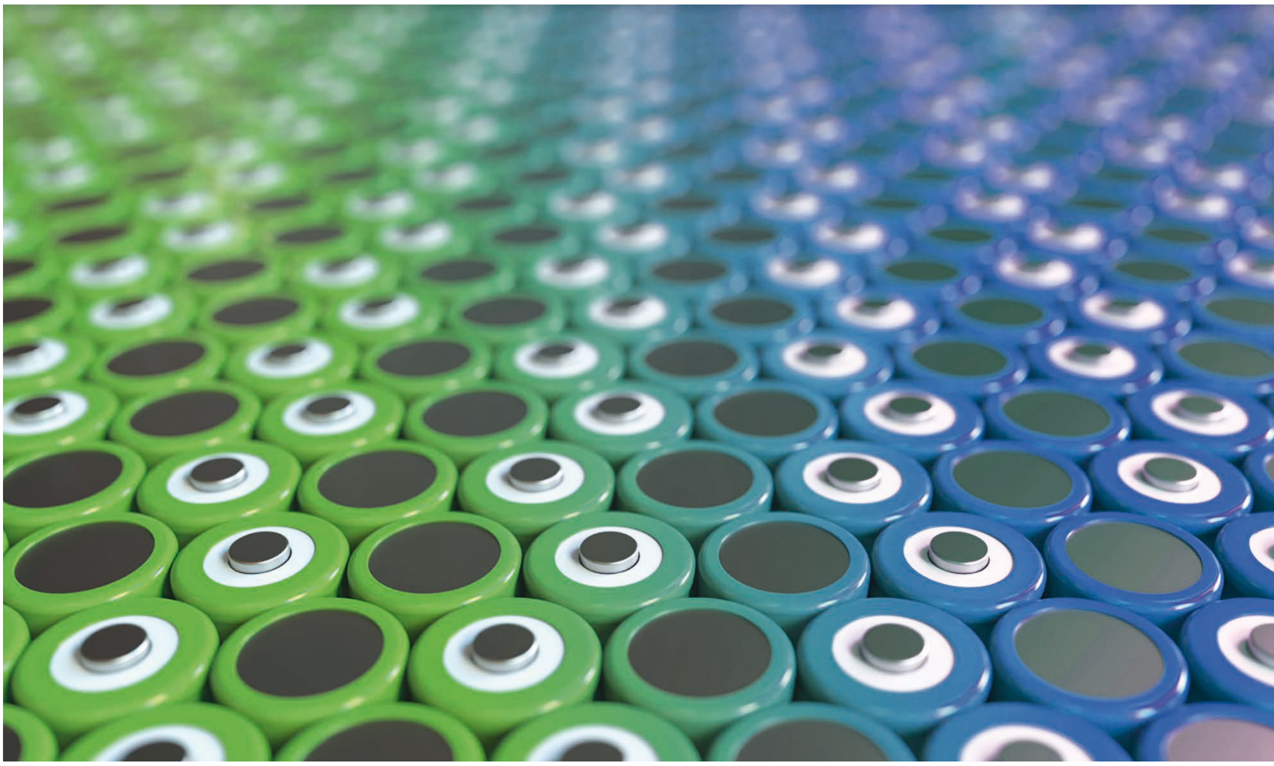
Application Note

Li-Ion Battery Electrolyte Analysis

Using InLab® 710 Conductivity Sensor

Using the InLab 710 conductivity sensor from METTLER TOLEDO for measuring the electrolyte of Li-ion batteries offers several advantages, like linear response for a broad conductivity range. The chemically resistant body of the sensor is ideal for measurements in harsh chemicals and organic solvents. The document also shares tips and hints related to Good Calibration and Measurement Practices to reduce errors while measuring the conductivity of battery electrolyte.

With their light weight and high energy efficiency, Li-ion batteries (LiB) are used in a wide range of everyday devices. Being responsible for the generation and transfer of charged species between the two electrodes, the electrolyte facilitates the charging and discharging of the LiB. Commercial lithium-ion battery electrolytes often consist of lithium hexafluorophosphate ($LiPF_6$) dissolved in mixtures of cyclic and linear carbonates like ethylene carbonate (EC), ethyl methyl carbonate (EMC), diethyl carbonate (DEC), and dimethyl carbonate (DMC). The conductivity of electrolyte solutions varies based on the change in composition and hence becomes an important parameter in monitoring the electrolyte quality. The InLab 710 conductivity sensor by METTLER TOLEDO is the right choice to measure the conductivity of electrolytes used in Li-ion batteries.



METTLER TOLEDO

The Right Sensor Choice: InLab® 710 Conductivity Sensor

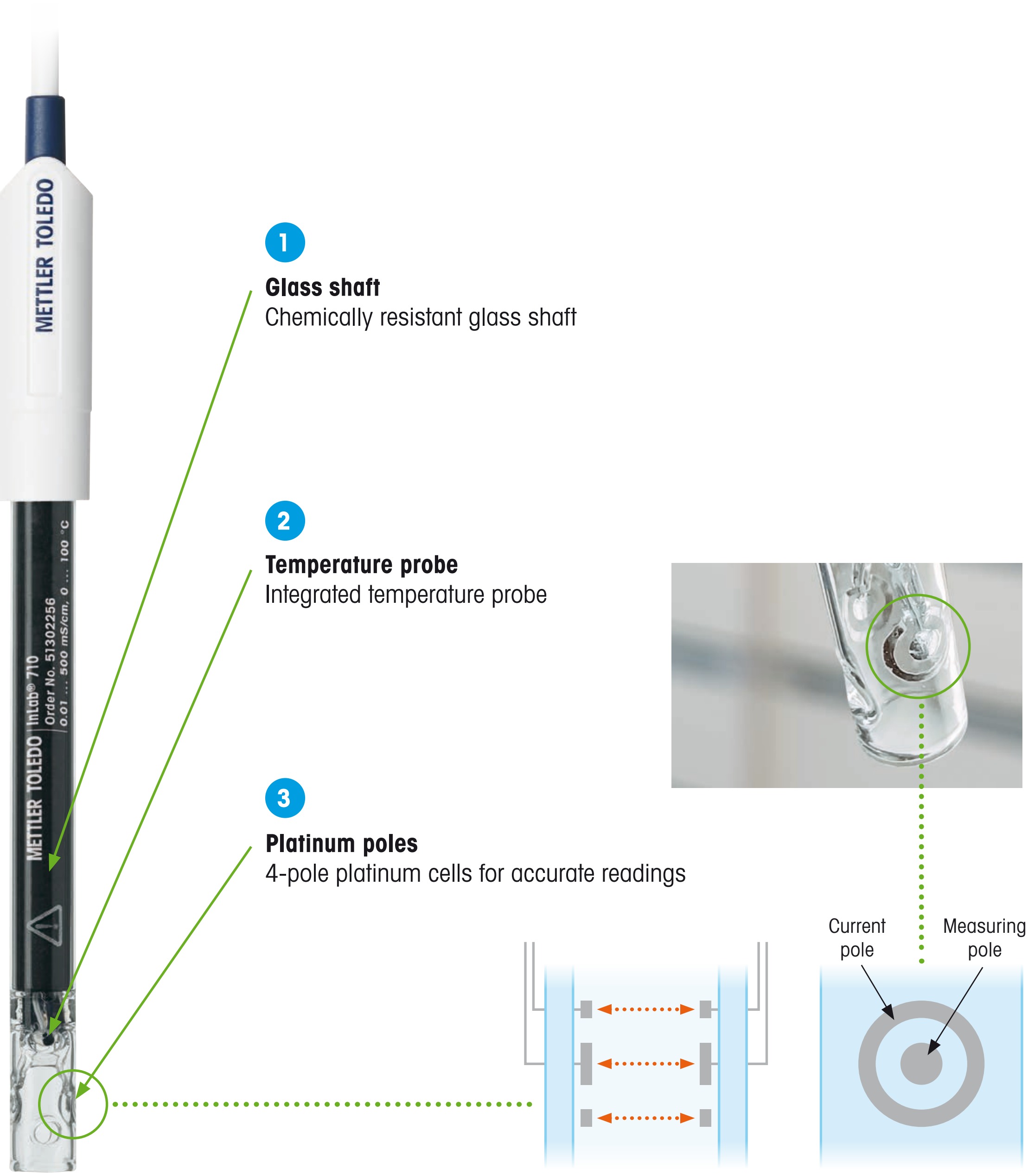


Figure 1. InLab 710 conductivity sensor.

No.	Challenge	Features	InLab 710 advantage
1	Organic solvent system	• Robust glass shaft material	The sensor is made of glass shaft material for measurement in harsh chemicals and organic solvents.
2	Wide temperature range	• Integrated temperature probe	Conductivity is temperature dependent. With the integrated temperature probe, the sample temperature is noted. Hence, results can always be matched to the right temperatures.
3	High conductivity range measurement	• 4 platinum poles • Cell constant (0.8 cm^{-1})	Provide linear measurements over a large conductivity range and are best suited for conductivity measurement in the range of 0.01–500 mS/cm. Ideal for high conductivity measurement of electrolyte samples.

Table 1. Features and advantages of the InLab 710 conductivity sensor.

Applying Good Practices for Consistent and High-Quality Results

A. Calibration and Verification

- Calibrate the conductivity sensor using MT conductivity standard solution, preferably 12.88 mS/cm, before the first use. The InLab® 710 conductivity sensor comes with a nominal cell constant; hence it is necessary to calibrate the sensor before the first use. The conductivity standard solution selected for the calibration should have a conductivity value nearing the sample conductivity. For example, use of 12.88 mS/cm standard solution for calibration if the sample conductivity value is expected around 5 to 10 mS/cm.
- Subsequently, perform a verification by measuring the conductivity of a standard of known conductivity value, which is preferably different from the calibration standard. Ensure that the conductivity value lies within 2% of the conductivity value of the standard solution.
- To obtain accurate results, conductivity standards and sample solutions must be at the same temperature.
- Perform a verification intermittently after a few measurements, preferably after 10 sample measurement readings. Verification should be within 2% of the conductivity value, otherwise perform a calibration.
- Perform a verification before the first measurement of the day. If the verification fails, only then a calibration is needed for the sensor.
- Verification is necessary after every calibration.

B. Measurement

- Pour the electrolyte solution into a measurement beaker and ensure that the poles of the sensor are well immersed.
- Ideally, the sample beaker and sensor should be rinsed at least twice with the sample to rule out the possibility of contamination leading to additional errors in conductivity results.
- In the general meter settings, keep the temperature correction off.
- Check the endpoint type as automatic unless a manual or timed endpoint is required for a specific study. Endpoint criteria can be kept as Standard (default setting).
- Ensure the cell chamber is free of bubbles when measuring. To reduce air bubbles, immerse the sensor at an angle and then straighten to a vertical position. It is usually best to position the sensor in the middle of the beaker.
- Record the conductivity at the required temperature. For temperatures below 0 °C, it is recommended to use an external temperature probe. Select the temperature capture mode as 'external' in the general settings in such cases.
- Measure the samples in triplicates or as required by the standard protocol.
- Rinse the sensor with deionized water first, then rinse again using a solvent like an isopropyl alcohol solution after every measurement to ensure thorough cleaning. Next, clean the sensor using deionized water to rinse off the excess solvent. Gently blot dry the excess water using tissue paper.
- The entire process can be automated using an InMotion autosampler and LabX software.
  The only change, in this case, would be the use of a water-miscible organic solvent instead of distilled water for rinsing the sensor.

C. Maintenance

- The InLab 710 conductivity sensor must be cleaned with water-miscible solvents followed by rinsing with deionized water.
- The sensor should never be cleaned using sharp/hard objects as they could damage the poles.
  The use of harsh chemicals for cleaning is not recommended.
- In case of unstable readings, immerse the sensor in deionized water for at least half an hour to condition the sensor properly before performing the measurement anew.

D. Storage

- For storage, keep the sensor dry.
- Ensure that the connectors are kept free from moisture and dirt.

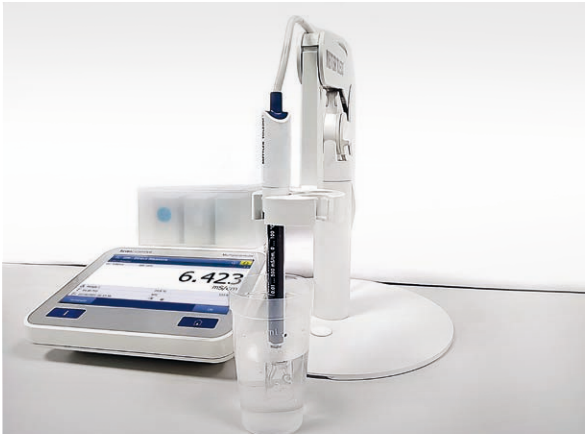


Figure 2. Measurement of conductivity sample using InLab 710.

Conductivity sample	Temperature (°C)	Conductivity value (mS/cm)	Standard deviation (n≥3)	Average response time (s)
Electrolyte Sample 1	25.8	6.42	0.01	6
Electrolyte Sample 2	25.0	2.26	0.01	6
Electrolyte Sample 2	35.0	2.82	0.02	6

Table 2. Conductivity values of electrolyte samples measured with InLab 710 sensor.

Results and Discussion

As can be seen in the table above, the conductivity of lithium-ion battery electrolyte samples comprising $LiPF_6$ salt dissolved in various organic carbonate solvents was measured, applying good practices as outlined in the previous section. Measurements were carried out using the SevenExcellence S700 conductivity meter and InLab 710 conductivity sensor as listed above in table 2.

Sample 2 was measured at constant temperatures of 25 °C and 35 °C. The temperature during the sample measurement was maintained using a thermostat. The conductivity measurement of lithium-ion battery electrolytes using the InLab 710 sensor yielded repeatable results. A standard deviation of less than 0.01 conductivity value (amounting to less than 1% RSD) and an average response time of around six seconds was observed. It was also possible to measure the conductivity of the sample at –10 °C with an InLab 710 conductivity sensor, indicating its applicability at subzero temperatures. Remember to use an external temperature probe for measurements below zero degrees. Please note that an appropriate setup for maintaining the sample and sensor at a constant temperature is needed to achieve consistent and precise results.

Conclusion

The InLab 710 conductivity sensor is recommended for the measurement of lithium-ion battery electrolyte solutions. It is made of chemical resistant glass shaft material and hence the best choice for measurements in organic electrolytes matrixes. This sensor can be paired up with benchtop meters like SevenExcellence and SevenDirect. Sample throughput may be maximized using an automated system, including a SevenExcellence meter, an InMotion autosampler, and LabX software. The use of Good Calibration and Measurement Practices, along with proper sensor maintenance, ensure accuracy of the results.

References

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- Buket Boz et al, (2021), Review-Electrolyte and Electrode Designs for Enhanced Ion Transport Properties to Enable High Performance Lithium Batteries, J. Electrochem. Soc. 168 090501.
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Further information from METTLER TOLEDO

- Good Electrochemistry Practice (GEP) for maintenance and proper handling of conductivity sensors and buffers: ▶ **www.mt.com/GEP**
- To learn the good practices of handling the pH electrode, watch our Electrode handling videos on YouTube: ▶ **www.mt.com/pHLab_ElectrodeHandlingVideos**
- Want to know the right sensor for your application? Visit our page on: ▶ **www.mt.com/sensor_finder**

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