Application Note # AN 56

FT-IR-Spectroscopy inside a Glovebox

Introduction

Infrared spectroscopy, combined with the (Attenuated Total Reflectance) ATR technique is ideal for the investigation of liquids and powders without any sample preparation. ATR spectroscopy, also known as internal reflection spectroscopy, is a versatile and non-destructive technique that can be used to measure the IR-spectrum. To perform the analysis, the sample is placed in contact with the surface of an IR transmitting crystal. The IR light is reflected from the inside surface of the crystal, but it penetrates a small distance into the sample and is therefore partially absorbed. No sample preparation is needed and the only requirement being that the sample is in intimate contact with the crystal surface. Liquid samples can simply be placed directly onto the crystal for the measurement, whereas Powder samples can be forced into close contact with the crystal surface using a press or clamp.

Gloveboxes are often used for the synthesis and handling of air and moisture sensitive compounds in an inert atmosphere, usually nitrogen. For analysis, sensitive liquid samples prepared inside the glovebox are traditionally injected into liquid cells and transferred to the instrument outside the glovebox. Unfortunately, the requirement for a sealed liquid cell precludes the possibility of using the quicker and easier ATR techniques to obtain the IR spectrum. Analysis of powder and solid samples is even more time consuming, and usually requires a complicated procedure to transfer the sample to the spectrometer.

ALPHA-P working in the Glovebox

Experimental Conditions

An alternative approach to transferring the sample to the spectrometer is to place the FT-IR spectrometer inside the glovebox. Until recently this approach has been difficult, however, because FT-IR spectrometers were too large to be passed into and out of most standard gloveboxes via the antechamber. This meant they had to be placed inside the glovebox more-or-less permanently, where they used up large amounts of the limited space available, and their accessibility for other measurements was restricted. With the introduction of the ALPHA FT-IR from Bruker, this problem has been solved. With only 8 x 11 inches footprint,

the ALPHA is the world's smallest FT-IR, and can easily be passed through the antechamber of a standard glovebox. Its electronics are also stable in a vacuum environment and the internal desiccant can easily be removed by the user, ensuring rapid exchange of the air inside the spectrometer with the inert gas atmosphere. The ALPHA can even communicate with a computer outside the glovebox, via a wireless connection.

Results

As an example, figure 1 shows the spectrum of LiAlCl4, one of the typical compounds used in the synthesis of electrolytes for modern power cells. The Li and Al based electrolytes in these cells are less chemically aggressive than those in previous generation cells and have a higher conductivity, meaning that the cells have significantly more power. These compounds are sensitive to moisture and oxygen, however, and the infraread analysis must be performed in an inert atmosphere.

Conclusion

Since there is no sample preparation required, and the sampling is easy with the ATR crystal, the ALPHA is clearly ideal for use inside a glovebox. The samples can be easily placed into position for analysis, even when wearing the thick rubber gloves. Using an FT-IR spectrometer inside the glovebox is safer, easier and much faster than transferring the samples out of the glovebox.

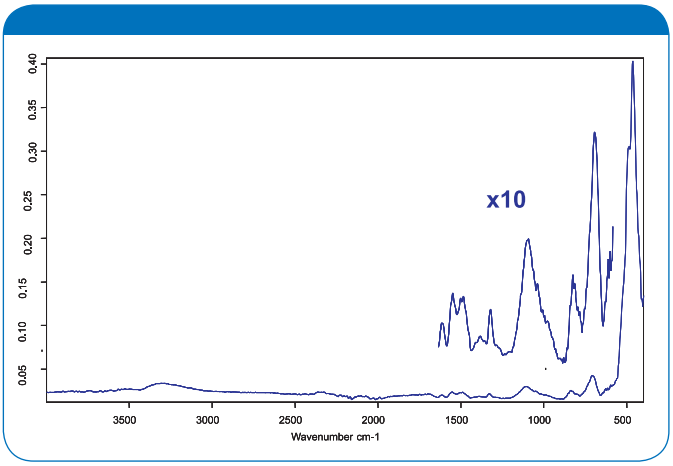

Fig 1: ATR-spectrum of $LiAlCl_4$ in the glove box under nitrogen atmosphere.

www.brukeroptics. com

Bruker Optik GmbH
Ettlingen · Germany
Phone +49 (7243) 504-2000
Fax +49 (7243) 504-2050
info@brukeroptics.de

Bruker Optics Inc.
Billerica, MA · USA
Phone +1 (978) 439-9899
Fax +1 (978) 663-9177
info@brukeroptics.com

Bruker Hong Kong Ltd.
Hong Kong
Phone +852 2796-6100
Fax +852 2796-6109
hk@brukeroptics.com.hk