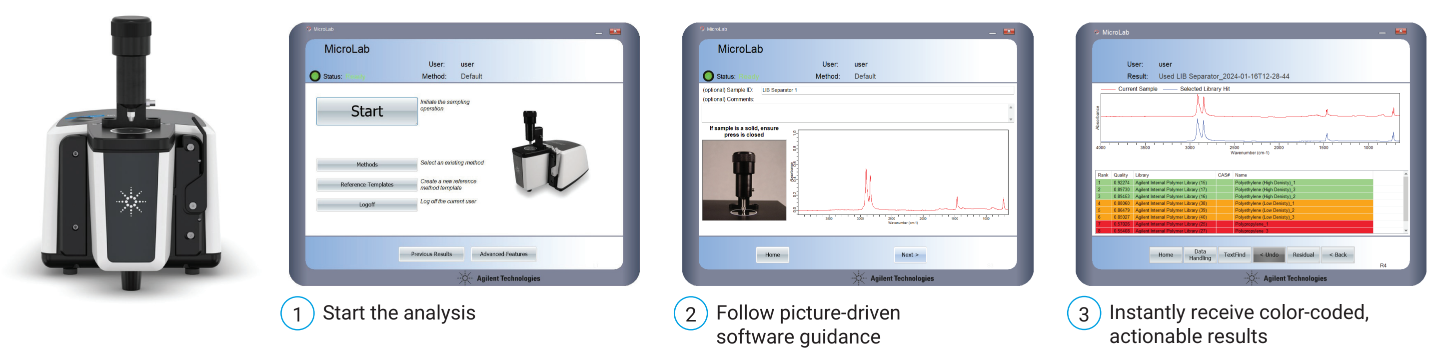

Figure 2. The Agilent MicroLab FTIR software uses a graphical user interface that reduces training needs and minimizes the risk of user-based errors.

Results and discussion

Sample 1 (new bottle) and sample 2 (opened eight months ago) were measured under controlled, moisture-free conditions in a glovebox. Both samples were identified as $LiPF_6$ with HQI values of 0.99392 and 0.91365, respectively.

However, sample 3 (opened eight months ago and measured in air) was also identified as $LiPF_6$, but with an HQI of 0.79151, as shown in Figure 3.The significantly lower HQI for sample 3 suggests potential degradation of the salt in air, as evidenced by changes in the six FTIR spectra acquired at two-minute intervals over 10 minutes (Figure 4).

The HQI values obtained for the three $LiPF_6$ samples aligned closely with their respective storage or usage conditions. The color-coded results emphasized the importance of not storing opened containers of $LiPF_6$ for long periods and handling the salt under dry conditions.

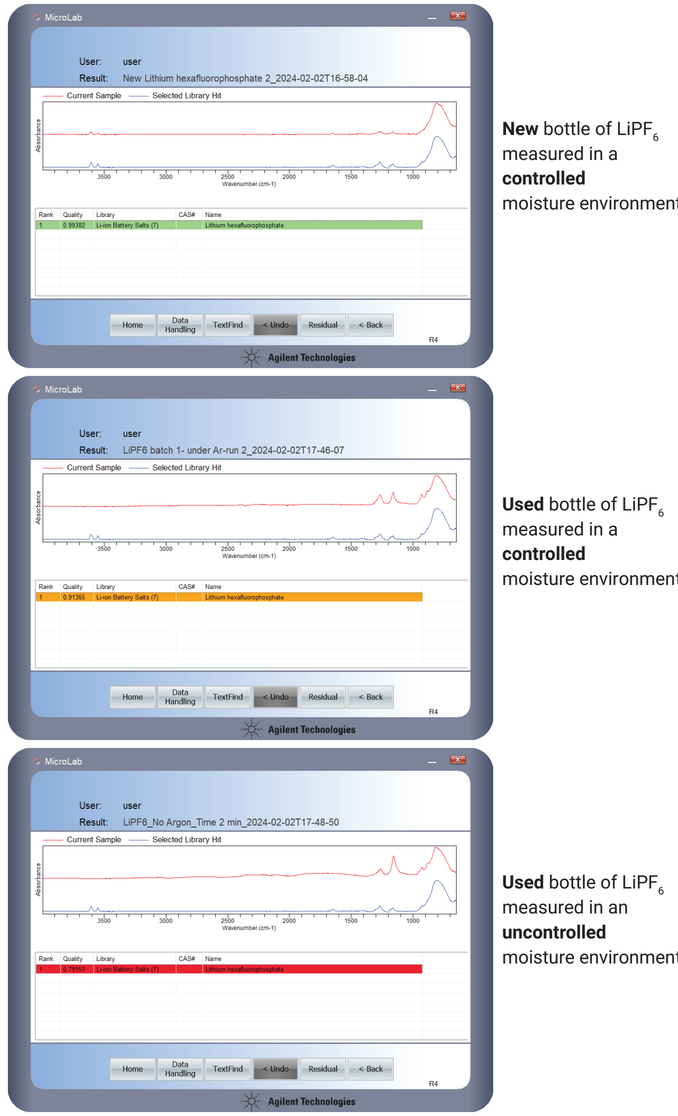

Figure 3. Material identification results for three samples of $LiPF_6$ analyzed under different laboratory conditions. Sample (red traces) and library hit (blue traces).

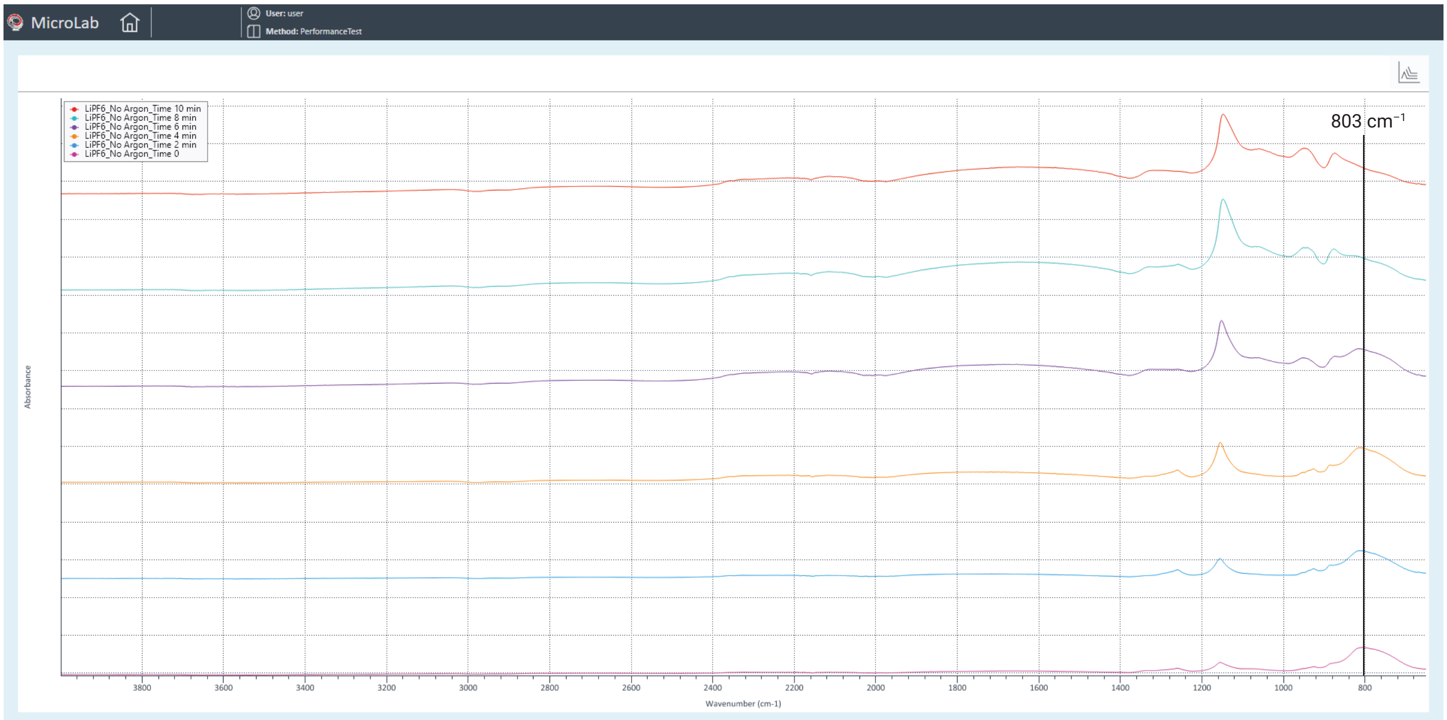

Figure 4. FTIR spectra of $LiPF_6$ sample 3 monitored at two-minute intervals between 0 and 10 minutes using the Agilent Cary 630 FTIR–ATR outside the glovebox. Changes in peaks around 803 cm^{-1} are highlighted across the six spectra.

Conclusion

- The Cary 630 FTIR spectrometer and MicroLab software can assess the degradation status of $LiPF_6$ using a simple and efficient quality testing workflow.
- The color-coded results and pass/fail criteria can be easily tailored for fast, informed decision making in the manufacturing environment.
- The Cary 630 FTIR is ideal for bench- and glovebox-based applications because of its size, simplicity, ease-of-use, and robustness under different environmental conditions.
- The system is ideal for the analysis of lithium salts that require handling in a moisture-controlled environment.
- The ability to rapidly assess the quality and identity of complex salts makes the Cary 630 FTIR a valuable tool in both manufacturing QC settings and research and development laboratories.

References

1. Larsson, F.; *et al.* Toxic Fluoride Gas Emissions from Lithium-Ion Battery Fires. *Sci. Rep.* **2017**, *30*, *7(1)*, 10018.
2. Han, J. Y.; Jung, S. Thermal Stability and the Effect of Water on Hydrogen Fluoride Generation in Lithium-Ion Battery Electrolytes Containing $LiPF_6$. *Batteries* **2022**, *8(7)*, 61.
3. Juba, B. W.; *et al.* Lessons Learned—Fluoride Exposure and Response, *Journal of Chemical Health and Safety* **2021**, *28(2)*.
4. Kraft, V.; *et al.* Ion Chromatography Electrospray Ionization Mass Spectrometry Method Development and Investigation of Lithium Hexafluorophosphate-Based Organic Electrolytes and Their Thermal Decomposition Products. *J. Chromatogr. A* **2014**, *8(1354)* 92–100.

Further information

- Agilent Cary 630 FTIR Spectrometer
- MicroLab FTIR Software
- MicroLab Expert
- FTIR Analysis and Applications Guide
- FTIR Spectroscopy Basics – FAQs
- ATR-FTIR Spectroscopy Overview

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