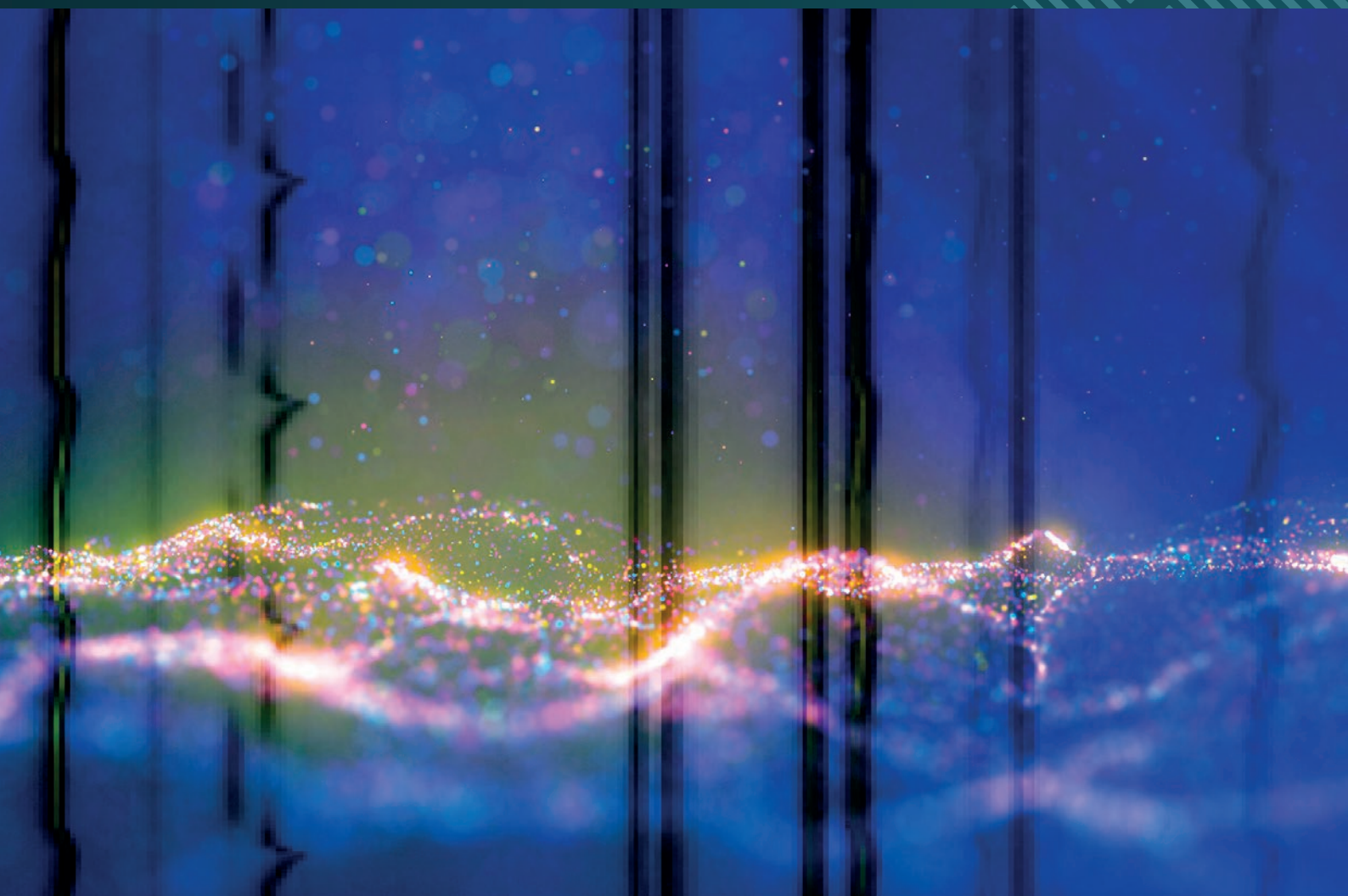




**Malvern
Panalytical**
a spectris company

In-operando XRD solutions for battery research and QC



Empyrean XRD: Perfect platform for battery in-operando studies

Empyrean X-ray diffractometer is a versatile platform for non-destructive materials investigation. X-ray diffraction (XRD) and scattering give insight into lattice parameters, crystallographic structures, oxidation stages, and particle size. It can be applied to samples of cathode, anode, and separator materials, as well as assembled, fully functioning batteries.



Winning the race with deeper insight

Battery research and development is progressing faster today than ever before, in a race to discover the highest-performance, safest, and cheapest material chemistries. Analysis, including *in-operando testing*, is key to this process.

Empyrean XRD also has extensive in-operando capabilities to investigate the changes occurring in cathode and anode materials as the cell is charged and discharged. Flexibility in X-ray source, optics, and detectors make the Empyrean able to investigate a wide range of battery cells.

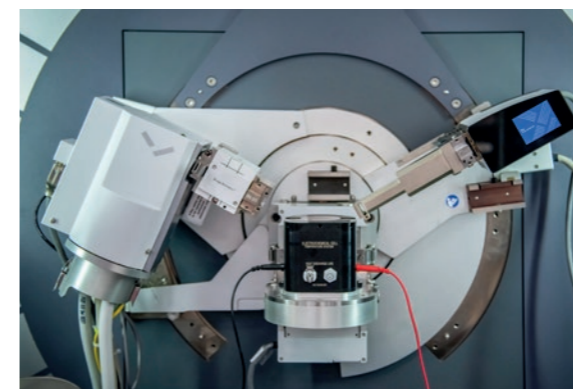


VTEC: Variable Temperature Electrochemical Cells on Empyrean XRD

VTEC

Electrochemical cell for in-operando XRD investigation at controlled temperatures in reflection mode:

- Max. electrode dimensions ≤ 18 mm
- Temperature range: -10 to 70°C
- 2θ scan range: 7 - 140°
- Window material: Be



VTEC-trans

A pouch cell non-ambient stage for in-operando XRD investigation at controlled temperatures in transmission mode:

- Max Pouch cell dimensions: ≤ 6.0 cm x 10.5 cm
- Temperature range: -10 to 70°C
- 2θ scan range: 0 - 70°
- Window material: Kapton

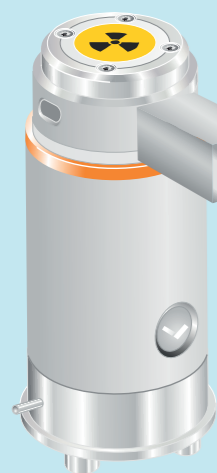


Enabling technologies for in-operando XRD

Optics:

BBHD or iCore: Multilayer optics with 450eV energy resolution for Bragg-Brentano reflection measurements

Focusing mirror: Optics with 450eV resolution delivering high-intensity, high-resolution beam for transmission measurement through pouch cells



X-ray source:

Cu / Co source for reflection and thin sample transmission XRD

Mo / Ag source for transmission through thick pouch cells

Detectors:

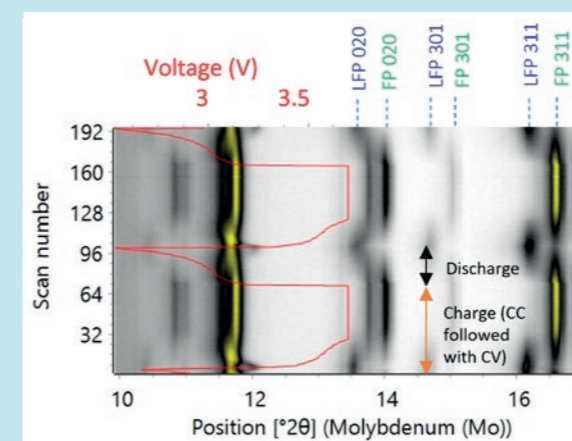
1Der: Detector with energy resolution of typically 340eV and works with Cu, Co, Mo, and Ag radiations

PIXcel: 1D or 2D detectors for multi-applications with Co / Cu radiation

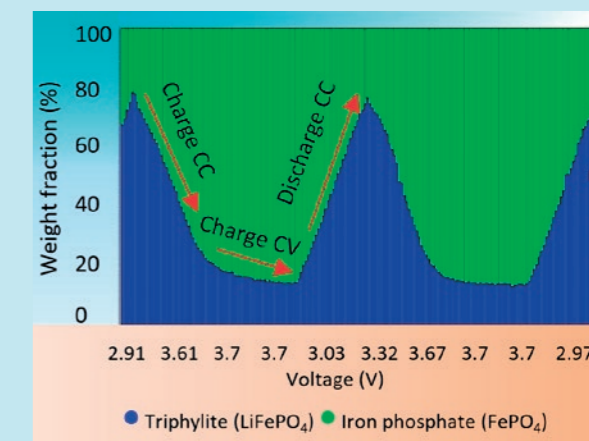
GaliPIX^{3D}: Large area detector with about 100% efficiency for Cu, Mo, and Ag radiation enabling ultrafast measurements on battery cells

VTEC and VTEC-trans electrochemical stages for in-operando XRD at non-ambient temperatures

An example of in-operando XRD measurement on an LFP pouch cell in VTEC-trans measured with Mo radiation. CC followed with CV modes were used during charge and CC mode during discharge cycle (Red curve). Cathode material shuttles between LiFePO_4 (LFP), and FePO_4 (FP) phases as the cell is discharged and charged, respectively.

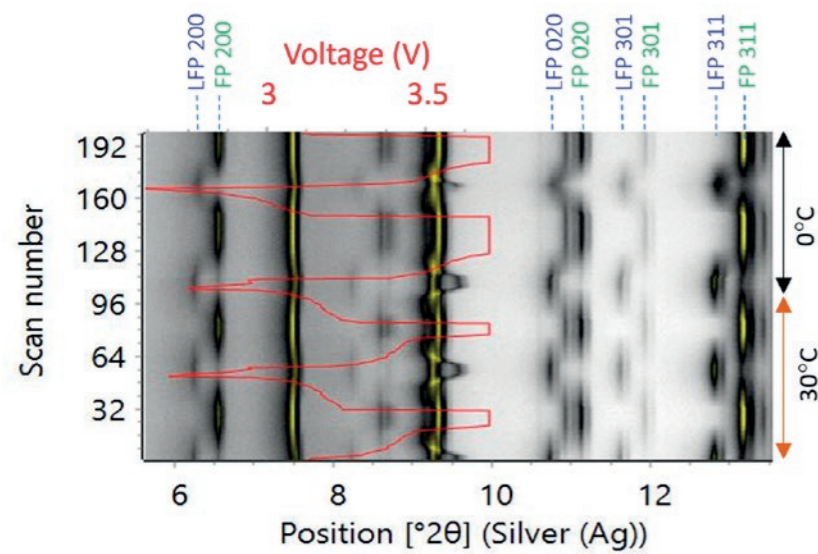


Relative concentration of triphylite (LFP) and iron phosphate (FP) phases as the cell is charged and discharged. This plot was generated in HighScore Plus software after phase quantification via automatic Rietveld refinement on the entire dataset.



Non-ambient in-operando XRD reveals performance degradation at lower temperatures

Cycling a battery at various temperatures produces a lot of data, which increases the analysis complexity. Our all-in-one software package HighScore Plus enables the user to automate phase identification and Rietveld quantification on large datasets. Its advanced toolkit allows extraction of all relevant information from the in-operando data with intuitive ease.

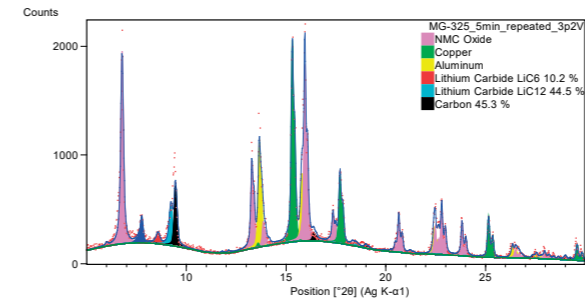


In-operando XRD at variable temperature

This graph is an example of an LFP pouch cell measured in-operando with VTEC-trans using Ag radiation source. Cell was cycled two times at 30°C followed by two cycles at 0°C. Charging was done in CC mode followed by CV mode.

Data Analysis with HighScore Plus

HighScore Plus is a unified platform for in-operando XRD data analysis. Its comprehensive toolkit enables data plotting, phase identification/quantification, crystal structure analysis, and much more. User scripting allows many tasks to be automated, like automated Rietveld quantification on large datasets with pre-defined refinable parameters.

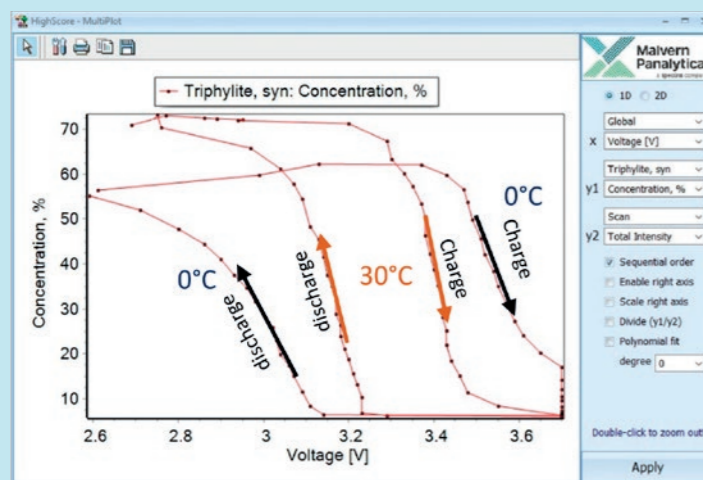
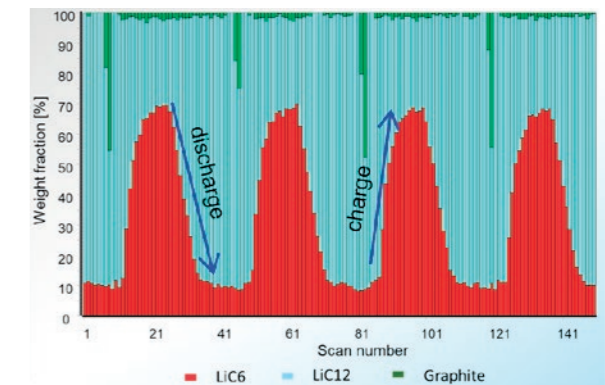


An example of Rietveld refinement on the datasets of in-operando XRD on an NMC pouch cell.

In this example, the Cu, Al, and NMC phases were Pawley fitted while graphite and its lithiated phases were Rietveld fitted to understand the lithiation of anode at different SOC.

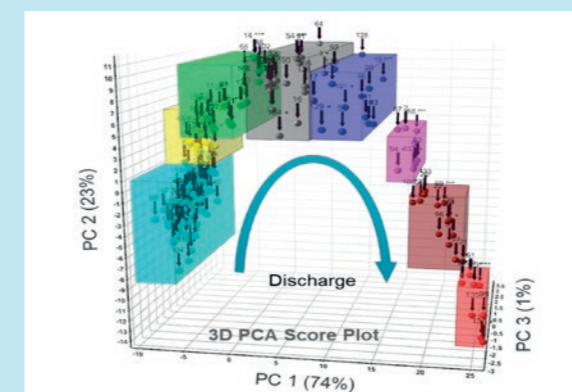
After automatic Rietveld refinement on all datasets of an NMC pouch cell cycled 4-times, the phase variations with charge-discharge can be easily visualized with 2D phase concentration plots.

This graph shows relative concentration of graphite, LiC₁₂, and LiC₆ phases as the cell is cycled.

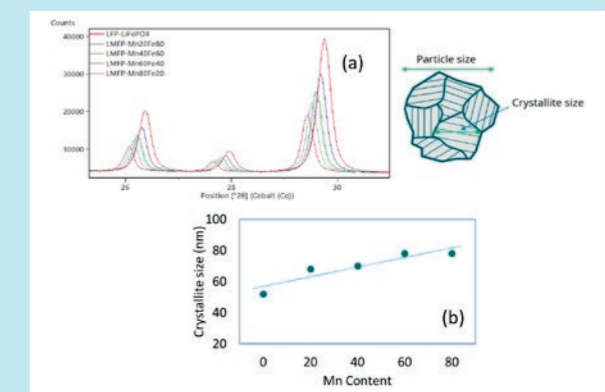


Voltage-crystal phase hysteresis

This graph shows the variation of the triphylite (LiFePO₄) phase in the cathode as the cell is cycled at 0°C and 30°C. As the cell is charged, the cathode material is gradually transformed to the iron phosphate (FePO₄) phase. Larger voltage hysteresis at 0°C indicates performance degradation at lower temperatures



Pattern correlation cluster analysis in HighScore Plus showing various microstates the NMC cell passes through as it is cycled.



(a) XRD pattern shift with increasing Mn content in LMFP samples due to lattice expansion, and (b) crystallite size estimated in HighScore shows size increase with increasing Mn concentration.

Fully integrated potentiostat and temperature controls

Data synchronization:

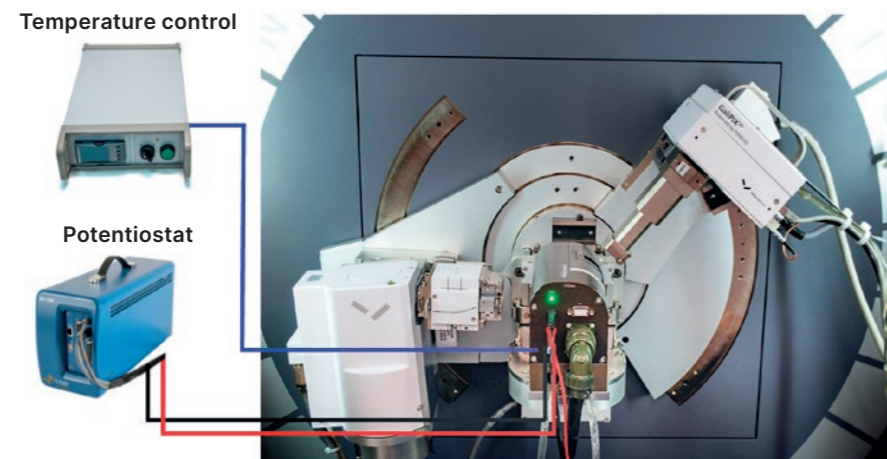
Data collection and analysis is made straightforward with an integrated software interface, controlling the Empyrean XRD, temperature controller, and biologic potentiostat enabling various charging modes such as:

- Constant current mode (CC)
- Constant current mode followed by constant voltage mode (CC-CV)
- Multi-stage constant current (MCC)
- Open circuit voltage mode
- Cyclic voltammetry mode

Convenient and safe operation

- Setting C-rate or mA, user battery library
- Temperature control
- Flexible XRD measurement programs (static and scanning)
- Cut-off current/voltage
- All parameters along with XRD data stored in the same file

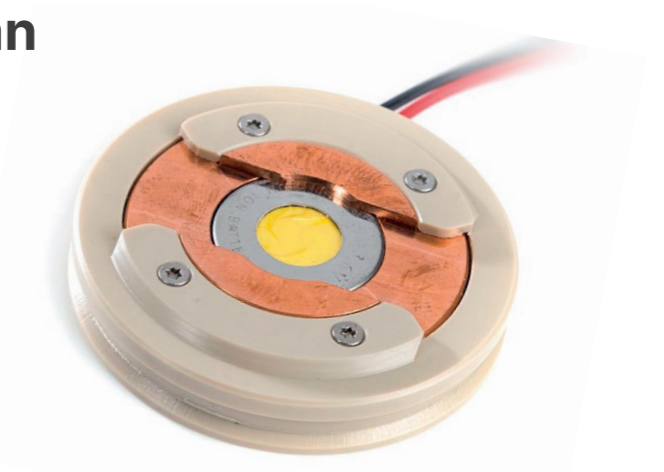
Integrate other potentiostat brands on request, e.g. Ivium, Lanhe, etc.



Ambient electrochemical battery cells on Empyrean

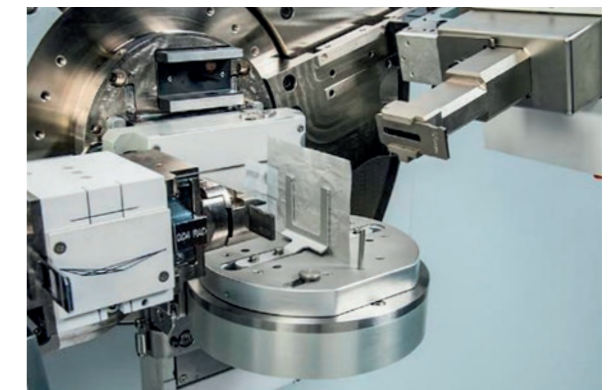
Reflection and transmission in-operando coin cell holders:

CR20xx coin cells with one or two sided Kapton window can be mounted for in-operando cycling on Empyrean or Aeris XRD



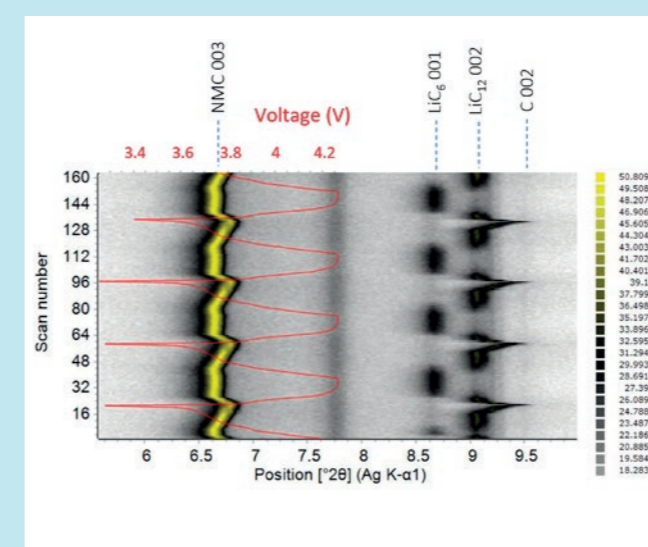
EL-CELL:

Electrochemical cell for assembling and investigating batteries in-operando at ambient temperature



Transmission clamping module:

to mount pouch cells for in operando XRD at ambient temperature



In-operando charge-discharge experiment on an NCM333 pouch cell performed on Empyrean XRD at Ambient temperature.

Isoline plot shows lattice expansion and contraction in cathode and phase transformations to LiC_{12} and LiC_6 phases in graphite anode as the cell is cycled.



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.

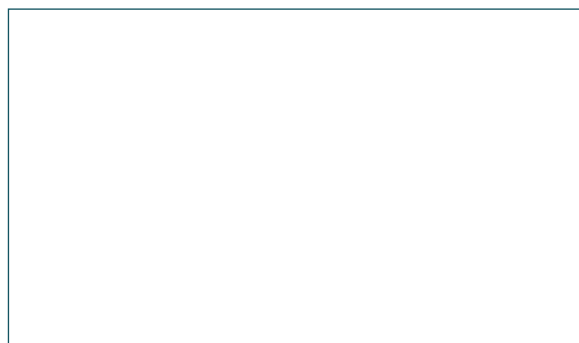
Malvern Panalytical. We're BIG on small™

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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