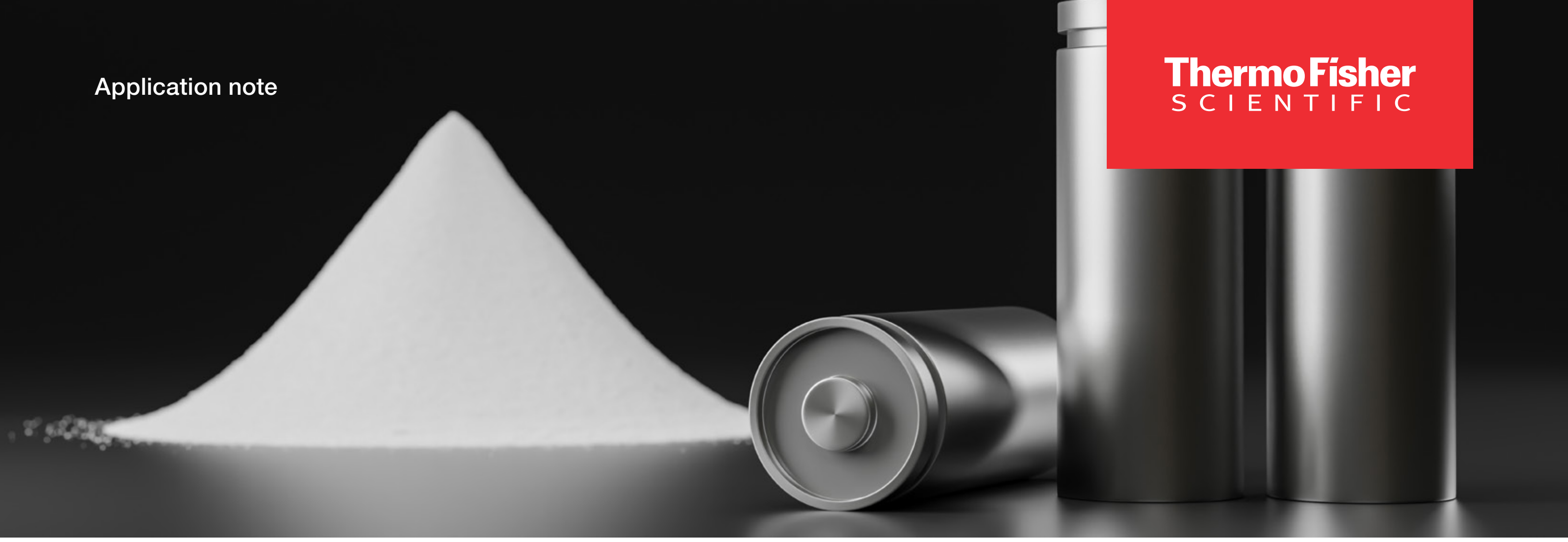
Application note



FTIR characterization of lithium salts in an inert atmosphere

Introduction

Fourier-transform infrared spectroscopy (FTIR) is used extensively to characterize materials in many research fields and industries. The ready availability of small single-reflection attenuated-total-reflectance (ATR) accessories makes it easy to acquire high-quality infrared spectra from small amounts of sample. While this has become routine for many samples, obtaining spectra from materials that are unstable in the presence of moisture or oxygen can be challenging. The most straightforward solution would be to place the FTIR spectrometer in an inert atmosphere glove box. The small size and high sensitivity of the Thermo Scientific™ Nicolet™ iS5 FTIR Spectrometer makes it an ideal system for this configuration. This application note describes an evaluation of the spectrometer when it is placed in a glove box with an inert argon atmosphere.

For many applications, argon is the preferred atmosphere, but its lower thermal conductivity compared to air raises concerns that the instrument might overheat while operating in an argon-filled glove box. For this study, the desiccant assembly was removed from the instrument, leaving an opening that permitted complete purging of the instrument with argon to ensure no moisture would slowly diffuse from the FTIR.

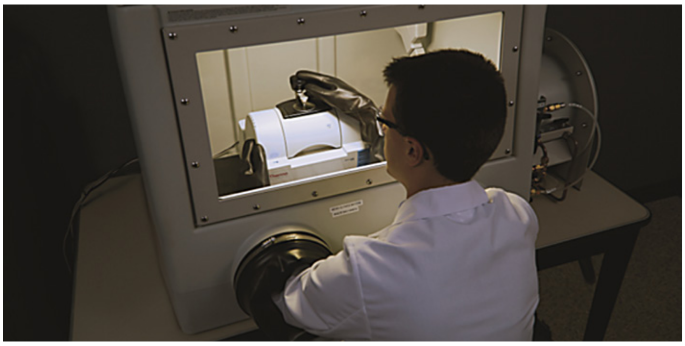


Figure 1. Mounting a sample on the ATR accessory of the Nicolet iS5 FTIR Spectrometer in a glove box.

Sample purity

Lithium hexafluorophosphate ($LiPF_6$) is a highly reactive salt frequently used as an electrolyte in lithium-ion batteries. It is extremely sensitive to moisture and needs to be handled and stored under inert conditions. In this experiment, several previously opened bottles containing lithium salts were placed and analyzed in the glove box with the instrument, including a bottle of $LiPF_6$ that was several years old. The initial spectrum revealed expected peaks near 800 cm^{-1} and 560 cm^{-1} as reported in the literature.* However, when the pressure on the ATR was released, the residual material on the diamond crystal showed a reduction and shift in the fluorophosphate peak near 800 cm^{-1}. Spectral subtraction eliminated the peaks attributed to hydration, leaving a relatively pure spectrum of $LiPF_6$. This suggests that the material in the bottles had degraded.

* Kock, LD, *et al.* Solid state vibrational spectroscopy of anhydrous lithium hexafluorophosphate ($LiPF_6$) *J. Mol Structure* **1026**, 145 (2012)

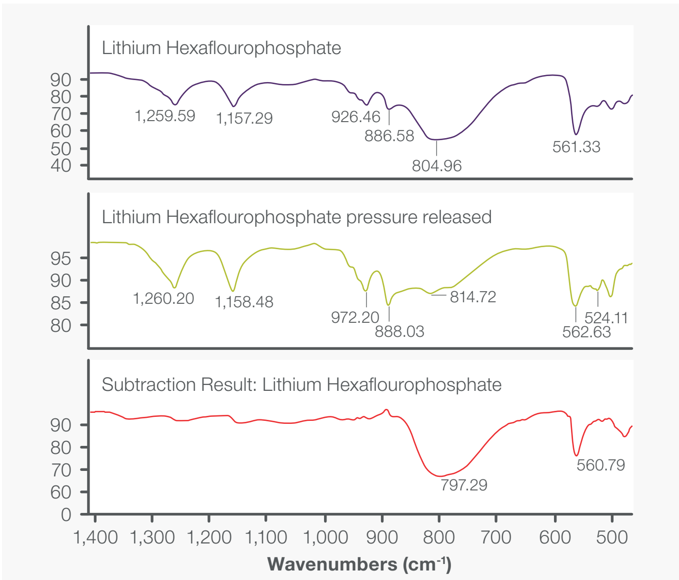


Figure 2. Spectra from a $LiPF_6$ sample, acquired on the Nicolet iS5 FTIR Spectrometer in a glove box.

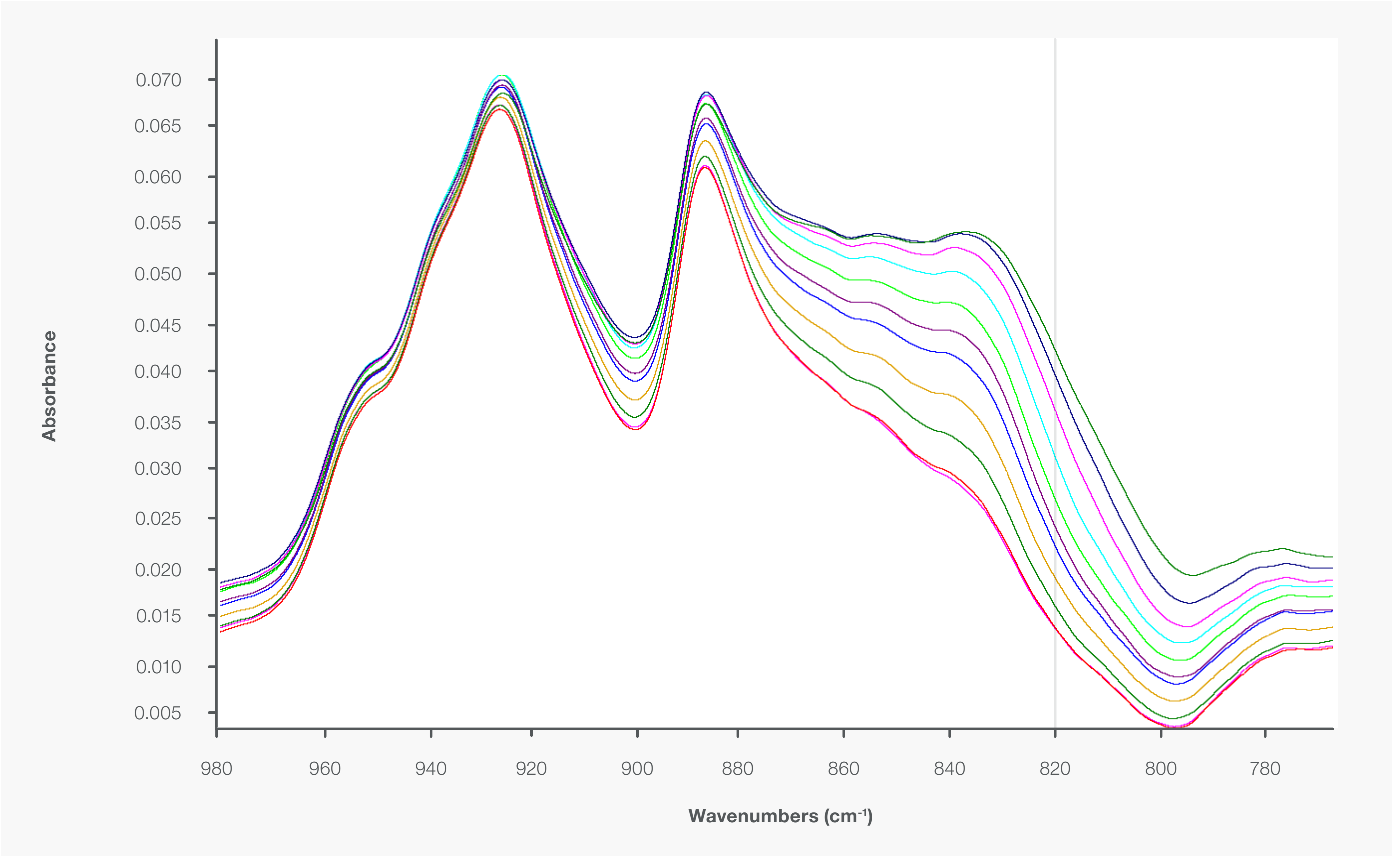


Figure 3. Spectra collected every half hour overnight, showing the loss of the hexafluorophosphate peak near 820 cm^{-1}.

To observe any changes in the sample over time, a workflow was developed to acquire spectra every half hour overnight. In this series of spectra, shown in Figure 3, the shoulder near 820 cm^{-1} (corresponding to the hexafluorophosphate) decreases, suggesting further decomposition of the sample.

Conclusions

The Nicolet iS5 FTIR Spectrometer, when equipped with a diamond ATR accessory, can operate effectively in the argon environment of a glove box. Excellent spectra were obtained from several lithium salts, and an overnight test was able to clearly monitor sample degradation.

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