Application Note

Materials

# Low-Level UV-Vis Haze Detection for Improved Reliability of Li-Ion Battery Electrolytes

## Overcoming quality control blind spots with the Cary 60 UV-Vis and the diffuse reflectance accessory (DRA)


**Author**

Wesam Alwan
Agilent Technologies, Inc.



## Abstract

The performance, safety, and lifespan of lithium-ion batteries are closely tied to the purity and stability of their electrolyte solutions. A key challenge in maintaining electrolyte quality is the presence of water-sensitive components that are prone to degradation. Even trace amounts of particulate contamination, often invisible to the naked eye, can compromise electrolyte performance and, ultimately, battery efficiency. This application note highlights the use of UV-Vis spectroscopy, specifically haze (turbidity) measurements, as a rapid and effective quality control technique for detecting particulate contaminants. The Agilent Cary 60 UV-Vis spectrophotometer equipped with a diffuse reflectance accessory (DRA) offers a sensitive method for monitoring and preserving electrolyte integrity. The proposed test method aims to raise awareness and address the challenges of electrolyte degradation.

## Introduction

Lithium-ion batteries (LIBs) have become indispensable in modern life, powering everything from portable electronics to electric vehicles. As demand grows for higher-capacity, longer-lasting, and more efficient batteries, research efforts continue to focus on optimizing every component, including the electrolyte.

The electrolyte plays a critical role in battery performance by enabling the transport of ions between the two electrodes. It typically consists of a lithium salt, most commonly lithium hexafluorophosphate ($LiPF_6$), dissolved in a mixture of organic carbonate solvents such as ethylene carbonate (EC), ethyl methyl carbonate (EMC), and dimethyl carbonate (DMC). However, these electrolyte formulations are highly sensitive to moisture and prone to degradation over time. This degradation can lead to the formation of particulate matter or impurities that are invisible to the naked eye but can significantly impact electrolyte quality during manufacturing.

In this application note, we explore how the Agilent Cary 60 UV-Vis spectrophotometer, paired with an Agilent Cary 60 diffuse reflectance accessory (DRA), can be used to assess LIB electrolyte quality.

Traditional UV-Vis transmission compartments can struggle with low-scattering samples like electrolytes, as scattered light may bypass the detector and lead to inaccurate readings. The Cary 60 DRA mitigates this limitation by enhancing light collection efficiency, making it well-suited for low-level haze analysis.

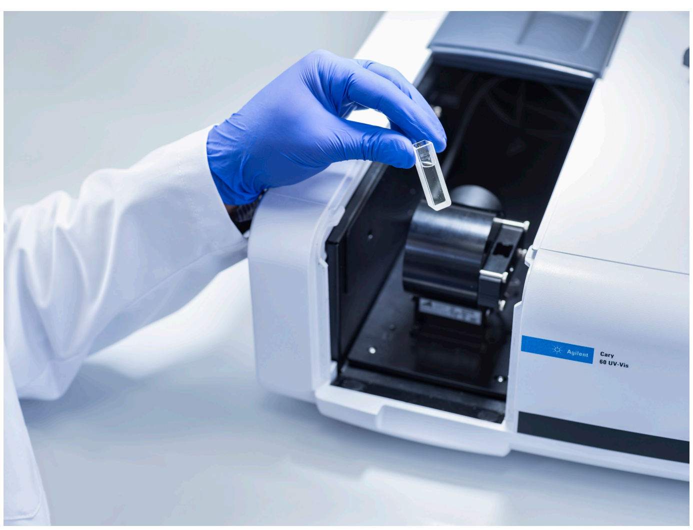


**Figure 1.** The Agilent Cary 60 UV-Vis spectrophotometer with the diffuse reflectance accessory and the standard 10 mm cuvette holder.

The instrumentation was used to assess haze levels at low levels by measuring scattered light caused by unseen particulates suspended in the liquid. Elevated haze levels may indicate the presence of undissolved materials or contaminants, which can interfere with ion flow, compromise battery safety, and reduce overall cell performance. To ensure optimal battery performance, electrolyte haze levels should be minimized during manufacturing, and the proposed method offers an effective means of detecting potential degradation.

## Experimental

### Samples and sample preparation

Two battery-grade LIB electrolytes, obtained from Merck, were used in this study:

- **Electrolyte A:** Lithium hexafluorophosphate solution in ethylene carbonate and ethyl methyl carbonate (1.0 M $LiPF_6$ in EC/EMC, 50/50 v/v; CAS number: 21324-40-3; product no: 746738-25ML)
- **Electrolyte B:** Lithium hexafluorophosphate solution in ethylene carbonate and dimethyl carbonate (1.0 M $LiPF_6$ in EC/DMC, 50/50 v/v; CAS number: 21324-40-3; product no: 746711-25ML)

Initially, 5 mL of each electrolyte was transferred into a glass vial, which was then sealed, and stored in a dark safety cabinet at room temperature for varying durations. The samples were analyzed immediately and at two more time points: after 1 day and after 6 months. Before measurement, 3 mL of the electrolyte was transferred into a standard 10 mm quartz cuvette for haze analysis.

### Instrumentation

Diffuse transmission spectra and haze measurements were collected using the Agilent Cary 60 UV-Vis spectrophotometer controlled by the Agilent Cary WinUV Color software. The software allows users to select from a range of calculation options, including standardized color coordinate systems such as those defined by the Commission Internationale de l'Eclairage (CIE).

For this application, the "Haze as per D1003" calculation mode was selected, which requires four individual scans to be performed using the DRA integrating sphere.

The four scan configurations were as follows:

- **T1:** Baseline scan with the white (PTFE) reference plate in position
- **T2:** Scan with both the sample and white reference plate in place
- **T3:** Scan with no sample in place and the white reference plate removed; light passes through the sphere and exits (a light trap may be used or the compartment closed to simulate one)
- **T4:** Scan with the sample in place and no white reference plate; light exits the sphere into the compartment (a light trap may be used or the compartment closed to simulate one)

Table 1 summarizes the instrument parameters used for the measurements.

**Table 1.** Agilent Cary 60 UV-Vis and Agilent WinUV Color software settings for haze measurements.

| Parameter | Value |
|---|---|
| Scan Range | 380 to 780 nm |
| Data Interval | 1 nm |
| Y Mode | %T |
| Spectral Bandwidth | 1.5 nm |
| Signal Averaging Time | 0.100 s |
| CIE Illuminant | CIE A |
| Observer Angle | 2 degrees |
| Calculations | Haze (ASTM D1003) |

## Results and discussion

Haze percentage levels were calculated using the Cary WinUV Color software Haze calculations feature.

As shown in Table 2, fresh electrolyte samples (1.0 M $LiPF_6$ in EC/DMC and 1.0 M $LiPF_6$ in EC/EMC) exhibited low haze values of 0.59 and 0.20%, respectively, indicating high purity and clarity.

After storing the samples in sealed glass vials for 24 hours, measurements were repeated using the same instrument parameters. Although no visual difference in clarity was observed, the haze levels increased significantly as follows:

- 1.0 M $LiPF_6$ in EC/DMC—haze increased from 0.59 to 2.15%
- 1.0 M $LiPF_6$ in EC/EMC—haze increased from 0.20 to 1.06%

These results highlight the sensitivity of the haze measurement to subtle changes in sample quality, which may go unnoticed by the naked eye. In contrast, samples stored for over six months showed obvious signs of degradation, including visible yellowish discoloration. The haze levels of these samples increased to 9.73 and 13.39% for EC/DMC and EC/EMC electrolytes, respectively, likely due to moisture-induced breakdown of the electrolyte components.

**Table 2.** Haze levels of LIB electrolytes under different storage conditions, measured by the Agilent Cary 60 UV-Vis with DRA.

| Sample | Status | Haze (%) |
|---|---|---|
| 1.0 M $LiPF_6$ in EC/DMC | Fresh | 0.59 |
| 1.0 M $LiPF_6$ in EC/DMC | Stored for 1 day | 2.15 |
| 1.0 M $LiPF_6$ in EC/DMC | Stored for 6 months | 9.73 |
| 1.0 M $LiPF_6$ in EC/EMC | Fresh | 0.20 |
| 1.0 M $LiPF_6$ in EC/EMC | Stored for 1 day | 1.06 |
| 1.0 M $LiPF_6$ in EC/EMC | Stored for 6 months | 13.39 |

Figure 2 shows the appearance of 1.0 M $LiPF_6$ in EC/DMC under different storage conditions. While early-stage degradation cannot be detected visually, the DRA captures light scattered by fine particulates providing a highly sensitive method to assess electrolyte quality. Because the integrating sphere collects all scattered light, this technique offers a powerful means to monitor even minor changes in electrolyte condition, making it especially useful for assessing samples stored or transported under varying environmental conditions.

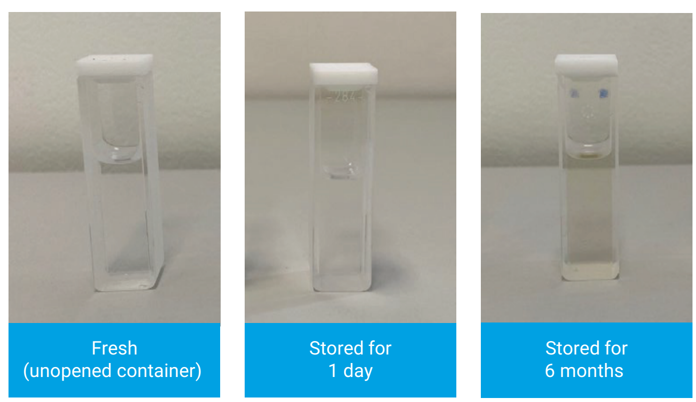
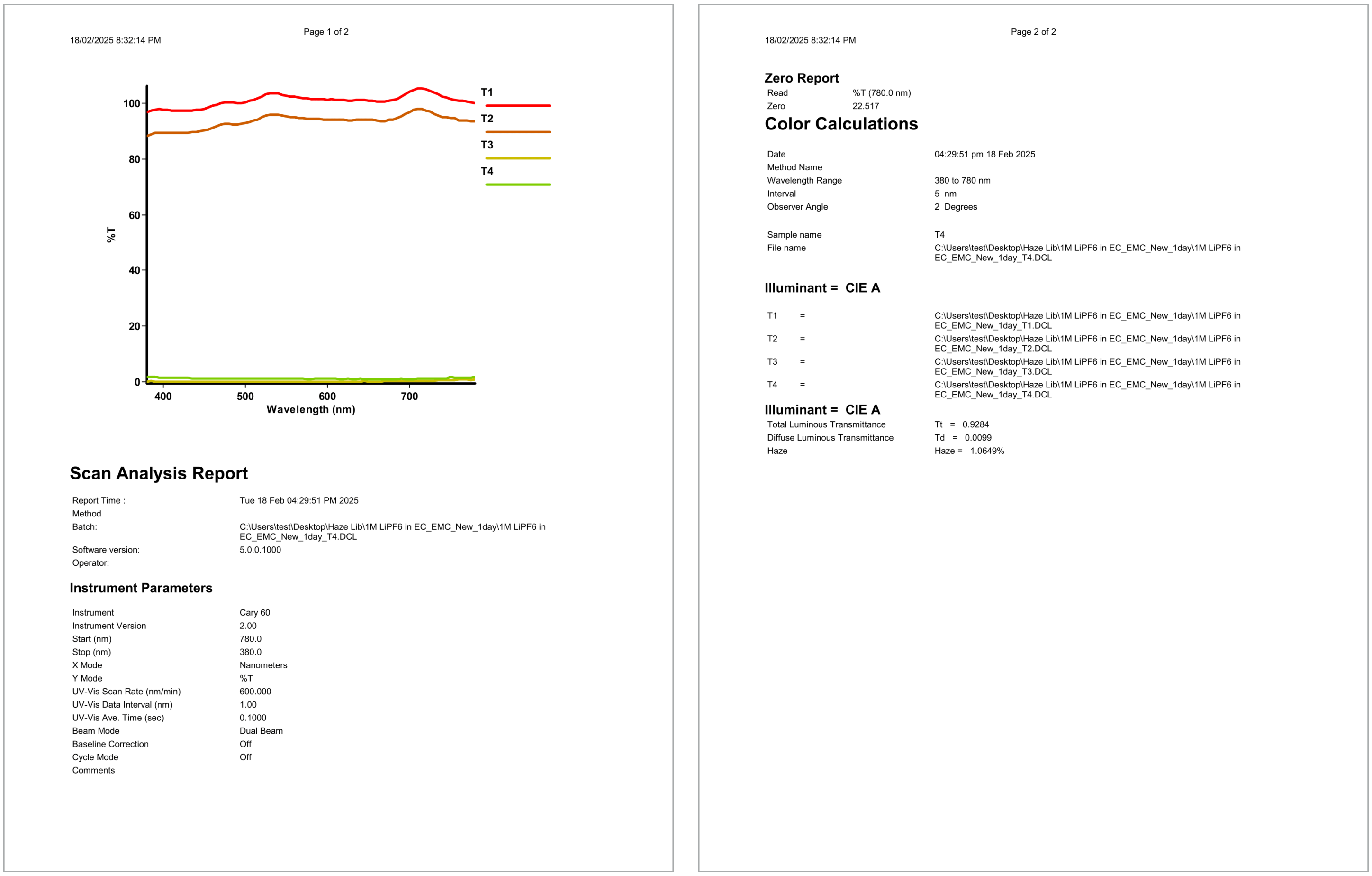



**Figure 2.** LIB electrolyte (1.0 M $LiPF_6$ in EC/DMC) in glass vials at different times. While particulates may not be visible to the naked eye, haze measurements using the Agilent Cary 60 UV-Vis with DRA reveal underlying turbidity and degradation after 1 day and 6 months.

The Cary WinUV Color software provides a simple method for haze, with built-in haze calculation options and the ability to automatically generate detailed reports.

As shown in Figure 3, the software enables haze calculation and facilitates the generation of comprehensive reports for each analysis.




**Scan Analysis Report**

| | |
|---|---|
| Report Time : | Tue 18 Feb 04:29:51 PM 2025 |
| Method | |
| Batch: | C:\Users\test\Desktop\Haze Lib\1M LiPF6 in EC_EMC_New_1day\1M LiPF6 in EC_EMC_New_1day_T4.DCL |
| Software version: | 5.0.0.1000 |
| Operator: | |

**Instrument Parameters**

| | |
|---|---|
| Instrument | Cary 60 |
| Instrument Version | 2.00 |
| Start (nm) | 780.0 |
| Stop (nm) | 380.0 |
| X Mode | Nanometers |
| Y Mode | %T |
| UV-Vis Scan Rate (nm/min) | 600.000 |
| UV-Vis Data Interval (nm) | 1.00 |
| UV-Vis Ave. Time (sec) | 0.1000 |
| Beam Mode | Dual Beam |
| Baseline Correction | Off |
| Cycle Mode | Off |
| Comments | |

18/02/2025 8:32:14 PM

Page 2 of 2

**Zero Report**

| | |
|---|---|
| Read | %T (780.0 nm) |
| Zero | 22.517 |

**Color Calculations**

| | |
|---|---|
| Date | 04:29:51 pm 18 Feb 2025 |
| Method Name | |
| Wavelength Range | 380 to 780 nm |
| Interval | 5 nm |
| Observer Angle | 2 Degrees |
| Sample name | T4 |
| File name | C:\Users\test\Desktop\Haze Lib\1M LiPF6 in EC_EMC_New_1day\1M LiPF6 in EC_EMC_New_1day_T4.DCL |

**Illuminant = CIE A**

| | |
|---|---|
| T1 = | C:\Users\test\Desktop\Haze Lib\1M LiPF6 in EC_EMC_New_1day\1M LiPF6 in EC_EMC_New_1day_T1.DCL |
| T2 = | C:\Users\test\Desktop\Haze Lib\1M LiPF6 in EC_EMC_New_1day\1M LiPF6 in EC_EMC_New_1day_T2.DCL |
| T3 = | C:\Users\test\Desktop\Haze Lib\1M LiPF6 in EC_EMC_New_1day\1M LiPF6 in EC_EMC_New_1day_T3.DCL |
| T4 = | C:\Users\test\Desktop\Haze Lib\1M LiPF6 in EC_EMC_New_1day\1M LiPF6 in EC_EMC_New_1day_T4.DCL |

**Illuminant = CIE A**

| | |
|---|---|
| Total Luminous Transmittance | Tt = 0.9284 |
| Diffuse Luminous Transmittance | Td = 0.0099 |
| Haze | Haze = 1.0649% |

**Figure 3.** Screenshot of the Agilent Cary WinUV Color software showing the haze calculation and an example of the report generated after analysis of a 1.0 M $LiPF_6$ in EC/EMC electrolyte solution.

## Conclusion

This study demonstrated the effectiveness of the Agilent Cary 60 UV-Vis equipped with a diffuse reflectance accessory (DRA) for assessing the quality of LIB electrolytes through haze measurements. The innovative method enabled the detection of particulates and early signs of degradation that are not visible to the naked eye, but can significantly impact battery performance, safety, and lifespan. Haze levels of the solutions increased notably with storage time and exposure to air, confirming the instrument's sensitivity to moisture-induced changes in electrolyte composition.

Measuring low-scattering samples in a standard UV-Vis transmission compartment can be difficult because scattered light may miss the detector, causing inaccurate measurements. The Cary 60 DRA improves light collection efficiency, making it ideal for low-level haze analysis. As the integrating sphere collects all scattered light, this technique monitors minor changes in electrolyte condition, which is useful for assessing samples stored or transported under different environmental conditions.

With the added advantage of automated calculations and reporting through the Agilent Cary WinUV Color software, the method offers a reliable, user-friendly solution for quality control in LIB electrolyte manufacturing and storage.

The Cary 60 UV-Vis method helps manufacturers make informed decisions about handling and using electrolytes, ultimately supporting the production of safer, higher-performing batteries.

## Further information

- Agilent Cary 60 UV-Vis Spectrophotometer
- Cary 60 UV-Vis Diffuse Reflectance Accessory
- Color Measurements by Agilent UV-Vis and UV-Vis-NIR Spectrophotometers
- Cary WinUV Software for UV-Vis Applications
- Cary WinUV Color Measurement Software
- UV-Vis Spectroscopy and Spectrophotometry FAQs
- UV-Vis Applications Guide

**www.agilent.com/chem/cary-60-uv-vis**

DE-007836


Printed in the USA, July 15, 2025
5994-8446EN

Agilent
Trusted Answers