ANALYTICAL SOLUTIONS FOR LITHIUM-ION BATTERY MATERIAL CHARACTERIZATION
Unlocking the full potential of lithium-ion batteries through material characterization

Greenhouse gas emissions such as carbon dioxide (CO₂) are the primary mitigation targets of the 2015 Paris Agreement. There are several mitigation strategies for achieving net-zero carbon emissions including more efficient transportation, adoption of renewable energy, and greater energy efficiency in homes and offices. One way to improve the efficiency of transportation, industrial equipment, and consumer electronics is by deploying lithium-ion battery technology.

Since Sony’s early introduction of lithium-ion batteries in 1991 for consumer electronics, lithium-ion batteries have experienced tremendous advancements in energy efficiency, power output, and safety leading to the adoption of the battery technology in transportation and energy storage. Today, lithium-ion batteries offer many advantages over other high-quality rechargeable battery technologies such as nickel-cadmium (Ni-Cd) or nickel-metal-hydride (Ni-MH), including one of the highest energy densities of any battery technology; the ability to deliver large amounts of current to high-power applications; and the fact that lithium-ion batteries do not require routine maintenance to maintain their battery life. As a result of these performance advantages, lithium-ion battery technology dominates the battery market.

Due to the significant importance of lithium-ion battery technology, the 2019 Nobel Prize in Chemistry was awarded to John B. Goodenough, M. Stanley Whittingham, and Akira Yoshino for their contribution to the development of lithium-ion batteries. In Yoshino-san’s Nobel acceptance speech, he stated that the “Lithium-ion battery is expected to create the sustainable society […]” Indeed, the rapid adoption of lithium-ion battery technology in the automotive and consumer electronics markets in recent years seems to support Yoshino-san’s vision. At the same time, the Nobel Laureates were very clear in their acceptance speeches that lithium-ion battery technology still requires more research in the areas of – cost, energy, power output, cycle-life, safety, and environmental impact – if the technology is to achieve its full potential. This document seeks to illustrate how Waters | TA Instruments can support R&D in new battery material and technology development by providing solutions for analytical characterization and testing.

Waters | TA Instruments understands the material characterization needs of lithium-ion battery developers and offers thermal analysis, microcalorimetry, rheology, liquid chromatography and mass spectrometry solutions to help battery researchers, formulators, and production specialists develop better performing and safer batteries.
AN OVERVIEW OF LITHIUM-ION BATTERY TECHNOLOGY AND MATERIAL CHARACTERIZATION

A lithium-ion battery is comprised of four main components – cathode, anode, separator, and electrolyte. In a working battery, lithium ions flow from the anode to the cathode during discharge. The lithium ions flow in the reverse direction during recharging. Each individual battery cell outputs only a limited amount of energy and is often combined with other cells to form battery packs. Battery packs can in turn be combined to form battery modules for energy storage applications that require higher amounts of energy output such as electric vehicles and grid storage. The materials comprising the cathode, anode, separator, and electrolyte together help define a battery’s run time, safety, cycle life, power, energy density, and costs. Hence, an analytical characterization of the materials comprising the main battery components for thermal, rheological, and molecular properties can lead to better performing and safer batteries. Tables 1 and 2 illustrate some of the areas where Waters | TA Instruments analytical solutions can provide insight into the battery components and materials.

![Schematic of a lithium-ion battery.](image)

**Table 1.** A mapping of where materials characterization techniques can provide insights to the four main components, battery cell, and application areas of a lithium-ion battery.

<table>
<thead>
<tr>
<th>Technique</th>
<th>Anode/Cathode</th>
<th>Separator</th>
<th>Electrolyte</th>
<th>Raw Materials</th>
<th>Battery Cell</th>
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<td>TGA</td>
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<td>Rheology</td>
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**Table 2.** Parameter insights provided by material characterization techniques of the four main components and the whole cell.

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**Figure 1.** Schematic of a lithium-ion battery. The four main parts of a lithium-ion battery are the cathode, anode, electrolyte, and separator.
Battery thermal analysis improves lifetime, performance, and safety

A lithium-ion battery operates in an environment that constantly changes with regards to temperature. Some temperature changes are inherent to the electrochemical reactions occurring inside the battery while others arise from the battery’s ambient working environment. Hence, temperature influences many factors important to the performance of a lithium-ion battery such as battery lifetime, energy density, power output, and safety. Due to the critical nature of understanding how battery materials respond to heat, the analytical suite of thermal analysis techniques play an important role in battery development. The following thermal analysis techniques provide key insights into lithium-ion battery material development:

**Thermogravimetric Analysis (TGA):**

Programmatically heats a sample material while measuring the material's mass change with a highly sensitive analytical balance. As a material is heated, cooled, or held constant, the mass of the sample may change. Loss of mass indicates possible decomposition or vaporization, while a gain in mass indicates possible oxidation, sorption or that the material is reacting with its gaseous surroundings. Thermal and oxidative stability of materials are critical properties examined by TGA. In battery research, TGA can provide insights into the temperatures at which battery materials start to degrade so that proper material selection can be achieved resulting in high performing and long-lasting batteries.

**Thermogravimetric Analysis and Evolved Gas Analysis (TGA-EGA):**

The connection, or hyphenation, of spectrometers to TGA units is common. They allow for the chemical analysis of gases that evolve from samples during TGA experiments. The most popular hyphenated spectrometers are mass spectrometers (MS), Fourier-transform infrared spectrometers (FTIR), and gas chromatograph-mass spectrometers (GC-MS). In battery research, understanding what, if any, chemical components are released during production or use can aid in better material selection, design, or additive modification. Impurity detections and failure analyses (e.g., thermal runaway) also benefit from off-gas analysis. As an example, detection of oxygen is quite common in battery research and a mass spectrometer is required for this.
Differential Scanning Calorimetry (DSC):  
DSC measures the heat absorbed or released when a sample material is heated, cooled, or held isothermal. The heat flow is determined by comparing the heat flow difference between a sample material and a reference. DSC provides insights into the battery material’s heat capacity, melting point and phase transitions such as crystallization behavior or glass transition. This can aid in quality control such as confirming whether a polyethylene batch is similar to the previous batch and detecting critical phase transitions that may influence battery operation.

Simultaneous DSC-TGA (SDT):  
Combines the techniques of DSC-TGA together so that battery researchers can measure heat flow and weight change data with a single instrument. This combination allows battery researchers to understand battery phase transitions such as melting points, crystallization and glass transition, and material thermal stability. Simultaneous instruments can also be hyphenated with off-gas spectrometers.
Thermomechanical Analyzer (TMA):
A TMA measures expansion or contraction of a material under temperature ramping or isothermal conditions using a variety of sample probes. It is associated with the measurement of softening points, glass transitions, and coefficient of thermal expansion (CTE). One of the applications of TMA within battery research is to study the barrier material’s dimensional stability.

Dynamic Mechanical Analyzer (DMA):
A Dynamic Mechanical Analyzer (DMA) measures the mechanical properties of solids as a function of temperature, time, and/or humidity. These measurements are routinely used to characterize thermal events such as the glass transition, crystallization, curing, and aging, especially in polymeric materials. The measurements can then be used by engineers and scientists to develop materials with the right balance of strength, flexibility, and durability when designing lightweight batteries with improved safety and longer cycle life over a wide range of operating conditions.

Laser/Light Flash Analysis:
The transient measurement technique of Laser/Light Flash provides the material properties of thermal diffusivity and thermal conductivity or characteristics of the heat transport within a material. Heat within a battery cell is generated while in use, during charging, and potentially during storage. The proper management of this heat will allow the battery components to remain within an acceptable operating temperature range that reduces the risk of thermal runaway and optimizes cell efficiency and product lifetime.

Rheometer:
Rheology is the study of the flow and deformation of materials. A rheometer is a precision instrument that applies deformations in a pre-programmed manner and measures the material’s response in terms of stress and modulus. The rheometers used in the production of electrodes are a mix of solid particles, binder, and solvents undergoing a range of deformations at different stages of the manufacturing process such as storage, mixing, coating, and drying. These slurries therefore need to have a specific rheological profile to form uniform, defect-free coatings required for production of consistent, high-quality electrodes with high batch-to-batch reproducibility and low scrap rates.

Isothermal Microcalorimetry (IMC):
Isothermal Microcalorimetry (IMC) is a non-specific and non-destructive technique for measuring the smallest reactions in a material during a physicochemical process. It is done by measuring the heat flow from the sample at a constant temperature. In battery research, isothermal calorimetry of lithium-ion cells covers three main areas of interest: the first is the thermal output of a cell from the point of view of heat management; the second is the understanding of structural evolutions in active materials as evidenced by entropy changes; the third is the isolation of heat from parasitic reactions to rank the performance of cells. Evaluations of pouch, coin, pacemaker, cell phone, and cylindrical batteries can be conducted under passive storage conditions or in tandem with a battery cycler.

AN OVERVIEW OF OUR BATTERY MATERIAL CHARACTERIZATION SOLUTIONS
High-resolution mass spectrometry (HRMS) and liquid chromatography (LC):

The intrinsic performance and safety of lithium-ion batteries depend on an analytical understanding of the electrolyte, additive raw materials and their chemical degradation. While additives make up approximately only 5% by volume or weight of the electrolyte solution, they play a key role in facilitating the formation of a solid-electrolyte interface, reducing gas generation, improving long-term cycling, enhancing thermal stability and improving battery safety. Liquid and gas chromatography (LC and GC) combined with mass spectrometry provide key insights by characterizing electrolyte raw materials and chemical degradation. Analyzing these chemical components can be challenging with only a GC or LC technique since some molecules are volatile while others are non-volatile as well as polar and non-polar. Often GC and LC are used as complementary separation technologies to capture a wide range of compound classes with various volatilities and polarities. High-resolution mass spectrometry (HRMS) hyphenated to Ultra Performance Liquid Chromatography (UPLC) and GC separation technologies deliver molecular insight over a broad volatile and non-volatile chemical space to minimize lose of critical information.

High-Resolution Mass Spectrometry (HRMS):

A mass spectrometry technique that facilitates structural elucidation of molecular species by delivering high-resolution accurate mass measurements of both the molecular and fragment ions (i.e., a molecular fingerprint). In HRMS, the accurate mass measurements are used to determine the elemental composition of an unknown chemical which can then be used to search structural databases (such as Chembank, PubChem etc.) to aid structural elucidation and ultimately the chemical annotation of a compound. For example, an elemental composition of C₃H₃FO₃ along with molecular fragmentation would return fluoroethyl carbonate (FEC) in a database search. In battery applications, the fine chemicals can be either volatile or non-volatile, so HRMS is hyphenated to a GC separation system for non-volatile chemical components and GC separation system for volatile organic components.

Ultra-Performance Liquid Chromatography (UPLC):

A liquid chromatographic technique for separating mixtures of non-volatile chemical components of a sample so that they are easier to detect by mass spectrometry and UV-detection. The UPLC mode of liquid chromatography exhibits increased resolution, speed and sensitivity over traditional high-performance liquid chromatography (HPLC). Separating battery mixtures with chromatographic aids battery researchers by simplifying the complex mixture of electrolytes, additives, and degradation products so that chemical identification and quantitation are easier to achieve.

Atmospheric Pressure Gas Chromatography (APGC):

A gas chromatographic technique for separating mixtures of gas-phase chemical components so that they are easier to detect by mass spectrometry. Traditional GC-MS utilizes electron ionization (EI) in a vacuum environment to ionize the chemical components prior to the analysis by mass spectrometry, but this technique leads to excessive fragmentation of the molecular ion of chemicals analyzed, making the identification difficult. Instead, the APGC is a soft chemical ionization performed at atmospheric pressure that utilizes a gentle ionization regime and enables almost intact molecular ion detection allowing for both structural elucidation and chemical quantification of compounds separated by GC capillary column. Finally, the gas chromatography aids battery researchers by simplifying the complex mixture of electrolytes, additives, and degradation products so that chemical identification and quantitation are easier to achieve.
**BATTERY MATERIAL CHARACTERIZATION APPLICATION EXAMPLES**

**TGA thermal stability and compositional amount of anode materials**

Electrodes require binders and additives to ensure proper adhesion to the metal collector. For the anode electrode, carboxymethyl cellulose (CMC) is a common binder and butadiene-rubber (SBR) is a common additive that provides flexibility. TGA measures the thermal degradation temperature and composition of CMC and SBR in graphite anode materials. The high sensitivity of TGA ensures accurate measurement of each component in the electrode. For this test, the sample was loaded directly on a TGA platinum pan without any sample preparation.

**SDT of Lithium Carbonate (Li$_2$CO$_3$) cathode precursor material**

Lithium Carbonate (Li$_2$CO$_3$) is often used as a precursor for cathode materials in lithium-ion batteries. High temperature sintering processes are required, and the sintering temperature will impact the lithium salt residue, thus affecting the battery efficiency and cycle performance. Figure 2. TGA curve of graphite anode in a TA Instruments Discovery TGA. The sample was heated to 1000 ˚C at 10 ˚C/min. The weight signal indicates a small amount of weight loss during melting which has been seen by other researchers as an evolved gas during the heating process. A residue of 1.289 mg was measured by the Discovery SDT 650. Evolved gas analysis can be used to identify off-gases with direct measurement during heating of the battery. The sample was loaded directly on SDT aluminum pan without any additional sample preparation.

**DSC thermal stability and heat of reaction of electrolyte during thermal runaway**

The thermal stability of lithium-ion batteries is a major concern in battery usage. Heating of the battery components due to either thermal or mechanical failure can cause thermal runaway, which is two important parameters for battery safety evaluation. Figure 3. DSC curve of electrolyte measuring the onset, peak temperature, and heat of reaction during thermal runaway. The onset temperature indicated the temperature runaway start, and the heat of reaction indicated the total heat release during the exothermic decomposition, or equivalently thermal runaway reaction. The significant difference measured in the thermal expansion coefficient in the two directions indicates an orientation effect in the separator. The X-direction showed thermal shrinkage onset at 136 ˚C while no thermal shrinkage was observed in the Y-direction. The TMA 450 measured the thermal expansion of the separator and identified an orientation effect in the separator. The TMA 450 measured the thermal expansion and melt phase transition of Li$_2$CO$_3$. Mass spectrometer data is also overlayed in the graph and indicates that CO$_2$ (m/z 44) is released during the heating process. A residue of 0.05 mg was measured by the Discovery SDT 650. Evolved gas analysis can be used to identify off-gases with direct measurement during heating of the battery. The sample was loaded directly on SDT aluminum pan without any additional sample preparation.

**Conclusion**

The TGA measured the thermal stability and compositional amount of anode materials, and TGA can also provide quality control of the materials to ensure the same amount of active material, binder and additive are in each batch of the electrode. TGA can also provide quality control of the materials to ensure the accurate measurement of each component in the electrode. For this test, the sample was loaded directly on a TGA platinum pan without any sample preparation. For the anode electrode, carboxymethyl cellulose (CMC) is a common binder and butadiene-rubber (SBR) is a common additive that provides flexibility. TGA measures the thermal degradation temperature and composition of CMC and SBR in graphite anode materials. The high sensitivity of TGA ensures accurate measurement of each component in the electrode. For this test, the sample was loaded directly on a TGA platinum pan without any sample preparation. For the anode electrode, carboxymethyl cellulose (CMC) is a common binder and butadiene-rubber (SBR) is a common additive that provides flexibility. TGA measures the thermal degradation temperature and composition of CMC and SBR in graphite anode materials. The high sensitivity of TGA ensures accurate measurement of each component in the electrode. For this test, the sample was loaded directly on a TGA platinum pan without any sample preparation. For the anode electrode, carboxymethyl cellulose (CMC) is a common binder and butadiene-rubber (SBR) is a common additive that provides flexibility. TGA measures the thermal degradation temperature and composition of CMC and SBR in graphite anode materials. The high sensitivity of TGA ensures accurate measurement of each component in the electrode. For this test, the sample was loaded directly on a TGA platinum pan without any sample preparation. For the anode electrode, carboxymethyl cellulose (CMC) is a common binder and butadiene-rubber (SBR) is a common additive that provides flexibility. TGA measures the thermal degradation temperature and composition of CMC and SBR in graphite anode materials. The high sensitivity of TGA ensures accurate measurement of each component in the electrode. For this test, the sample was loaded directly on a TGA platinum pan without any sample preparation.
DMA mechanical properties of separator

The mechanical properties of any polymer are closely related to its structure and morphology. These properties can be easily measured using DMA to examine the behavior of DMA solutions as a function of time, temperature, and/or humidity to provide valuable information about the polymer structure and gain critical information of material behavior under a wide range of conditions. The key material properties obtained from a DMA experiment are shown in the table below:

<table>
<thead>
<tr>
<th>Definition</th>
<th>Temperature (˚C)</th>
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<tbody>
<tr>
<td>Viscous or viscous modulus (E”)</td>
<td>Tg: 9.41 ˚C</td>
</tr>
<tr>
<td>Elastic or storage modulus (E’)</td>
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<td>δ</td>
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Higher E” is desirable for higher toughness under load and deformation. Higher damping is desirable for decreasing unwanted vibrations and over time. Higher E’ indicates better mechanical integrity under load and deformation. Increasing toughness.

The data in Figure 6 also shows that E’ (blue curve) is reduced by almost 90% as the material undergoes a glass transition. Note the glass transition, Tg, at 9.41˚C (green curve) and crystallization, curing, aging.

The data in Figure 6 also shows that E” (blue curve) as the material undergoes a glass transition. The stored energy loss modulus (E”) can be measured with various techniques, but DMA is by far the most sensitive. As shown in Figure 6, one of the ways to measure Tg using the DMA is using the peak of the tan δ curve (green). The measurement can serve as an important characterization metric for the separator both in R&D labs for accelerated material development and in QC labs to ensure batch-to-batch repeatability.

The data in Figure 6 also shows that E” (blue curve) is reduced by almost 90% as the material undergoes a glass transition. Initially, under low shear rates which simulate storage conditions, the viscosity is high to prevent settling and reduce the energy of mixing prior to coating. The low torque sensitivity of the DHR allows for the measurement to be performed in less than 20 minutes with a direct readout of the viscosity. Initially, under low shear rates which simulate storage conditions, the viscosity is high to prevent settling and reduce the energy of mixing prior to coating. The low torque sensitivity of the DHR allows for the measurement to be performed in less than 20 minutes with a direct readout of the viscosity. Initially, under low shear rates which simulate storage conditions, the viscosity is high to prevent settling and reduce the energy of mixing prior to coating. The low sensitivity of the DHR enables accurate, repeatable measurements in this low shear rate region providing greater confidence in the data.

The slurry rheology continues to play a critical role in the film formation stage (a low shear rate process) where the rate of viscosity decrease (known as thixotropy) ensures the levelling of the coatings. It is especially critical when electrodes with high cost are desired.

Electrode slurries are complex, non-Newtonian fluids that are a mixture of solid particles and polymeric binder in a solvent. They are subjected to a wide range of changing shear deformation rates at different stages of the electrode manufacturing process. The ideal slurry has a low viscosity at mixing and settling, but a high enough viscosity for good levelling during drying and to minimize particle settling and agglomeration during storage (low shear rates).

The data in Figure 8 shows the viscosity of an anode slurry under different shear rates on a TA Instruments Discovery Hybrid Rheometer (DHR). The sample was mixed before loading on the rheometer. The measurements were performed from 0.01 to 1000 s⁻¹ at 20 °C using a 40mm parallel plate with solvent trap.

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Electrolyte Waters Xevo G2-XS QTof Electrolyte solution samples were extracted with dimethyl carbonate from ACQUITY I-Class UPLC 5 Column: HSS T3 2.1 x 100, 1.7 µm

The determination of the heat from parasitic reactions is useful for ranking cell chemistries and electrolyte chemistries. This approach has been successful in ranking electrolyte chemistries for various cathode active materials (LCO, NiCoAl, LFP) and anode active materials (natural and artificial graphite, silicon).

Table 4. UPLC-HRMS experimental conditions

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<th>Sample preparation</th>
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Conclusion: Determination of parasitic reactions for a battery cell can be calculated from the extracted voltage and heat flow data. This approach has been successfully used to allow comparison of different cell chemistries and sizes. The TAM IV microcalorimeter is used to rapidly determine the parasitic power of an 18650 Li-cell.

BATTERY MATERIAL CHARACTERIZATION APPLICATION EXAMPLES

Thermal conductivity and thermal diffusivity of copper thin-films

Thermal conductivity is a very important parameter needed for designing a reliable battery thermal management system (TMS). The flash method measures thermal diffusivity that is related to thermal conductivity by specific heat and density. Flash diffusivity provides the most versatile, effective, accurate thermal diffusivity and conductivity measurements.

Copper thin-film is used as anode collector and its in-plane thermal diffusivity is measured with DSF 200+. A copper thin-film sample with 25 µm thickness was loaded on in-plane fixture and measured at 25˚C. The measurement and analysis are simple, direct, and reliable.

Conclusion: Agreement. The thermal diffusivity was determined as 1.17 cm\(^2\)/s.

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A COMPREHENSIVE THERMAL ANALYSIS AND RHEOLOGY SOFTWARE SOLUTION

Our instruments’ robust software package uses innovative technology for instrument control, data collection, data analysis, and reporting for thermal analysis and rheology. The intuitive user interface allows you to seamlessly program experiments and move easily between processing experiments, viewing and analyzing data.

Thoughtful features such as automated calibration routines, multiple calibration sets, real-time test method editing, and inter-laboratory data and test method sharing provide unmatched flexibility, while one-click analysis and custom reporting streamline productivity to new heights.

**Reports:** Easily prepare your data for presentation using the TRIOS Report feature. Drag and drop elements, corresponding to various data file components, can be inserted into a familiar word processing workspace, allowing for clear and concise formatting of all experimental parameters. Once created, simply apply the complete format to any data file, automating analysis and relevant details to communicate your findings.

**Analysis:** Save routine analyses to the Analysis library, then apply them with a single click, streamlining your workflow and increasing productivity. Share these analyses by saving an Analysis Template to ensure the exact parameters are used in collaborative environments.

**Control Charts:** Conveniently store and track analysis results from your data archive in a single file. Detect trends in your data and flag unacceptable results so you can keep your process moving without interruption.

**User Models and Variables:** Define your own analysis model to precisely fit your experimental data within TRIOS. The intuitive interface gives you full control over mathematical functions and adjustable fitting parameters, while TRIOS solves the equation. Save your custom models to quickly apply them to future experiments. Every TRIOS data file contains a standard set of variables based on instrument type. If the required variable isn’t available, quickly create your own using the User Variable feature to obtain the results you require.

**Our experience with over 10,000 installations has shown that when users are well trained, systems receive routine preventive maintenance, and problems and concerns are addressed promptly, the result is improved instrument performance, increased uptime, and reduced cost of ownership.**

The Lifetime Support Plan (LSP) has been designed to make this comprehensive support available for a modest, easily budgeted annual subscription fee, that is predicated on the benefits of uninterrupted coverage initiated at installation and continues for the lifetime of the instrument. Benefits of this plan include:

- Optimized instrument performance
- Well trained users
- Maximized uptime
- Protection from unexpected repair costs
- Easily budgeted operating expenses
- Reduced cost of ownership

This support product is available only for new instruments at the time of purchase, or prior to the end of the warranty period. Once initiated, the LSP is renewable annually to provide uninterrupted coverage for the life of the instrument.

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