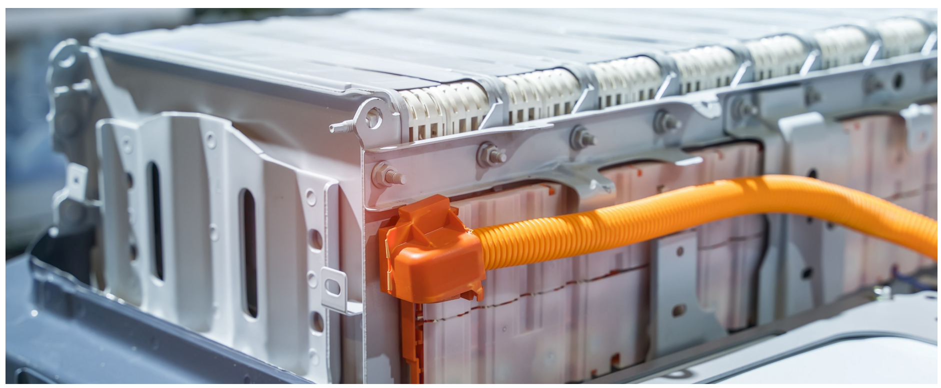
Application Note

Chemicals and Energy

Quick and Easy Material Identification of Solvents Used in Lithium-Ion Batteries by FTIR

Using the Agilent Cary 630 FTIR Spectrometer to identify common LIB electrolyte solvents



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Abstract

The demand for lithium-ion batteries (LIBs) is increasing due to the widespread use of portable electronic devices and the rise in popularity of electric vehicles (EVs). There is also a growing need for battery storage associated with sustainable electricity generated from intermittent sources such as wind, solar, and tidal. Manufacturers of LIB electrolytes must quality assure (QA) raw materials to ensure the components meet the required specifications before use. This study demonstrates the suitability of the Agilent Cary 630 FTIR spectrometer with Attenuated Total Reflectance (ATR) sampling technology for the fast and reliable identification of LIB electrolyte solvents using a simple method. The methodology is also useful for research and development (R&D) teams working to improve battery technology.

Introduction

The electrolyte is a key component of lithium-ion (Li-ion) batteries (LIBs), as it facilitates the transfer of charged ions between the anode and cathode during battery operation. The overall performance of LIBs in terms of cost, capacity, charging time, and lifetime, relies heavily on the composition of the electrolyte. LIB-electrolytes contain lithium salts, solvents, and additives.[1] Lithium hexafluorophosphate ($LiPF_6$) dissolved in carbonate solvents, such as ethylene carbonate (EC), diethyl carbonate (DEC), dimethyl carbonate (DMC), and ethyl methyl carbonate (EMC) is a commonly used electolyte.[2,3]

Raw materials used in the production of batteries play a critical role in the overall performance of LIBs, as these materials can affect the reliability and durability of the final products. To ensure that the correct raw material is used in the manufacturing process, raw material identification testing is an essential QA and safety analysis in the LIB industry.

Fourier Transform Infrared Spectroscopy (FTIR) spectroscopy is a non-destructive technique that is widely used for raw material identification testing applications. FTIR yields a characteristic chemical fingerprint of the sample by measuring the absorption of IR radiation. The easy-to-use technique, which does not require any sample preparation steps, provides rapid identification of materials.

In this study, the Agilent Cary 630 FTIR spectrometer fitted with a diamond ATR module (Figure 1) was used to qualify commonly used LIB-electrolyte solvents. The note describes the creation of a reference spectral library using the Agilent MicroLab software and the application of a method-based approach to confirm the identity of various electrolyte solvents.

Figure 1. The Cary 630 FTIR spectrometer with its ultracompact, lightweight design (20 x 20 cm and 3.6 kg) can be easily handled and positioned relative to the sample, ensuring high-quality results.

Experimental

Instrumentation

Two Cary 630 FTIR spectrometers, both fitted with a diamond ATR module, were used in this study. An instrument at the Global Solution Development Center, Agilent Technologies, Inc., Singapore was used to create the spectral reference library listed in Table 1. The library was used to create a routine material identification method. This method was then transferred to another instrument at the Agilent Spectroscopy Center of Excellence in Melbourne, Australia, where it was used to identify four "unknown" solvents (Figure 2).

Library generation

The library was developed using the chemicals listed in Table 1. Spectral libraries can be easily created, maintained, and managed in the MicroLab software. A new library can be created in a few seconds and spectra can be added directly from the results screen, either at the time of creation or at any other time.

Table 1. LIB solvents used as spectral reference material for library generation.

Solvent Name	Short Name	CAS	Supplier
Ethylene Carbonate	EC	96-49-1	Sigma-Aldrich Co
Dimethyl Carbonate	DMC	616-38-6	Sigma-Aldrich Co
Ethyl Methyl Carbonate	EMC	623-53-0	Tokyo Chemical Industry Co. LTD
Ethyl Acetate	EA	141-78-6	Sigma-Aldrich Co

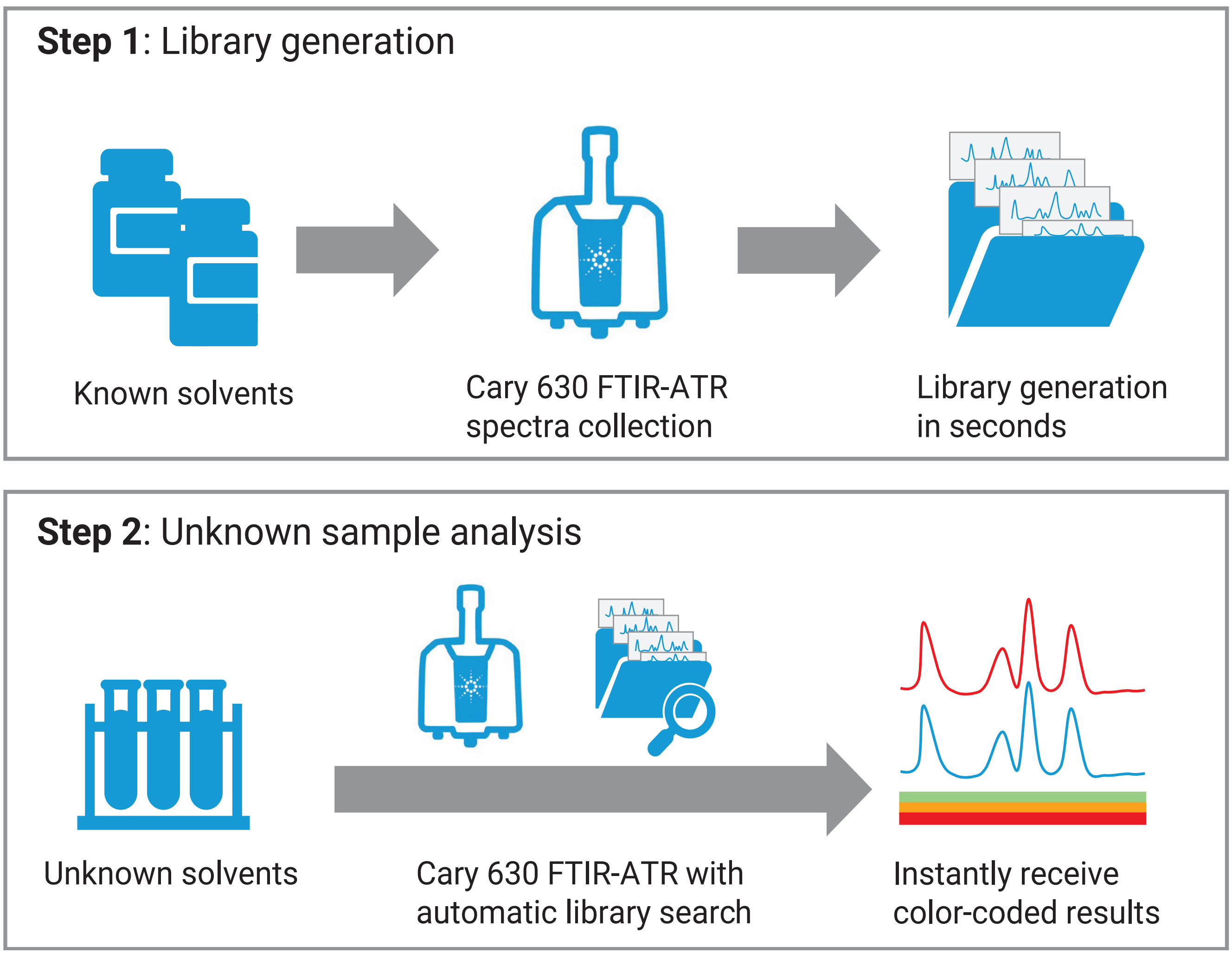

Figure 2. Identification method creation for LIB solvents identification using the Agilent Cary 630 FTIR spectrometer and Agilent MicroLab software.

Figure 3. The intuitive Agilent MicroLab software makes finding answers with the Agilent Cary 630 FTIR spectrometer as easy as 1, 2, 3. The picture-driven software also reduces training needs and minimizes the risk of user-based errors.

Software

The MicroLab instrument control software for the Cary 630 FTIR spectrometer uses a pictorial interface to guide users through the steps of the analysis, from sample introduction to reporting (Figure 3).

Samples

The user-generated library was tested by analyzing four independent "unknown" solvent samples (these samples were commercially available solvents with the substance name stated on the container label) using the Cary 630 FTIR. The samples included two solutions of ethyl methyl carbonate, dimethyl carbonate, and ethyl acetate.

Analysis

To analyze liquid samples using the Cary 630 FTIR with ATR module, a small drop of the sample is placed onto the ATR crystal. The measurement is taken, and, after completion, the crystal can be wiped clean using ethanol, if necessary.

Instrument operating conditions and parameters are shown in Table 2.

Table 2. Agilent Cary 630 FTIR–ATR operating parameters.

Parameter	Setting
Method	Library search
Library Used	User-generated LIB solvents library
Search Algorithm	Similarity
Spectral Range	4,000 to 650 cm^{-1}
Background Scans	10
Sample Scans	24
Spectral Resolution	2 cm^{-1}
Background Collection	Air
Zero Fill Factor	None
Apodization	HappGenzel
Phase Correct	Mertz
Color-Coded Confidence Level Thresholds	Green (high confidence): >0.95 Yellow (medium confidence): 0.90 to 0.95 Red (low confidence): <0.90

Results and discussion

Each of the four "unknown" solvents was analyzed using the Cary 630 FTIR. Using the Similarity algorithm to search the user-generated LIB-solvents spectral library, unknown samples 1 and 2 were identified as EMC with a hit quality index (HQI) of 0.99393 and 0.94530, respectively. Unknown sample 3 was identified as DMC with an HQI of 0.97820 and sample 4 as EA (HQI of 0.99679), as shown in Table 3.

The HQI, which is automatically calculated for each library item by the software, 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. Analysts can set their own HQI-based thresholds in the MicroLab software.

Table 3. LIB solvents identification results obtained using the Agilent Cary 630 FTIR–ATR, user-generated LIB solvents library, and Similarity search algorithm.

Sample Name	Material Identification	Hit Quality Index
Unknown Sample 1	Ethyl methyl carbonate (EMC)	0.99393
Unknown Sample 2	Ethyl methyl carbonate (EMC)	0.94530
Unknown Sample 3	Dimethyl carbonate (DMC)	0.97820
Unknown Sample 4	Ethyl acetate (EA)	0.99679

Color-coded results

For easy review of the data generated by the Cary 630 FTIR, the material identification results obtained for each sample are color-coded based on user-defined confidence level thresholds (Figure 4).

In this study, results with an HQI above 0.95 were color-coded in green, indicating a good spectral match and providing a high degree of confidence in the identification of the material. As shown in Figure 4, unknown sample 2 was identified with medium confidence (HQI: 0.90 to 0.95) and was color-coded in orange. Depending on the objectives of the analysis, a medium-confidence result may indicate to the analyst that the tested solvent batch needs to be investigated further.

Color-coding the results turns the Cary 630 FTIR system into an easy-to-use, turnkey solution that enables quick decision-making. Once the sample has been measured, the MicroLab software shows the final answer directly on screen, without any input needed by the user. The software automatically performs the library search and provides the operator with the final color-coded results.

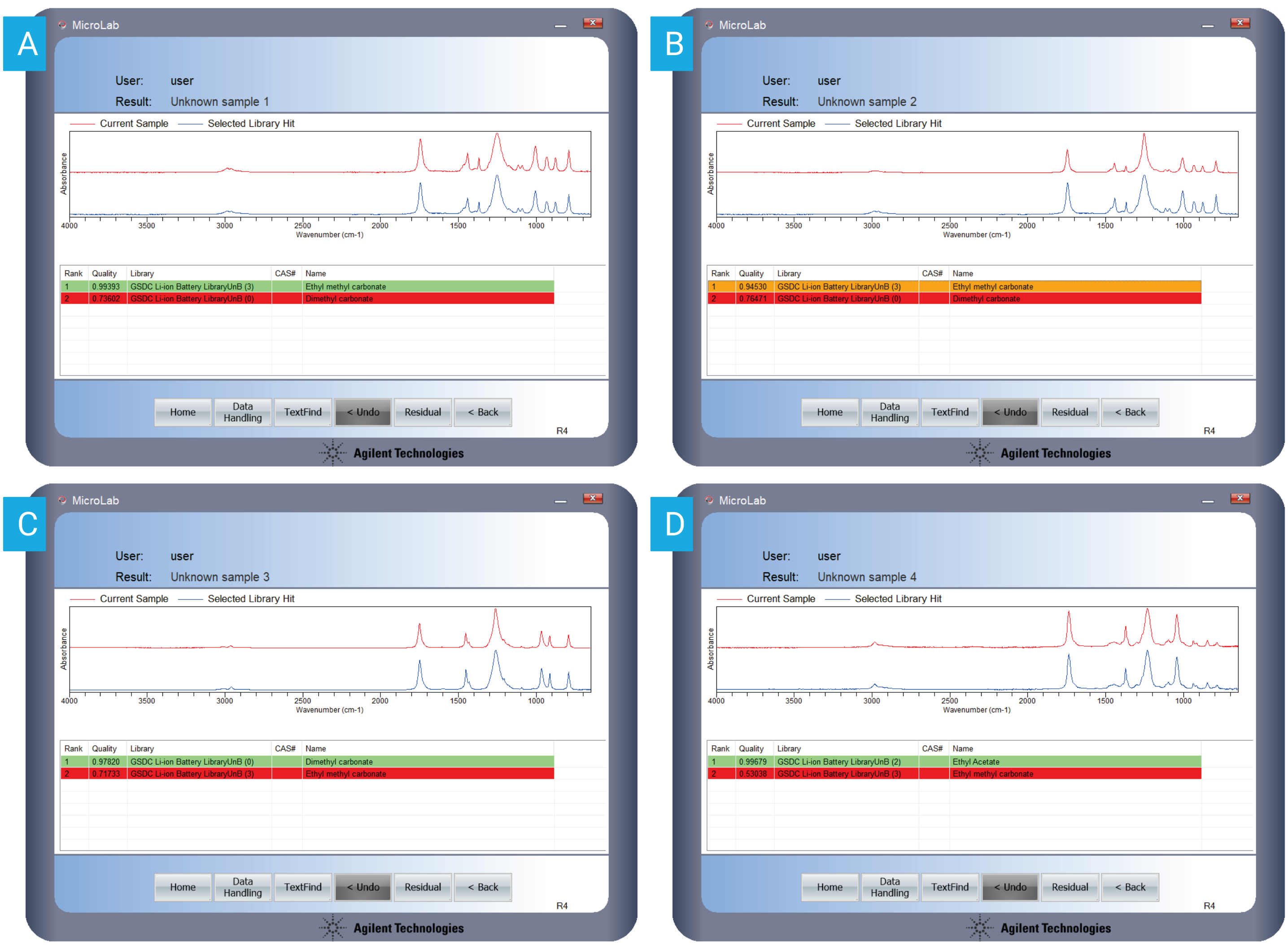

Figure 4. The Agilent Cary 630 FTIR spectrometer identification analysis of the four LIB solvents samples (red traces) and library hit (blue traces). The table shows the hit quality, library used, and the hit name for unknown samples 1 to 4 (labeled A to D, respectively).

Conclusion

The Agilent Cary 630 FTIR spectrometer provides a simple-to-use methodology for the material identification of solvents used in the production of lithium-ion battery (LIB) electrolytes.

The Cary 630 FTIR and MicroLab software facilitated the quick and easy generation of a LIB solvent library, which enabled the fast identification of four "unknown" solvent samples.

The MicroLab software applied color-coding to the identification results based on the hit-quality index (HQI), simplifying the review of the data. All four solvents were correctly identified via the library, although one of the samples was flagged as needing further investigation.

This study has shown the flexibility of the Cary 630 FTIR fitted with the ATR sampling module for material qualification of LIB-related solvents. The Cary 630 FTIR provides accurate and reliable material identification methods for manufacturers of LIB-raw materials and LIB producers. It also supports R&D groups involved in the development of next-generation materials.

References

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Further information

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

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