



**Malvern  
Panalytical**  
a spectris company

# Battery research and quality control solutions

Benefit from physical, chemical and structural insight



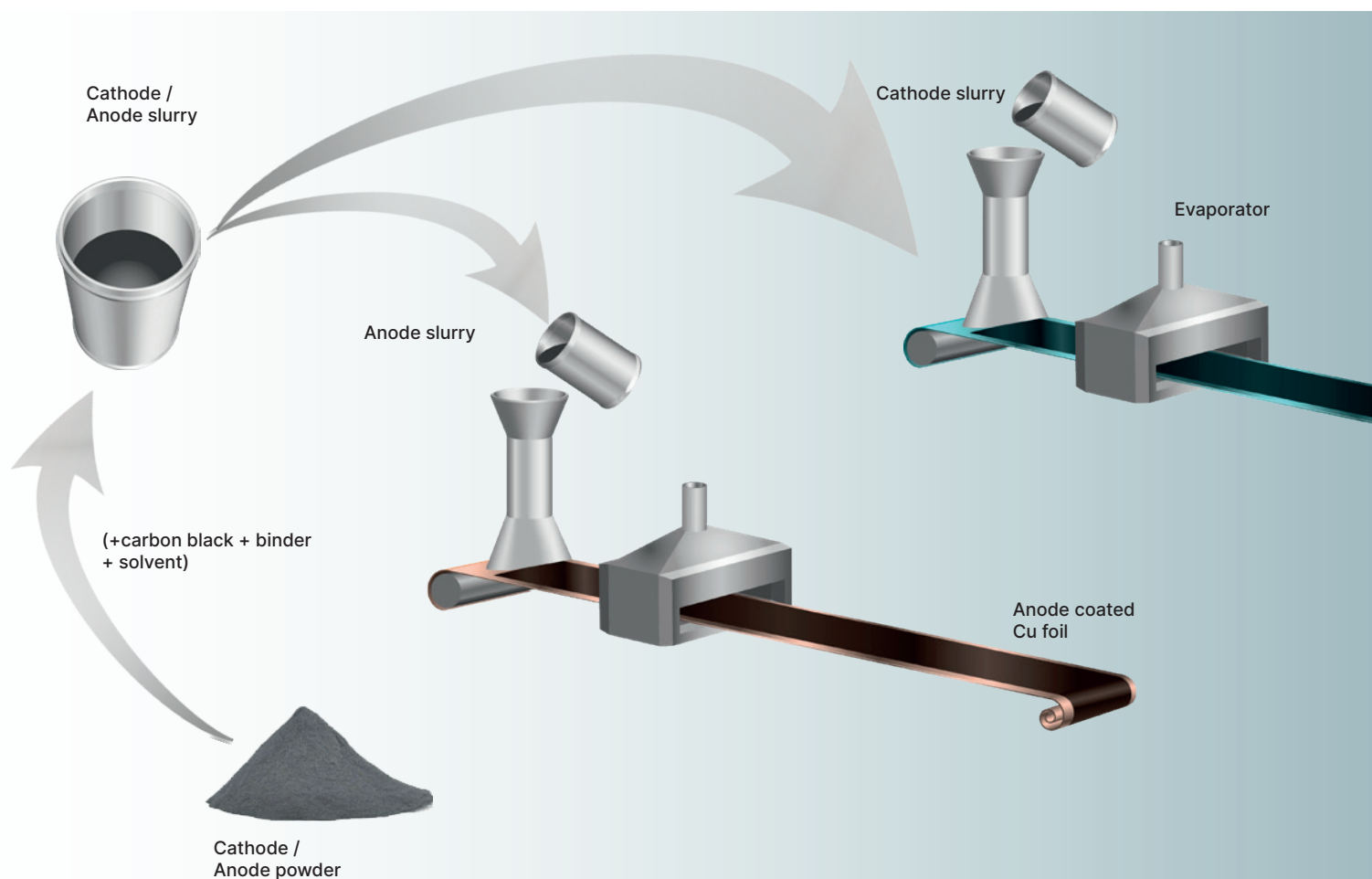
# Discover new horizons in battery quality

## Empower your research and production with advanced analytical solutions

The age of the battery is well underway. Lithium-ion batteries, for example, have already revolutionized our day-to-day lives – from smart mobile devices to pollution-free electric cars and intelligent power management solutions. And, looking ahead, batteries also have the potential to provide an economical solution for mass energy storage and complement renewable energy resources for power grid applications.

Despite these successes, gaps in battery technology remain, both in terms of safety and performance. What's more, for their mass-scale adoption in applications like electric vehicles, large cost reductions will be needed. Indeed, with regulators becoming more stringent and consumers more demanding, these core issues are driving not only research into new battery materials, but also improvements in production efficiency to minimize production costs. When success is defined by such fine margins, today's manufacturers must be able to ensure total quality and performance – every time.

At Malvern Panalytical, we leverage over six decades of experience in the design, manufacture, and supply of analytical instruments to offer a complete range of laboratory and online analytical solutions. Whether you are a battery component manufacturer looking for greater process efficiency and better quality control, or a researcher trying to determine the performance parameters of newly emerging battery materials, our solutions will offer you the new levels of insight and control needed to power the production of superior-quality batteries.



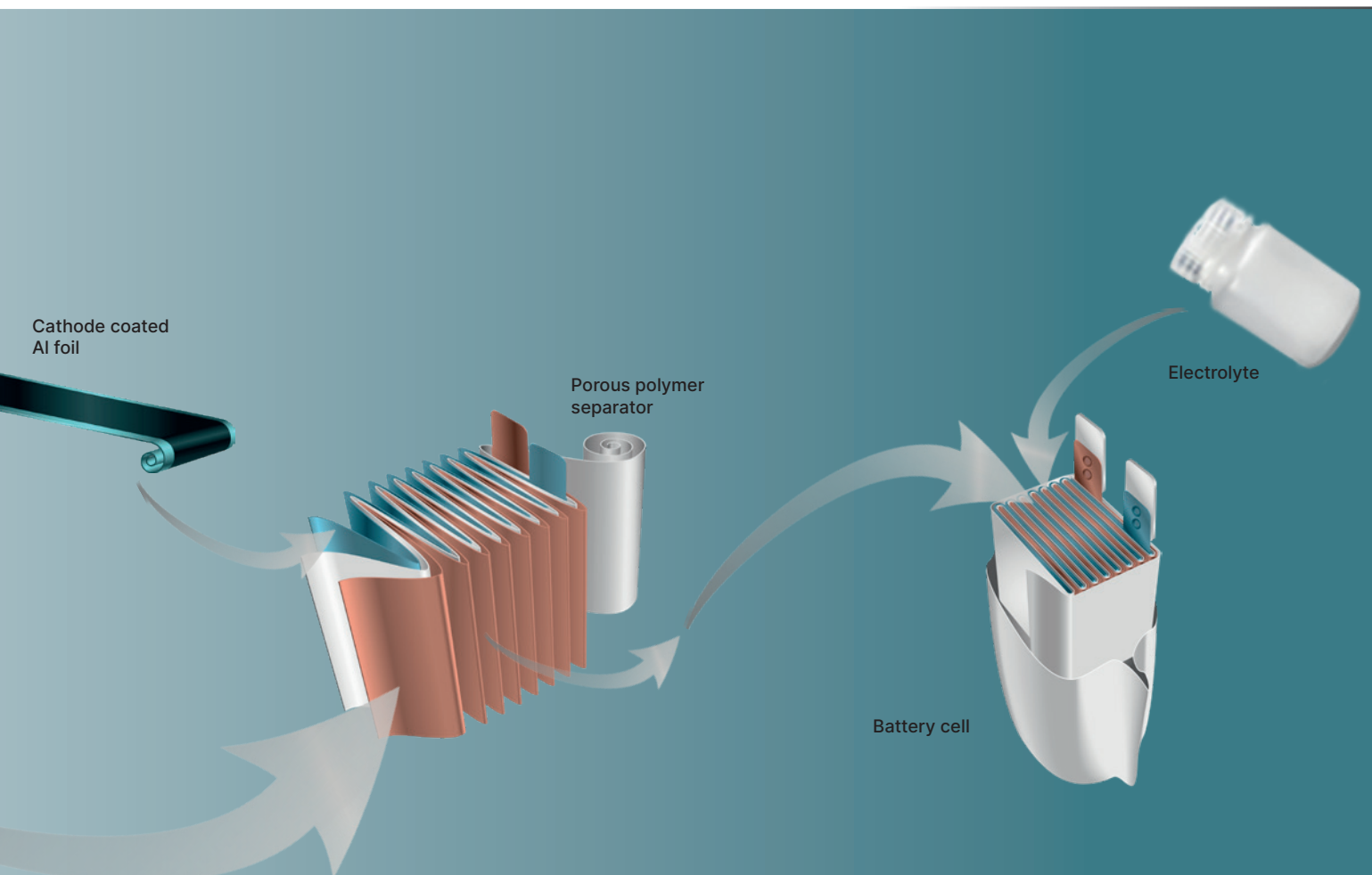
# Monitor and optimize at every stage

Battery component manufacturers must not only deliver consistent overall quality – they must deliver it throughout the manufacturing process. The continuity of the manufacturing process means errors or impurities at an early stage will accumulate, resulting in much larger consequences further down the production line. Quality needs to be monitored at every stage – from raw materials through to cell assembly – to maintain production efficiency and minimize waste. Likewise, research into new battery materials must ascertain all the critical parameters that could affect battery performance throughout the entire manufacturing process.

At Malvern Panalytical, we've developed a range of research and quality control solutions to help manufacturers monitor and optimize every part of the battery manufacturing process. The unique set of physical, chemical and structural analysis possibilities that our technologies offer can be utilized across various stages of battery component production. From improving electrode material quality to accelerating the successful development of new high-performance battery materials, we've got you covered – enabling the development and production of the highest-performing batteries.

Table 1 Battery materials and analytical solutions along the battery value chain

| Battery materials                         | Critical Parameters                                                      | Analytical techniques                                                             | Product and solutions                                                           |
|-------------------------------------------|--------------------------------------------------------------------------|-----------------------------------------------------------------------------------|---------------------------------------------------------------------------------|
| Cathode precursor and electrode materials | Particle size<br>Particle shape<br>Chemical composition<br>Crystal phase | Laser diffraction<br>Automated Imaging<br>X-ray fluorescence<br>X-ray diffraction | Mastersizer 3000<br>Morphologi 4<br>Epsilon 4 / Zetium XRF<br>Aeris compact XRD |
| Battery slurry                            | Particle size<br>Particle shape                                          | Laser diffraction<br>Automated Imaging                                            | Mastersizer 3000<br>Morphologi 4                                                |
| Electrode coating                         | Coat weight<br>Coating thickness                                         | Photon sensor<br>Micro caliper thickness sensor                                   | Photon and Micro caliper sensors from NDC technologies                          |
| Battery cell                              | Degradation mechanism during cycling                                     | <i>In operando</i> XRD                                                            | Empyrean XRD                                                                    |



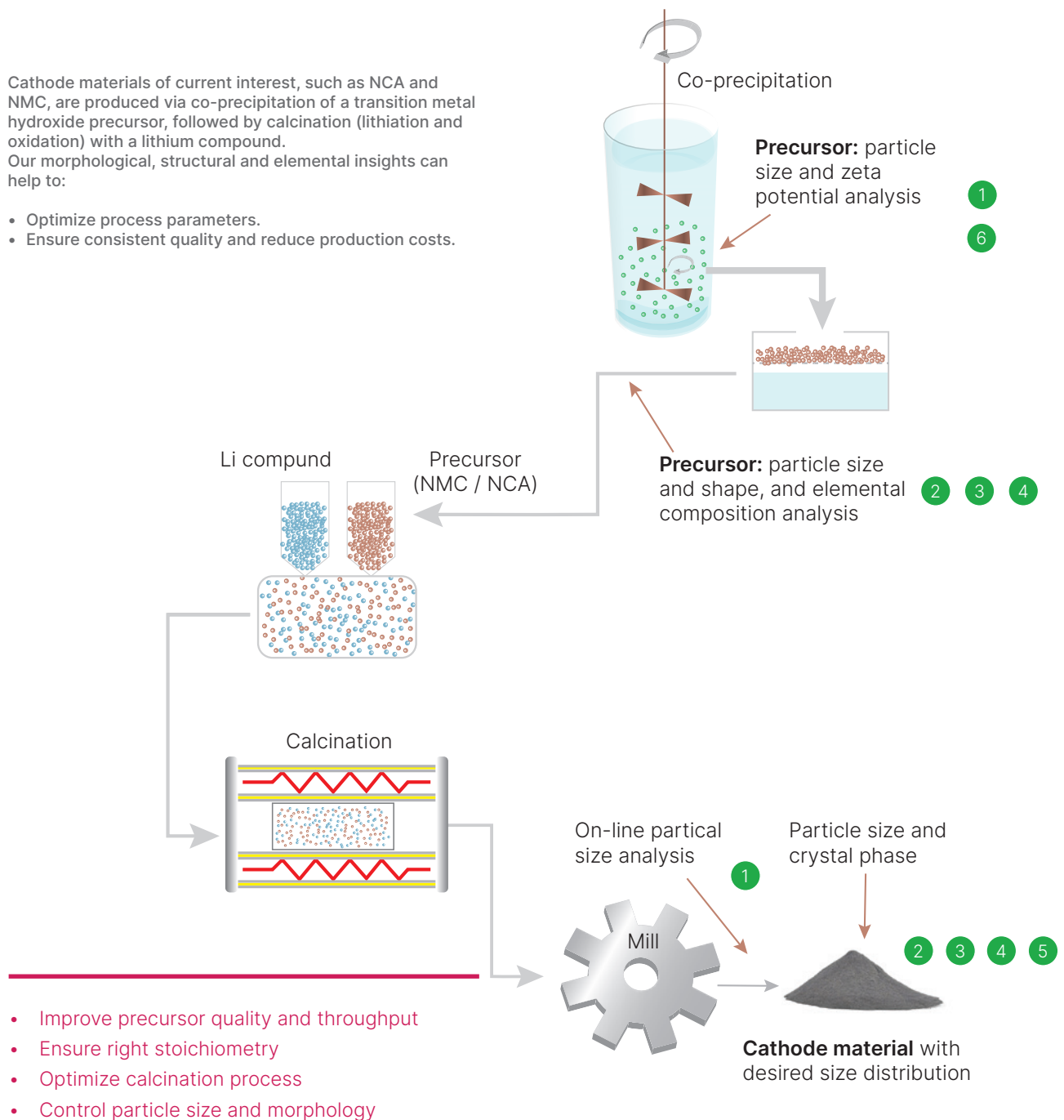
# Optimize cathode materials

Cathode material production is just one example of how our solutions enable quality at every stage of the manufacturing process. As one of the first stages in battery production, quality control is especially important to cathode manufacturing – and battery manufacturers must implement it all while minimizing costs.

Our solutions can be used as cathode characterization tools at several stages of the cathode production process, from co-precipitation and precursor quality control, down to optimizing calcination and the final material. By providing manufacturers with morphological, structural, and elemental insights, these solutions enable them to optimize process parameters accordingly to ensure the highest cathode quality.

Cathode materials of current interest, such as NCA and NMC, are produced via co-precipitation of a transition metal hydroxide precursor, followed by calcination (lithiation and oxidation) with a lithium compound. Our morphological, structural and elemental insights can help to:

- Optimize process parameters.
- Ensure consistent quality and reduce production costs.

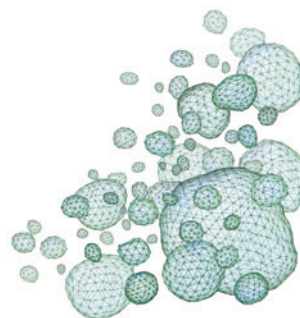


# Characterization tools

## 1 Particle size

Consistent cathode-material particle size starts with the precursor – with our **Insittec** online tool, manufacturers can analyze particle size in real time at the precursor or milling stage.

- 2 Fine control over particle size distribution plays an important role in the quality of the electrode coating. With the **Mastersizer 3000**, you can analyze particle size with accuracy and ease.



## 3 Particle shape

Particle shape influences critical parameters like electrode slurry rheology and packing density. Analyze particle size and shape at the precursor stage with **Morphologi 4** imaging – automatic analysis of thousands of particles with high statistical accuracy.



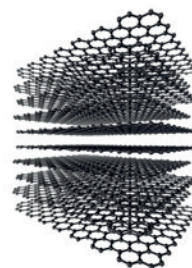
## 4 Elemental composition

Combined with fusion sample preparation recipes, our **Epsilon 4** benchtop X-ray fluorescence (XRF) spectrometer or high-end **Zetium** spectrometer can simplify the chemical composition and elemental impurity analysis of electrode materials.

|    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |   |    |    |
|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|---|----|----|
| H  |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    | He |    |   |    |    |
| Li | Be |    |    |    |    |    |    |    |    |    |    |    |    |    |    | B  | C  | N  | O | F  | Ne |
| Na | Mg |    |    |    |    |    |    |    |    |    |    |    |    |    |    | Al | Si | P  | S | Cl | Ar |
| K  | Ca | Sc | Ti | V  | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |    |   |    |    |
| Rb | Sr | Y  | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I  | Xe |    |   |    |    |
| Cs | Ba | L  | Hf | Ta | W  | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | Rn |    |   |    |    |
| Fr | Ra | A  |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |   |    |    |
|    |    |    | L  | La | Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |   |    |    |
|    |    |    | A  | Ac | Th | Pa | U  | Np | Pu | Am | Cm | Bk | Cf | Es | Fm | Md | No | Lr |   |    |    |

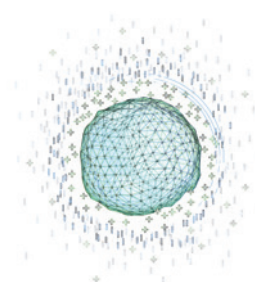
## 5 Crystalline phase

Crystalline phase and crystallite size are the key attributes defining the quality of electrode materials. Our **Aeris** compact X-ray diffractometer sets the industry benchmark for crystalline phase composition analysis, which can be used to optimize the calcination process.



## 6 Zeta potential

Zeta potential can be used to optimize process parameters like pH and concentration to control slurry stability, agglomeration, and sedimentation behavior. Our **Zetasizer** can analyze zeta potential of a dispersion and also the size and agglomerate state of nanosized materials.





# Our research and quality control solutions

## Superior particle size distribution measurement

Delivering an optimal and consistent particle size distribution doesn't just add value to electrode materials, it's key to ensuring final product quality – enabling manufacturers to optimize; slurry viscosity and flow behavior; coating packing density and porosity; battery cell charge rate capacity and cycling durability.

To meet this need, Malvern Panalytical offers an industry-standard particle size measuring tool; the Mastersizer 3000. Based on laser diffraction, it offers industrial-level performance, delivering more reliable

and faster measurements than sieving, faster measurements than sedimentation, and statistically superior information compared to microscopy. The Mastersizer 3000 laser diffraction particle size analyzer delivers rapid, accurate particle size distributions for both wet and dry dispersions with the minimum of effort. Measuring over the nanometer to millimeter particle size ranges, it packs exceptional performance into the smallest of footprints, bringing operator-independent measurements that every user can rely on.

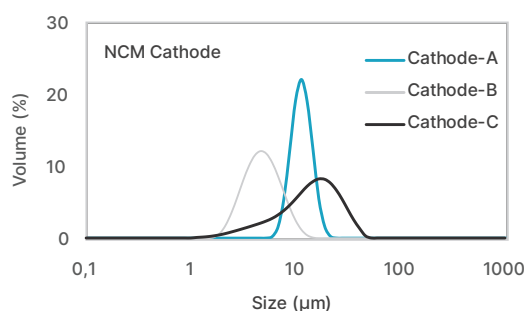


Figure 1 Particle size distribution of three batches of NCM811 cathode materials synthesized with different processing parameters.

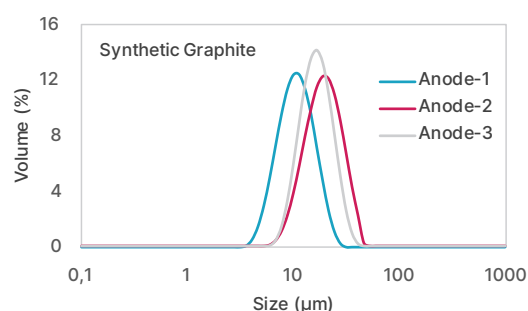


Figure 2 Particle size distribution of three batches of synthetic graphite synthesized with different heating conditions.



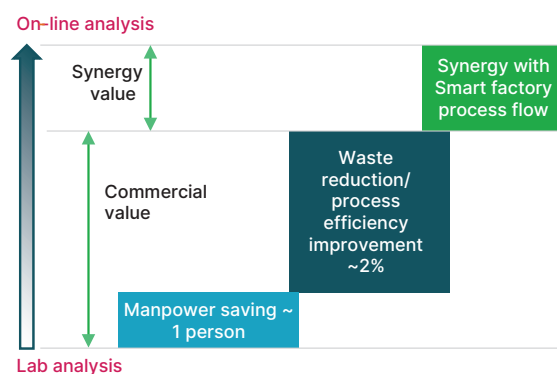


# Real-time process automation

## Optimizing process parameters

Manufacturers also need to monitor particle size as the production process continues. And, if you're manufacturing at industrial scale with limited human resources, finding an efficient way to analyze particle size in real time can be difficult. Usually, lab analysis will deliver feedback in about an hour, whereas for optimum quality control, a feedback loop of a few minutes is required.

Our on-line, automated Insitac particle size analyzer is ideal to meet this need in a production environment, delivering real-time analysis every few seconds using a feedback loop. It can be used to control precursor particle size evolution over time, or just after milling to control electrode particle size – enabling manufacturers to reduce waste and align their processes with smart factory manufacturing flows.



Typical value gain for a cathode manufacturing plant with Insitac online particle size analysis of precursor slurry, in comparison to lab analysis.

# In-line control of elemental composition for roll-to-roll electrode coating process

## Opening new process control possibilities

When it comes to electrode coatings, elemental consistency is important. Small changes in elemental composition may significantly affect the process efficiency and final battery performance. And, when the measurements are so critical, electrode manufacturers need fast, efficient ways to measure these variations. To address these challenges and deliver unparalleled process visibility and control, Malvern Panalytical offers an in-line elemental metrology system – the Epsilon Xline. Covering a wide range of elements from Na to U, this tool opens the new levels of control and precision that deliver outstanding process reliability and ultimately industry-leading, high-performance batteries.

### Epsilon Xline

By combining our advanced Epsilon 4 technology with in-line functionality, this tool offers real-time material monitoring and up-to-the-minute process control for both the ultrasonic spray coating and roll-to-roll coating processes. This regular analysis means material composition and loading are continually optimized, helping to minimize off-specification production and maximize the cost efficiency. In addition to precise and accurate process control, the Epsilon Xline is adaptable to a wide range of surfaces and coating materials, including but not limited to the commercially used LFP and NCM chemistries.



### Key benefits

- Non-destructive and suitable for in-line process control
- A range of scanning modes, to accommodate patch, continuous, and multi-lane coating processes
- Easy to integrate into the production process with standard communication protocols
- Able to measure all elements of interest
- Capable of measuring thin layers with relative standard deviations up to 5%, with a precision rate of over 95%
- Accommodates a range of roll widths

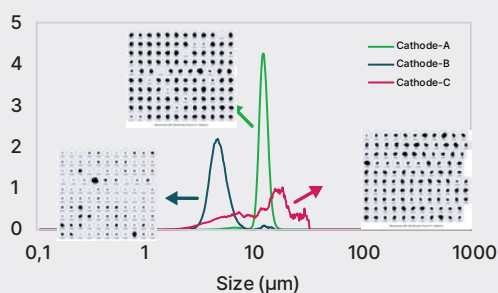


Figure 3

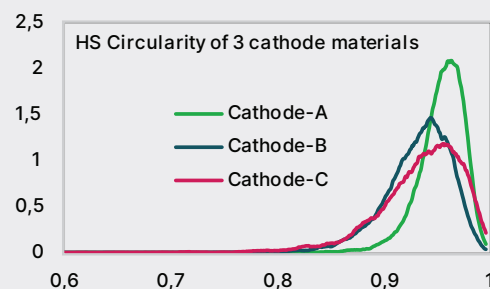


Figure 4

Example Cathode Materials Circular Equivalent (CE) size distribution of three NCM cathode materials, as obtained from Morphologi 4 (Figure 3). These are the same samples as those measured with the Mastersizer 3000 in Figure 1. Circularity for these samples is compared in Figure 4. Circularity index 1 corresponds to perfect spheres and smaller values to larger deviation from circularity. A narrow distribution in circularity means uniform shape particles, whereas broad distribution represents large variance in particle shapes. Cathode A has circular particles, whereas B and C are irregularly shaped with large shape variations.

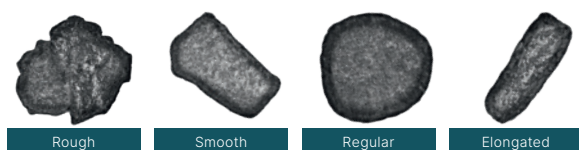


# Particle morphology

## Enabling better insight into electrode material morphology

The role of particle shape in battery electrode materials is usually ignored or underestimated. However, this may be the key to unlock full potential of a given battery material to be translated into the best performing battery. Particle shape affects slurry rheology as well as the

electrode coating in terms of packing density, porosity and uniformity. To achieve the highest level of battery performance, manufacturers must also understand and optimize the particle morphology.



### Do your particles have the right morphology?

To help manufacturers resolve these critical questions, we offer our powerful optical imaging tool, Morphologi 4. Equipped with fully automated image analysis, it enables manufacturers to measure parameters such as circularity, elongation/aspect ratio, Circular Equivalent (CE) diameter, and transparency.

With analysis based on 10,000 – 500,000 particles from 0.5  $\mu\text{m}$  onwards, manufacturers can gain reliable insight into the shape of the smallest particles with high statistical accuracy. What's more, the Morphologi 4-ID combines automated static imaging features of the Morphologi 4 with chemical identification of individual particles using Raman spectroscopy - enabling automated measurement of particle size, particle shape and chemical identity, on a single platform.

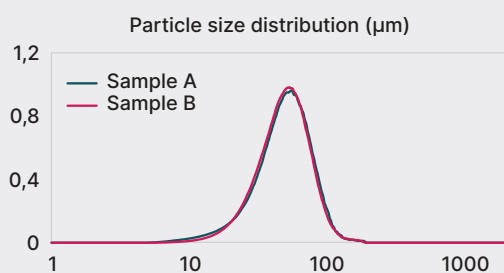


Figure 5

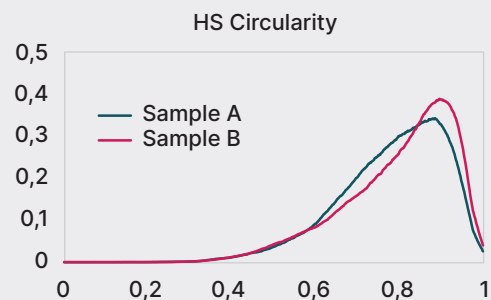


Figure 6

Example Graphite electrode material: size distribution of two samples as measured with Mastersizer 3000 (Figure 5) and circularity of the same samples (Figure 6) as measured with Morphologi 4. Though the samples have similar circular equivalent size distribution, sample A is more irregular in shape.

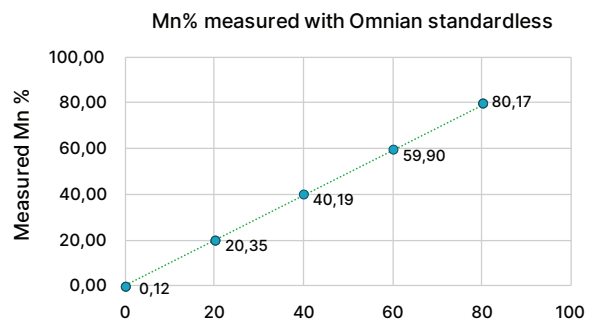
# Elemental composition

## Simplifying chemical composition and impurity analysis

Deviations in chemical composition or impurities in electrode materials can significantly affect final battery performance. For this reason, chemical composition and elemental impurity analysis are an integral part of the battery manufacturing process. However, the often-used inductively coupled plasma (ICP) analysis is not always the best tool for this. Requiring sample digestion and frequent calibration, ICP is inefficient and expensive for most elemental analysis needs.

To provide a simpler way to analyze elemental composition and detect impurities down to ppm level, we offer X-ray fluorescence (XRF) solutions that require no sample digestion or frequent calibration and are up to three times cheaper in terms of per sample analysis. In particular, chemical composition analysis of cathode materials at low-percentage elemental levels is more reliably measured with X-ray fluorescence.

Specifically, our **Epsilon 4** benchtop energy dispersive XRF spectrometer can accurately measure elemental composition in just a few minutes. When better light element sensitivity is desired, our **Zetium** wavelength dispersive XRF spectrometer is recommended. These solutions can help manufacturers optimize cost and use of human resources, while saving ICP for very low-level impurity detection.



Composition and impurities analysis on five samples of  $\text{LiFe}_x\text{Mn}_{1-x}\text{PO}_4$  measured on the Epsilon 4 is shown in the table below. Measured Mn composition shows a straight-line calibration with Target Mn composition in these samples.

| Sample | Target Material                               | Measured composition and impurities |       |       |       |       |       |       |       |       |       |       |       |       |       |
|--------|-----------------------------------------------|-------------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
|        |                                               | Mn %                                | Zr    | Al    | Ca    | Cl    | Co    | Cr    | Cu    | Ni    | S     | Si    | Ti    | Y     | Zn    |
| SMP1   | $\text{LiFe}_{0.2}\text{Mn}_{0.8}\text{PO}_4$ | 80.2                                | 0.11  | 0.011 | 0.014 | 0     | 0     | 0.017 | 0     | 0.026 | 0.008 | 0.022 | 0.009 | 0.006 | 0     |
| SMP2   | $\text{LiFe}_{0.4}\text{Mn}_{0.6}\text{PO}_4$ | 59.9                                | 0.19  | 0.012 | 0     | 0     | 0.001 | 0.024 | 0.018 | 0.013 | 0.004 | 0.02  | 0.01  | 0.009 | 0.006 |
| SMP3   | $\text{LiFe}_{0.6}\text{Mn}_{0.4}\text{PO}_4$ | 40.2                                | 0.117 | 0.041 | 0     | 0     | 0.003 | 0.034 | 0.011 | 0.023 | 0.006 | 0.024 | 0     | 0.007 | 0.005 |
| SMP4   | $\text{LiFe}_{0.8}\text{Mn}_{0.2}\text{PO}_4$ | 20.4                                | 0.161 | 0.008 | 0.014 | 0.037 | 0     | 0.042 | 0.009 | 0.015 | 0     | 0.02  | 0     | 0.006 | 0.006 |
| SMP5   | $\text{LiFePO}_4$                             | 0.1                                 | 0.155 | 0.015 | 0     | 0     | 0     | 0.055 | 0.009 | 0.024 | 0.007 | 0.024 | 0     | 0.009 | 0.004 |



# Sample preparation for XRF

## Ensuring more accurate analysis

Sample preparation is a major source of error in elemental composition analysis. All too often, preparation methods such as pressed pellets (in XRF) or acid digestion (in ICP) are affected by mineralogical or particle size effects in metal samples, compromising the accuracy of the results.

To meet the need for reliable sample preparation for XRF or ICP, Malvern Panalytical offers a high-performance fusion solution. Fusion involves dissolving a fully oxidized sample at high temperature in a suitable solvent (a flux) in a platinum, zirconium or graphite crucible. The melted mixture is agitated and poured into a mold to create a glass disk for XRF analysis. It can also be poured into a beaker to create a solution for Atomic Absorption Spectroscopy (AAS) or ICP analysis.

At Malvern Panalytical, you can count on our 40-plus years of expertise in designing and delivering fusion recipes for various kinds of complex samples. What's more, we offer two fusion instruments. Our robust LeNeo instrument is ideal for battery sample fusion. This automatic electric instrument can prepare glass disks for XRF analysis, as well as borate and peroxide solutions for AA and ICP analysis. With one fusion position, it delivers excellent ease of use, operator safety, and superior analytical performance in the laboratory – eliminating doubt and driving high-precision quality control. To meet high sample throughput, our TheOx advanced instrument, which has six fusion positions, can be used.



# Crystalline phase: XRD analysis

XRD can analyze crystalline phase related critical quality parameters of the battery electrode materials, like:

- **Phase composition:** determines if the reactants have fused to the desired and stable crystalline phase.
- **Crystallite Size:** plays an important role in Li ion migration (discharge rate capacity)
- **Cation mixing:** Degrades the battery energy density.
- **Degree of graphitization** in synthetic graphite
- **Orientation Index:** how the graphite particles are oriented in electrode coating

Use of XRD to optimize process parameters can eliminate material quality issues



Aeris, the compact X-ray diffractometer provides all information inherent to crystalline phases in just a few minutes.

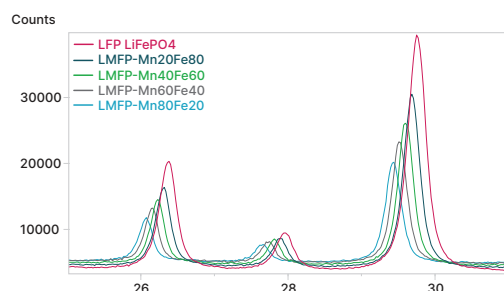


Figure 7

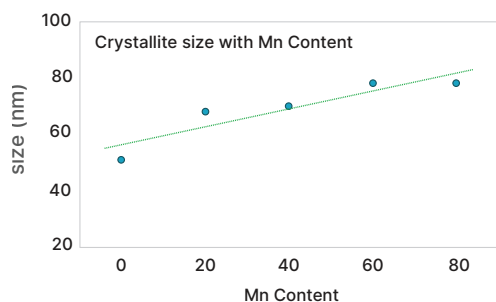


Figure 8

Example LMFP Cathode Materials with varying Mn content: section of XRD pattern (Fig. 7) showing peak shift to lower angles, meaning lattice expansion along c-axis with increasing Mn content. XRD also reveals that crystallite size increases with Mn content (Fig. 8) – higher Mn facilitates larger crystallite size.

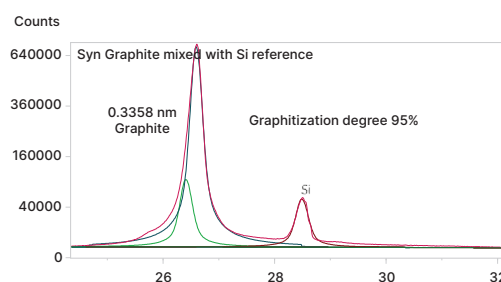
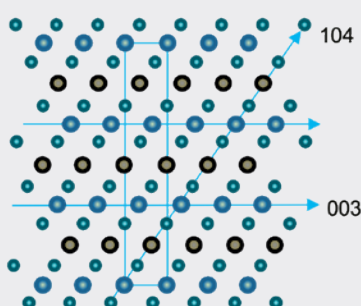


Figure 9

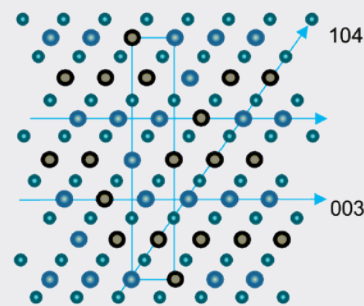
Example Synthetic graphite: Degree of graphitization and orientation index are important attributes of synthetic graphite, which is a commonly used anode material due to its superior consistency and purity compared to natural graphite. Aeris XRD can measure both the graphitization degree and orientation index. Fig. 9 shows measurement of the degree of graphitization in one such material.

## Cation Mixing

No cation Mixing



cation Mixed



● Ni/Co/Mn

● Li



# Zeta potential analysis

## Enabling slurry stability insight

In electrode slurry production, zeta potential (related to charge charge on particle) plays an important role in slurry stability. Low zeta potential can result in agglomeration and aggregation of particles – causing unstable particle dispersion in slurries and, in turn, compromising battery quality. So, being able to accurately monitor and optimize zeta potential isn't just useful to battery manufacturers – it's essential.



High zeta potential  
= stable dispersion



Low zeta potential  
= unstable dispersion /  
agglomeration



To meet this need, we offer the **Zetasizer**. Whether it's to understand agglomeration and sedimentation in precursor slurry, or to ensure electrode slurry stability, or to understand slurry wetting behavior on flat surfaces, this tool measures zeta potential with excellent accuracy, repeatability, and consistency. And, for high-concentration and highly conducting samples, its specialized cell and constant current mode have you covered – enabling manufacturers to optimize Ph and concentration for stable slurries to manufacture high quality electrodes.

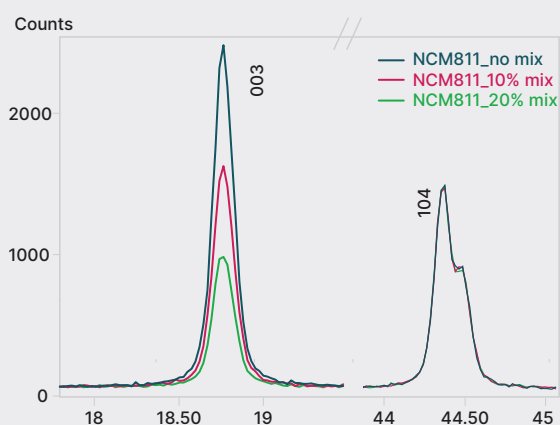


Fig. 10 shows the effect of cation mixing (mixing of Li<sup>+</sup> and Ni<sup>2+</sup> ions in the crystal lattice) on the intensities of the 003 and 104 peaks of the XD pattern – while 104 peak intensity does not change, the 003 peak intensity falls rapidly with cation mixing. Thus, the ratio  $I(003)/I(104)$  can be used to estimate cation mixing. A Rietveld refinement of site occupancy factors would provide higher accuracy.

# Battery cell characterization

## Delivering *in operando* X-ray diffraction

The biggest issue faced when developing new battery materials with high energy density is capacity degradation with cycling. Causes of capacity degradation can include particle cracking, lithium retention in electrodes, electrolyte degradation, and dendrite formation. Understanding these degradation mechanisms is therefore an important step towards the successful development of new battery materials.

*In operando* X-ray diffraction (XRD) can investigate these failure mechanisms by analyzing underlying crystal structure changes during battery cycling. Our Empyrean XRD platform offers options for the *in operando* cycling of various types of battery cells – from coin cells and electrochemical cells to pouch cells and prismatic cells.

## Coin and electrochemical cells

### Opening possibilities for non-ambient exploration

All types of coin cells with at least one side with an X-ray transparent window can be studied on the Empyrean XRD. We provide a specialized coin cell holder, which can be used for charge-discharge cycling.

Another solution is the electrochemical cell, with an X-ray transparent window made from beryllium or glassy carbon. We supply the electrochemical cells with the option of heating and cooling that are mounted on the Empyrean XRD.

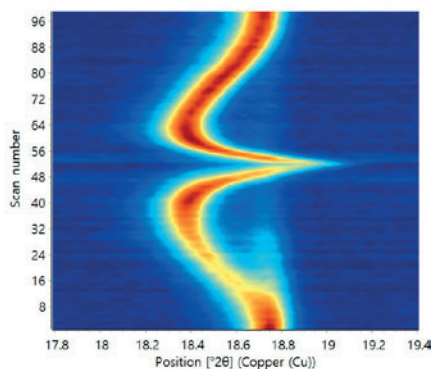


Figure 11

Example of *in operando* cycling of an NCM cathode and graphite anode in an electrochemical cell. Fig. 11 shows how the 002 peak shifts during cycling. Fig. 12 shows how *c* and *a* lattice parameters change during charge and discharge. Any abrupt change in lattice parameter is usually associated with crystal phase changes and may cause particle cracking.

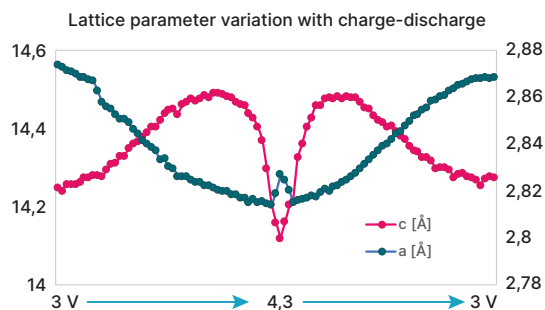
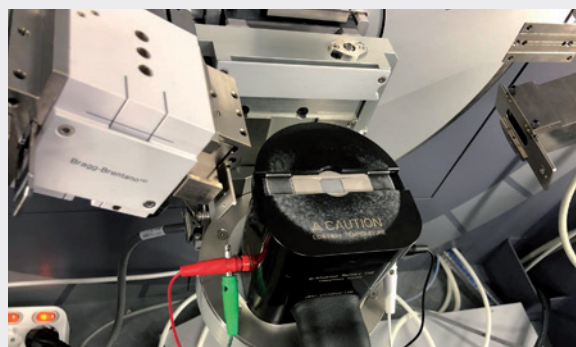


Figure 12



VTEC: Variable Temperature Electrochemical cell for *in-situ* (-10 to 70°C) *in-operando* XRD measurements.

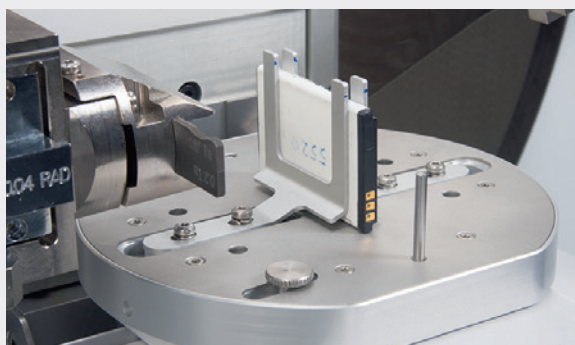
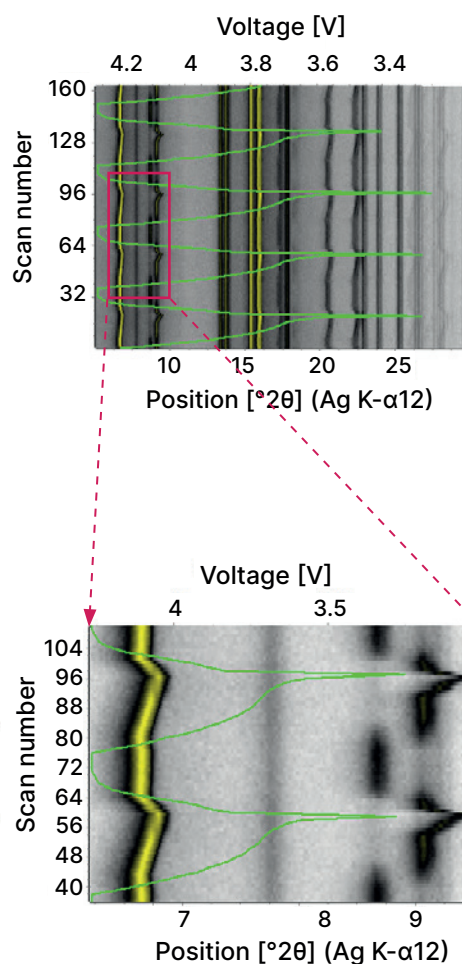
# Pouch and prismatic cells

## Unlocking the full story with hard radiation transmission

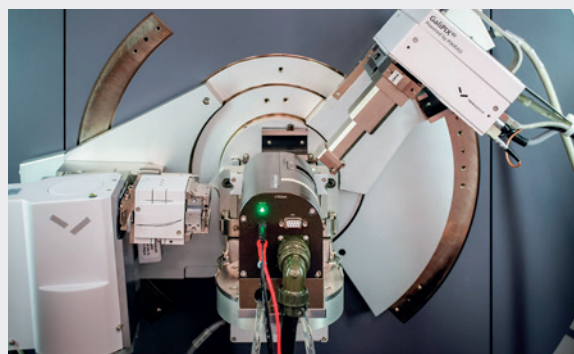
What's more, multilayer **pouch cells** up to 5 mm thick can also be analyzed on the Empyrean XRD platform, when equipped with high energy **Ag radiation** and a **GaliPIX<sup>3D</sup>** detector. The Empyrean supports 60 kV excitation, enabling the high-intensity 22.16 keV Ag radiation suitable for pouch cell research. Special multilayer focusing mirrors deliver high resolution and a high brilliance X-ray beam, further shortening the measurement times.

Example of *in operando* cycling of a pouch cell. Measurements were performed on the Empyrean XRD platform configured with **Ag K $\alpha$**  radiation and the **GaliPIX<sup>3D</sup>** detector. Every XRD scan over 5-30  $2\theta$  range was measured in just five minutes. A total of 166 scans were measured over five complete charge-discharge cycles.

The voltage variation (3.2V in discharged state to 4.3V in fully charged state) is plotted in green line colour. The isoline plot shows cathode and anode peak positions as the cell is charged or discharged. The peak around 6.8°  $2\theta$  is the 003 peak of the NCM cathode and its changing position reflects changes in c-parameter with cycling. The discontinuous peak around 9°  $2\theta$  is from the graphite anode, which changes from C to LiC6 or LiC12 during the charge cycle and then reverses back to LiC12 and then C during the discharge cycle.



Pouch and prismatic cell mounting on the Empyrean XRD. A mechanism to apply pressure on the pouch cell is also supported.



VTEC-Trans: for non ambient (-10 to 70°C) *in-operando* measurements on pouch cells.

## About Malvern Panalytical

We draw on the power of our analytical instruments and services to make the invisible visible and the impossible possible.

Through the chemical, physical and structural analysis of materials, our high precision analytical systems and top-notch services support our customers in creating a better world. We help them improve everything from the energies that power us and the materials we build with, to the medicines that cure us and the foods we enjoy.

We partner with many of the world's biggest companies, universities and research organizations. They value us not only for the power of our solutions, but also for the depth of our expertise, collaboration and integrity.

We are committed to Net Zero in our own operations by 2030 and in our total value chain by 2040. This is woven into the fabric of our business, and we help our employees and customers think about their part in creating a healthier, cleaner, and more productive world.

With over 2300 employees, we serve the world, and we are part of Spectris plc, the world-leading precision measurement group.

**Malvern Panalytical. We're BIG on small™**

## Service & Support

Malvern Panalytical provides the global training, service and support you need to continuously drive your analytical processes at the highest level. We help you increase the return on your investment with us, and ensure that as your laboratory and analytical needs grow, we are there to support you.

Our worldwide team of specialists adds value to your business processes by ensuring applications expertise, rapid response and maximum instrument uptime.

- Local and remote support
- Full and flexible range of support agreements
- Compliance and validation support
- Onsite or classroom-based training courses
- e-Learning training courses and web seminars
- Sample and application consultancy



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